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Effect of sealing temperature on the sealing edge performance of vacuum glazing

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ABSTRACT

Because inadequate control of the temperature of vacuum glazing may cause changes in form, stress, microstructure, or performance and thereby affect its lifetime, vacuum welding has been adopted to seal the vacuum glazing from the side, and experiments using different sealing temperatures have been executed. The impact of different sealing temperatures on the microstructure of the sealing layer was analyzed to evaluate the combination of material science features on the interface and to reveal the influence of the sealing temperature on the hardness and residual stress of the sealing layer. The results show that a sufficiently high sealing temperature will drive the sealing layer to transform from a hybrid structure to the liquid phase, accelerate the element migration, eliminate pores, stabilize and compact the structure and improve the sealing performance of the vacuum glazing. As the sealing temperature increases, the residual stress and hardness substantially increase. However, when the sealing temperature reaches 460 °C, the residual stress and hardness begin to plateau, and when the sealing temperature reaches 470 °C, no further change can be detected. Therefore, a sufficiently high sealing temperature is beneficial to the bonding of glass and sealing solder and can promote the sealing performance of the vacuum glazing.

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1. Introduction

Vacuum glazing is a new energy-saving transparent construction material with excellent thermal insulation, ageing resistance, visible light transmission and infrared light reflection properties. These properties result in good development potential, and have made vacuum glazing a research focus of glass processing technology [1–3]. In 1989, the first successful vacuum glazing sample was fabricated by Collins and Robinson at Sydney University [4].

Vacuum glazing is produced by coating all around two glass panes with sealing solder, and then put them into the vacuum heating furnace for sealing. To avoid deformation from the high temperature, the sealing should be performed below the softening temperature of the glass, which is generally low (for example, the softening temperature of common soda-lime glass is lower than 600 °C) [5]. The sealing of vacuum glazing is a low-temperature process, and the vacuum glazing sealed in a heating furnace should maintain its shrinkage during the cooling process [6]. This shrinkage could generate stress on the sealing edge of the vacuum glazing, and if the stress exceeds the allowable stress of the glass or sealing layer, the glass, the sealing layer or the sealing interface could break. Therefore, the sealing solder must have a thermal expansion coefficient that matches that of the glass. Different sealing temperatures may change the growth form and the grain size of the solder, and the element diffusion caused by the element affinity on the bonding interface between the solder and the glass substrate may further change the sealing form, stress, microstructure and performance of the glass and thereby lifetime [7,8]. Thus vacuum glazing has a very great need for high-performance sealing solder and sealing technology. Due to the difficulty of the manufacturing vacuum glazing and the few people who have mastered this technology, research has mainly focuses on the bearing stress, the residual stress of the sealing edge and heat transfer mechanisms of the vacuum glazing [9–12]. Manufacturing theory and design theory remain to be established, as the theoretical research in this field is still at an exploratory stage and achievements are few.

Based on the vacuum welding method of side sealing vacuum glazing proposed at Yangzhou University, this paper takes full advantage of the excellent performance of $PbO{-}TiO_2{-}SiO_2{-}R_xO_y$

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Table 1
Composition and various performance indexes of the sealing solder.

Composition/wt%	PbO	TiO_2	SiO_2	CuO	Fe_2O_3
	75	15	10	<2.5	<2.5
Properties	Sintering temperature/°C 420–520	Coefficient of thermal expansion/$°C^{-1}$ 90–94 × 10^{-7}		Melting temperature/°C min^{-1} 460–480	

system welding solder. It discusses the manufacture of the solder and the sealing process [13,14]. Then it describes a series of trails on vacuum glazing at different sealing temperatures, analyses the impact of the sealing temperature on the microstructure of the sealing layer, studies the diffusion of the affinity element on the sealing interface, evaluates the effectiveness of the combination of material science features on the interface and reveals the influence of the sealing temperature on the hardness and residual stress of the sealing layer. This research not only provides significant data related to the application and verification of vacuum glazing, the prediction of its lifetime and the design and improvement of its structure, but also lays the foundation for the manufacturing and commercialization of the vacuum glazing.

2. Testing material and method

2.1. Testing material

The material used in the experiment is two 800 × 600 mm soda-lime glasses, each 4 mm thick with an elasticity of 72.45 MPa, a Poisson's ratio 0.22, a tensile strength of 40 MPa and a compressive strength of 880 MPa (both the tensile strength and the compressive strength were selected at their lower limits). The spacing distance between each pillar is 40 mm. The diameter of each pillar is 0.6 mm, with a height of 0.2 mm, an elasticity modulus of 55 GPa, Poisson's ratio of 0.25 and a density of 2500 kg/m^3.

