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Microstructure characteristics of vacuum glazing brazing joints using laser sealing technique

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ABSTRACT

Two pieces of plate glass were brazed into a composite of glazing with a vacuum chamber using PbO-TiO_2-SiO_2-R_xO_y powder filler alloys to develop a new type of vacuum glazing. The brazing process was carried out by laser technology. The interface characteristics of laser brazed joints formed between plate glass and solder were investigated using optical microscope, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) techniques. The results show that the inter-diffusion of Pb/Ti/Si/O elements from the sealing solder toward the glass and O/Al/Si elements from the glass toward the solder, resulting in a reaction layer in the brazed joints. The microstructure phases of $PbTiO_3$, AlSiO, SiO_2 and PbO in the glass/solder interface were confirmed by XRD analysis. The joining of the sealing solder to the glass was realized by the reaction products like fibrous structures on interface, where the wetting layer can help improve the bonding performance and strength between the sealing solder and the plate glass during the laser brazing process.

1. Introduction

In recent years, vacuum glazing which is a new deep processing glass product with the energy-saving characters and high transmittance has been widely used because of its superior thermal insulation, noise insulation and visible light transmission properties, especially in the field of vegetable greenhouse, construction, solar energy application and refrigerated transport [1]. Vacuum plate glass is a new low carbon, environment friendly and leading-edge product with independent intellectual property. The thermal radiation and heat conduction can be reduced by means of proper coating of glass surface, reduction of support pillar and optimal design of sealing [2,3]. These properties have made the vacuum glazing as the most advanced light heat preservation material in the world [4,5]. The sealing forming technology of plate glass has been one of the research hotspots in national and international all the time [6–8]. The first piece of vacuum plate glass sample was successfully developed by Dr. Collins and Dr. Robinson at Sydney University in 1989 [9]. The structure of vacuum glazing is composed of two sheets of soda lime glass with a thickness of 3.5 mm or 4 mm shown in Fig. 1. The edge sealing is made with solder filler which is a low melting point filler alloy. The thermal expansion coefficient of sealing filler matches that of the glass sheets. The support pillars with about 0.2 mm in diameter and 0.15 mm height are matrix-arranged between the sheets, which are a kind of nickel-based alloy with high strength and resistance [8,9].

High temperature vacuum furnace sealing is a method that widely used in the sealing of vacuum glazing. The principle is that the support pillars are placed between two pieces of plate glass, the solder filler is applied in the sealing edge, the whole sample is put into high temperature vacuum furnace for heating, two pieces of plate glass are sealed together, and a sealing vacuum chamber is formed between two pieces of plate glass [3]. There are two main problems on sealing the glazing using the relatively mature vacuum furnace method. Firstly, it has high requirements on the high temperature vacuum furnace. In the case of the larger glass size, it is difficult to manufacture the large inner chamber high temperature furnace. Secondly, the coating film on the plate glass suffers from the thermal decomposition at high temperature, and the visible light transmittance and infrared reflectivity of the coating film are adversely affected, so as to reduce the sealing efficiency and influence the sealing quality. Therefore, it is urgent to develop a new sealing method to solve the problem at the present stage [7–9].

The advantages of laser welding are high energy, small heat affected zone, non-contact, fast welding speed and high welding efficiency [10,11], which can effectively solve the problem of high temperature vacuum furnace sealing [12,13]. Nowadays, the

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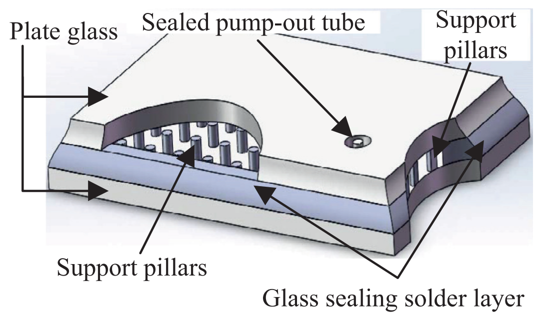

Fig. 1. Schematic diagram of a vacuum glazing.

aspects on laser welding heat transfer, stress strain, technological parameter and brazing quality of vacuum glazing on the laser welding of vacuum plate glass are the mainly research focuses of domestic and overseas scholars [13–16]. Although the theory of laser sealing has been improved gradually, the technology related to the laser welding of vacuum glass has yet to be further studied. This paper takes side edge sealing of vacuum glazing using laser soldering method under vacuum environment. The sealing solder filler is developed taking advantages of the excellent performance of $PbO-TiO_2-SiO_2-R_xO_y$ solder system [17–19]. The vacuum glazing is laser brazed using the sealing solder. The interfacial microchemistry plays an important role in determining a successful brazed joint, and thus it is necessary to investigate the microstructure and energy spectrum analysis of the reaction products formed in the sealing layer [20]. It is expected that a firm bonding strength and surface quality between plate glass and solder filler could be realized on this investigation, thus providing an important theoretical basis for the application, verification, life prediction and structural optimization design of the vacuum glazing, and offering guidance for the manufacturing and industrialization of the vacuum glazing [21,22]. This research work can provide a reference for further research of the edge sealing of vacuum glazing.

