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Glass-Copper anodic bonding through activated Sn-0.6Al solder

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A B S T R A C T

The application of an electric field promoted the migration of Na^+ ions on the glass substrate. A sodium-depleted layer was formed on the glass surface, playing a key role in a successful bonding. Al accumulated at the glass/solder interface and enhanced the interfacial reactions. Al_2O_3 + Al_2SiO_5 + SnO, Cu_6Sn_5 and Cu_3Sn reaction layers were respectively detected on both sides. The electric field and bonding temperature were varied to illustrate their impact on the interfacial bonding. The joint formation mechanism is discussed. When the bonding voltage was 1400 V and the bonding temperature was 400 °C, the glass/copper joint was free of cracks, and had a maximum shear strength of 11.5 MPa.

1. Introduction

Due to its good oxidation and corrosion resistance, as well as superior electric and thermal insulativity, a broad use of the glass has been in wafer-level packaging (Schmidt, 1998), edge seal for vacuum solar collectors (Koebel et al., 2010), and micro-sensors (Briand et al., 2004), etc. Its reliable bonding to metal, especially copper (Elrefaey et al., 2014), is essential and has been an attractive topic and in urgent need.

As a challenge to researchers for more than 150 years, such a bonding is of great difficulty due to the inevitably significant differences in the chemical and physical properties between glass and metal. In practical applications, the bonding is required to be not only mechanically strong (Sugiyama et al., 2008), but also be highly stable under the effect of heat, gas, moisture, or sometimes X-ray (Cao et al., 2014). Such harsh service conditions result in the exclusion of the adhesion bonding with organic glue. Conventional glass/metal joints are mainly achieved through hot pressing at the glass softening temperature to ensure a sufficient surface contact and a high atomic interdiffusion between the glass and metal (Sato et al., 2003). However, the high bonding temperature and large deformation of the glass restrict its application in many fields. As for brazing, a widely-used technique to join dissimilar materials (Feng et al., 2017), commonly used metal liquid does not wet glass. Filler metal containing a small amount of active elements (such as Ti, Nb, V, Zr, which can promote the interfacial reactions between the glass substrate and the filler metal) (Feng et al., 2016), has a high melting point (Guo et al., 2017) and cannot apply on joining glass to metal. These issues make a sound glass/metal bonding become nearly impossible.

For glass-metal bonding, an attention must be paid to (i) improving the surface atomic mobility, (ii) lowering the bonding temperature, (iii) as well as relieving the adverse effect of CTE mismatch between glass and metal on joint properties (Li et al., 2016). Anodic bonding well meets above stringent requirements. By applying a DC voltage across the bonding couple with the glass side being cathode, the migration of the positive ions in glass can be activated at low temperature (Takahashi et al., 2004), resulting in the formation of a negative charge layer on the glass surface (Denee, 1969). The non-bridging oxygen anions in this layer lead to the simultaneous anodic oxidation on the metal surface, thus forming a permanent bond. Since the first successful anodic bonding of glass to metal by Wallis and Pomerantz (1969) in 1969, many researches have been done in this field (Dunn et al., 2000). But one major problem that the bonding quality suffers from imperfections because of the uncomplete contact between the bonded surfaces, has still been unsolved until (Koebel et al., 2011) and (El Hawi et al., 2013) developed the so called Activated Liquid Tin Solder Anodic Bonding (ALTSAB). By adding an intermediate layer of tin-based solder, the surface contact between the liquid solder and the substrates is significantly improved, thus ensuring sufficient interaction prior to actual bonding. The large CTE mismatch can also be relieved due to the low Young's modulus of Tin Solder. Thus far, most researches in ALTSAB mainly focus on glass/aluminum (Schjølberg-Henriksen et al., 2006), glass/titanium (Rocha et al., 1995), and glass/steel (Susan et al., 2012) couples. There has been little discussion on bonding glass to copper.

In this work, the glass was anodic bonded to copper with the

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assistance of a Sn-0.6Al solder. The aluminum acted as an active element in the solder to form solder/glass anodic bonds by the diffusion of Al into glass. The joint microstructure as well as the impacts of bonding parameters on the joint microstructural evolution and mechanical properties, were systematically investigated. Meanwhile, the proposed joint formation mechanism was discussed.

2. Experimental procedure

2.1. Materials

The bonded materials used in this work were a standard, 3 mm thick soda-lime float glass, and a 1.5 mm thick commercial pure copper. The float glass was supplied by China Luoyang Float Glass Group Co., Ltd. The copper substrate was supplied by China Yantai Lubao Nonferrous Alloy Co., Ltd. The glass and copper were cut into 10 × 10 × 3 mm and 30 × 15 × 1.5 mm pieces, respectively. Prior to bonding, copper surfaces were polished by SiC papers up to girt 1000. The glass was cleaned in HNO_3 solution. This cleaning step is essential for successful bonding and making the surface of the glass hydrophilic. Then all the bonding samples were cleaned ultrasonically in acetone for 15 min. The Sn-0.6Al (wt.%) solder alloy were synthesized by repeated melting of a mixture of high purity Sn (45 μm, 99.99 wt.%) and Al (45 μm, 99.99 wt.%) powders. The Sn and Al powders were supplied by China Beijing Xing Rong Yuan Technology Co., Ltd. The mixed powders were sealed in a quartz tube under high purity argon. The temperature was cycled between 500 °C and 800 °C for at least 10 times during 30 min. After the melting, the resulted Sn-Al alloy was cold rolled to a 200 μm thick foil.