Mix 99% pure and 30-μm-diameter PbO and TiO_2 powders in a proportion of 70:15, and then add 10%, <2.5% and <2.5% SiO_2, CuO and Fe_2O_3 respectively (purity ≥ 99.8% and average size ≤50 nm). Place the powder into a stainless steel ball grinder, add some dispersing agent, and mix the powder by the wet milling method, with a powder quality ratio of 10:1, a powder milling time of 0.5 h and a revolving speed of 250 r/min. Then heat the powder in a vacuum drying oven to obtain the system welding solder $PbO-TiO_2-SiO_2-R_xO_y$. The ingredients and properties of the final mix powder are as shown in Table 1. The grain size of the welding solder for the experiment is within 1–25 μm, with an average size of 12.15 μm, as shown in Fig. 1.

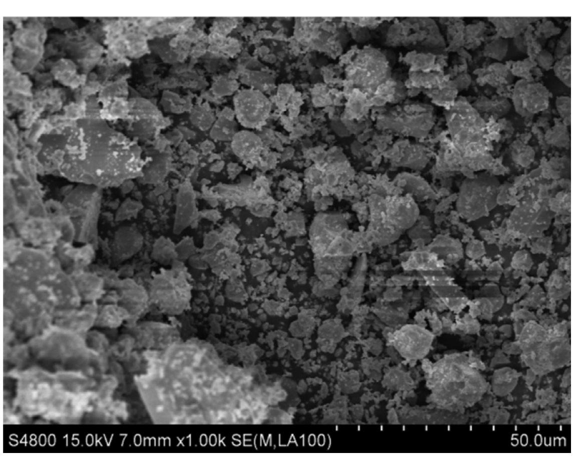

Fig. 1. SEM photo of the sealing solder.

2.2. Testing

The vacuum glazing is performed by placing one clean glass pane over another clean glass pane with pillar array, and then coating around the two glass panes with welding solder. The panes are than heated them in a 420–520 °C vacuum environment for 15–60 min. The two glass panes are sealed with welding solder as the water and organic mater are removed from the glass to form an integral part of the structure [6–8]. The structure of the Low-E vacuum glazing is as shown in Fig. 2.

Using a diamond wire saw cutting machine at the Nanjing University of Aeronautics and Astronautics, we cut a 20 × 20 × 8.2 mm sample from the sealing edge of the sealed 800 × 600 × 8.2 mm vacuum glazing, removed impurities such as oil contamination from the sample, and conducted ultrasonic cleaning in acetone for 15 min. The samples was then repeatedly washed with alcohol and dried and the microstructure of the section and the phase structure of the sealing layer were analyzed using a Hitachi S-4800 thermal field emission scanning electron microscope (SEM) and a D8-Advance poly-crystal X-ray diffraction instrument, Bruker AXS, German [14]. The X-ray testing conditions are a Cu target, a working voltage of 60 kV, a working current of 450 mA and a scanned area of 10°–80°.

A hardness test was conducted by using an MHV-1000 digital Vickers micro-hardness test to determine the hardness of the sealing layer of the vacuum glazing following different sealing temperatures. The hardness measurement conditions were a load of 200 g load and a duration of 15 s. The average value was taken after measuring several points, and the hardness testing position of the sealing layer is as shown in Fig. 3.

The residual stress of the sealing layer was tested using an electrical measuring method, adopting the KHCR-5-120-G16 weldable high-temperature strain gage produced by the Japan KYOWA Company [3]. The high-temperature strain gages were Pre-buried in the welding solder between the two glasses at a distance of 100 mm. Then the two wiring ends of the strain gage were set aside, and they were placed in the vacuum sealing furnace for sealing. The cooled and educed strain gage was connected to the deformation instrument to test the residual stress of each point, and use the average value was used.

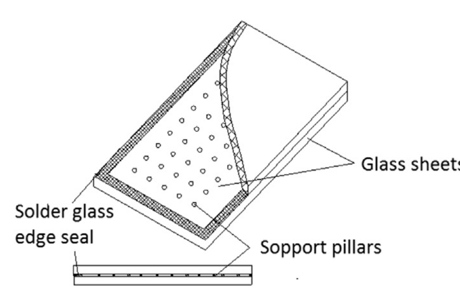

Fig. 2. Schematic diagram of vacuum glazing with a fused edge seal.

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