2. Testing material and method

2.1. Testing material

The size of vacuum plate glass by laser sealing test is 20 mm × 20 mm × 4 mm. The mechanical properties of plate glass are shown in Table 1. $PbO-TiO_2-SiO_2-R_xO_y$ solder system powder is used in the testing process. The ingredients and various indexes are shown in Table 2.

PbO, TiO_2, SiO_2, CuO and Fe_2O_3 powders are mixed uniformly by definite mass fraction ratio, and put into the stainless steel ball mill for ball milling after adding a certain amount of dispersing agent. After gaining the $PbO-TiO_2-SiO_2-R_xO_y$ solder with the particle size range of 1–25 μm, it is put into the drying box for treatment. Fig. 2 shows the SEM diagram of sealing solder powder. The solder is formed by pressing of the powder pressing machine, with the pressure of 8 MPa and pressure maintaining for 12 min.

2.2. Experiment and testing method

The glass solder powder was formed by dry pressing to be cylindrical shape with dimensions of Φ6 mm × 20 mm. The sealing solder's thermal expansion property was tested by Netzsch DIL 402C thermal expansion analyzer (Germany). The test conditions were as follows, the heating rate was 5 °C/min, and the temperature range was from 20 °C to 460 °C.

The solders were screened with mesh size of 74 μm, and then were formed by dry pressing (5 MPa, 20 min) to be cylindrical shape with dimensions of Φ12 mm × 4 mm. The dry pressing forming solder cylindrical with the dimensions of Φ12 mm × 4 mm was put flat on the sold-lime glass substrate which was ultrasonically cleaned using acetone and dried to remove the impurities. After that, the sold-lime glass substrate was put into the vacuum furnace (Model: ZDZK30-1650) and heat-treated under nine different brazing temperature (410 °C, 420 °C, 430 °C, 440 °C, 450 °C, 455 °C, 460 °C, 470 °C and 480 °C) and kept for the same dwell time (4 h). During the brazing process, the specimens were all heated and cooled at the rate of 2.5 °C/min in the vacuum furnace. In order to ensure the sealing temperature of the $PbO-TiO_2-SiO_2-R_xO_y$ system, the relationship between the deformation of the sample and the temperature was recorded by digital camera.

The glazing was ultrasonically cleaned using ethanol and dried to remove the impurities for 10 mins. The cream solders were coated on the inner edge of the flat glass as the test sample. Finally, the sample was placed on the fiber laser platform, and different laser parameters were selected to test. The vertical surface of laser brazing sealing surface was cut into 8 mm × 8 mm × P mm (P represents the thickness of the sample, according to the shape of the sample) by diamond wire saw. The cutting parameters were as follows: the saw silk speed was 1.5 m/s, the feed rate was 1 mm/min, the diamond wire pneumatic tension was 0.22 MPa and the cutting process was using water-based coolant to cooling. The cutting sections were polished with different mesh sand-paper and polishing machine for further examination.

The spraying sections were measured by Hitachi S4800 scanning electron microscope (SEM) to observe the micromorphology of the interface. The interface was line scanned using SEM on six different elements (Ti, Al, O, Na, Pb and Ca). An energy dispersive spectroscopy (EDS) system was applied to analyze the distribution pattern of the filler elements. X-ray diffraction (XRD) with XD-3A Cu Kα radiation was employed to detect the phase composition of the bonding interface.

3. Results and discussion

3.1. Thermal expansion property of sealing solder

The effect of thermal expansion property of sealing solder on glazing is very important. A better safety performance of sealing edge cannot be achieved under extreme environment (excessive cold and heat) unless the similar thermal expansion performance between sealing solder and glass matrix. Fig. 3 shows the thermal expansion curves of $PbO-TiO_2-SiO_2-R_xO_y$ solder and sodium calcium glass materials. In the temperature range of 20 °C to 445 °C, the thermal expansion coefficient of the sealing solder is $9.1 \times 10^{-6}\ K^{-1}$, which is similar to that of sodium calcium glass (CET: $10.2 \times 10^{-6}\ K^{-1}$).

3.2. The welding temperature of sealing solder

Fig. 4 shows the welding deformation of cylindrical sealing solder (Φ12 mm × 4 mm) of dry pressing forming at different temperatures. As shown in Fig. 4, the cylindrical sealing solder was not melting at 440 °C; the solder began to melt and had a slight collapse at 450 °C which was the initial melting temperature of the $PbO-TiO_2-SiO_2-R_xO_y$ system. Therefore, the lower limit sealing temperature of glazing was set at 450 °C. The whole serious collapse deformation and flat structure of cylindrical solder were presented at 460 °C because of its own gravity. The overall collapse, low viscosity and molten state of the solder were presented at 470 °C. Therefore the sealing temperature of glazing should be controlled within the softening point in the manufacturing process.