2.2. Bonding experiments

Fig. 1(a) shows the schematic of anodic bonding process. The solder foil was sandwiched between glass and copper. The glass side of bonding couple was attached to the negative pole while the copper was connected to the positive pole. Then the bonding couple was placed in a chamber which was continuously flushed with high-purity argon to sufficiently remove oxygen. The samples were heated to the peak temperature (400–420 °C) in 30 min. Next, a voltage (1000 V, 1200 V, 1400 V) was applied to the bonding couples. At the end of each experiment, the sample was cooled to room temperature at a slow rate of 5 °C/min to reduce the thermal stress.

2.3. Joint characterization

The cross-section surfaces of the joints were firstly polished using diamond millstones with different particle sizes (500-grit, 800-grit, 1000- grit and 1200-grit, successively). Then the samples were polished using 1 μm diamond polishing spray and were fully dried in a drying oven. The microstructure analysis was performed by scanning electron microscopy (SEM, Helios Nanolab 600i, FEI Co., Ltd, USA) equipped with energy dispersive spectrometer (EDS) in the back-scattering mode at 20 kV. The X-ray diffraction analysis (XRD, D8 ADVANCE) with Cu/Ka1 radiation was used to determine the phases of the joint.

Mechanical performance of the joint was studied by room-temperature shear test. The test was performed with a universal testing machine (INSTRON 5569, USA) equipped with a special fixture at a shear rate of 0.5 mm min^{-1}. Fig. 1(b) shows the schematic diagram of the tested specimen's configuration.

3. Results and discussion

3.1. Microstructure

Fig. 2 represents the microstructure of the glass/copper joint bonded at 420 °C for 10 min under a voltage of 1200 V. The Sn-Al solder well bonded with adjacent substrates without any voids and cracks. A large amount of Sn-Al solder remained in the joint, forming an 87.8 ± 2.5 μm thick intermediate layer. To investigate the joint microstructure in detail, the magnified images of each side are shown in Fig. 2(b) and (c). Two continuous layers were observed at the copper/solder interface, marked as I (the gray layer, 1.17 ± 0.5 μm) and II (the light gray layer, 1.8 ± 0.6 μm). The EDS analysis results indicate that the chemical compositions of the layer I and II were respectively 73.71Cu-25.97Sn-0.32Al (at.%) and 56.34Cu-43.45Sn-0.21Al (at.%). Since their Cu:Sn atomic ratios were close to 3:1 and 6:5, layer I and II were respectively identified as the Cu_3Sn, Cu_6Sn_5 reaction layers according to interfacial reactions between Sn-Al solder and copper ($Cu + Sn \rightarrow Cu_6Sn_5$, $Cu_6Sn_5 + Sn \rightarrow Cu_3Sn$) (Mo et al., 2015). These two layers have also been reported in brazing copper using Sn-based solder (Zhang et al., 2016).

Unlike the winding interface on copper side, the glass/solder interface was straight. No distinguishable reaction or dissolution phenomenon could be observed. This morphology was consistent with the observations by Elrefaey et al. (2014) and Malfait et al. (2016) in bonding glass to titanium, steel, Fe-Ni alloy. To characterize the atomic diffusion behavior, the elemental linear scanning was conducted along the white line (shown in Fig. 2(c)). Results in Fig. 2(d) indicate that Na represented a relative low content in the glass substrate near the interface, forming a ~ 4 μm thick sodium-depleted region. This sodium-depleted region was reported as the feature of anodic bonding. Its formation is mainly due to the migration of Na^+ away from the glass/solder interface toward the cathode. As a result, the ≡Si—O—Na would turn to $\equiv Si{-}O^{-1}$, forming a negative charge layer on the glass surface. Metal atoms which diffused into this layer from the solder were oxidized, creating a bond between the solder and glass. Since Al has a stronger affinity to O than Sn, it accumulated near the glass/solder interface, leading to the concentration peaks of Al near the glass/solder interface. During a shear test, some joints fractured within glass/solder

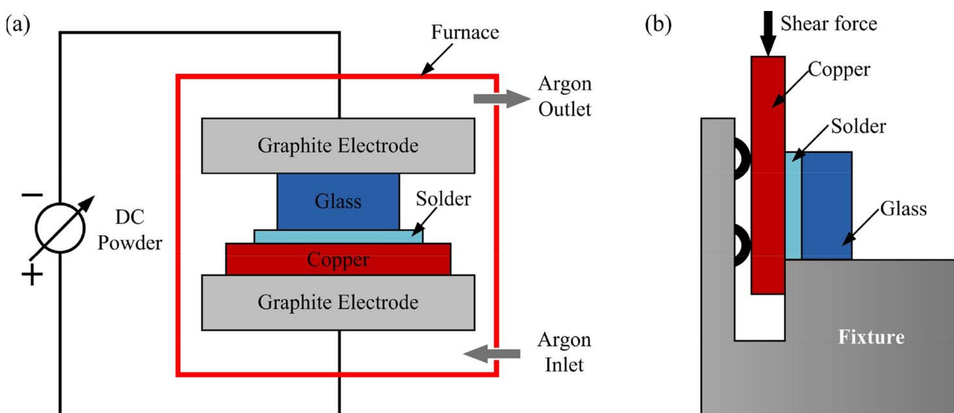

Fig. 1. Schematic diagrams of (a) the experimental equipment and (b) the shear test.

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