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Interfacial microstructures and glass strengthening in anodic-bonded Al sheet/glass and sputtered Al film/glass

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ABSTRACT

Two anodic bond interfaces were fabricated at 300 °C, between glass and either an Al sheet or a sputter-deposited Al film, and their microstructures and bending strengths were comparatively studied. In the Al sheet/glass interface, numerous local intrusions of crystalline Al_2O_3 with a long (100–350 nm) dendritic structure were formed in the glass adjacent to the aluminum. However, in the sputter-deposited Al film/glass interface, a continuous, thin (~30 nm) amorphous layer with Al-oxide nanocrystals along the interface was present without the formation of dendrites after anodic bonding. The dendritic structures in the Al sheet/glass are attributed to an electrostatic instability imposed by the roughness and local oxidation of the Al sheet surface or, presumably, by microheating via gas discharge at the interface. The bending fracture strength for both types of bonded glasses increased by approximately 1.7 times compared with that of the bare glass due to the interfacial reaction.

1. Introduction

Anodic bonding is a bonding technique that directly joins thin metals or silicon to alkali-ion-conductive glasses via field-assisted diffusion at elevated temperatures below their glass softening point, thereby forming an interfacial bond with sufficient strength to withstand a load up to the glass fracture. The method has been widely applied to electronic and microelectromechanical systems (MEMS) packaging [1–3], including high-quality hermetic sealing and encapsulation [4,5], device fabrication for optical and photonic uses [6,7] and medical components [8,9]. The interfacial reaction during bonding has been widely studied. When a high voltage is applied for contact between glass and thin metals that are connected to the cathode and anode, respectively, mobile alkali ions in glass migrate away from the interface, leaving behind a negatively charged alkali-ion depletion layer [10–13]. In an Al/glass contact under a high electric field, Al ions diffused into the depletion layer in the glass, thereby forming an Al–O bond and oxide or compound phases at the interface region [11,12,14].

The reason for the high joint strength of anodic bonds has been explored through observations of the interfacial microstructure. The diffusion of Al ions into glass occurs to a depth of tens to hundreds of nm and results in the formation of numerous covalent Al–O bonds in the interface region [11,13]. The dendritic structure of γ-Al_2O_3, which grows into the alkali-ion depletion layer, could create strong bonding through the anchoring effect of Al onto glass [11,12,14]. In a recent study, the development of the dendritic structure was suggested to be associated with the non-uniform distribution of electrostatic fields along the glass/Al interface due to surface roughness [15]. However, the literature contains few studies focused on the microstructural features that cause the instability of electrostatic fields at the interface. In this work, to identify the relationship between interfacial imperfections and the bonding microstructure, we have comparatively studied two anodic bond interfaces that were formed by an Al sheet in simple contact with glass and by the sputter coating of an Al film onto glass.

2. Experimental details

A Pyrex 7740 glass wafer (100 × 100 × 0.5 mm^3, Corning) and an aluminum sheet with thickness of 6 μm (99.5% purity) were used in the present work. Both were cleaned in methanol and acetone and dried with flowing nitrogen gas. The surface roughness of the as-received Al sheet and glass was measured using an atomic force microscope (AFM) (Veeco NanoMan VS) in tapping mode. The bonding system is schematically illustrated in Fig. 1. The glass was placed on a Cu plate in an open-top chamber and heated to 300 °C. The open-top was covered with an Al sheet, and the contact between the edge of the chamber top and the Al sheet was tightly sealed using a ring and suction pump. The distance between the Al sheet and the glass wafer was 10 mm. The Al sheet was then bulged and contacted against the entire glass surface by evacuating the chamber using a vacuum pump. The Cu plate below the glass and the top Al sheet were connected to the cathode and anode, respectively, of a DC power source. The voltage was increased to 900 V over a period of 10 min and then maintained at 900 V for

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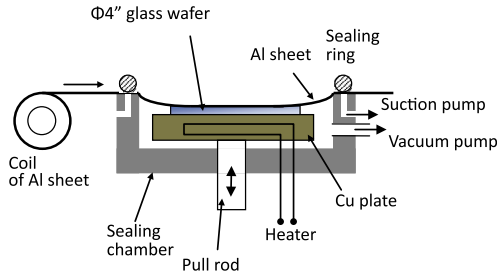

Fig. 1. Schematic diagram of the anodic bonding equipment. The pull rod controls the gap between the Al foil and glass wafer.

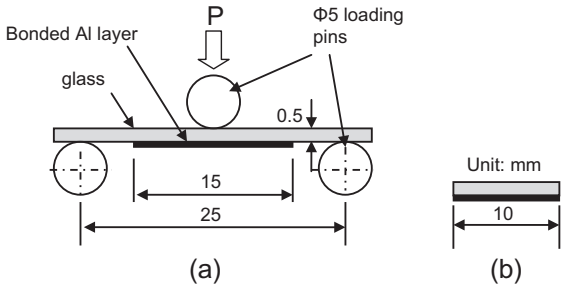

Fig. 2. Schematic configuration of the bending test specimen and loading pins: (a) and (b) are the front and side views, respectively. The loading pins in (a) were excluded in the side-view image in (b).

5 min. The sample was then cooled to room temperature. Another glass wafer was deposited with a layer of pure Al (99.99%) with a thickness of 700 nm using an rf magnetron sputtering system. The rf power was 300 W, and the distance between the target and substrate was 10 cm. The base pressure in the sputtering chamber was 5.0×10^{-7} Torr, and the deposition was performed with an Ar gas flow rate of 20 sccm. The sputtered Al film/glass was subjected to the same bonding process used for the Al sheet/glass.

Cross-sectional samples for transmission electron microscopy (TEM) were prepared using an *ex situ* lift-out technique in an FEI NOVA200 focused ion beam (FIB) system. A JEOL JEM-2100F field-emission transmission electron microscope equipped with a JEOL JED-2300T energy-dispersive X-ray spectroscopy (EDS) detector was used to observe and analyze the bond interfaces.

The bending strength of the bonded glasses was evaluated using a three-point bending test jig installed in an INSTRON 5848 micro material tester, as illustrated in Fig. 2. Four different sample types—bare glasses, as-sputtered Al/glass, anodically bonded Al sheet/glass and sputtered and anodically bonded Al film/glass samples—were tested for 2 to 5 samples of each sample type. Additionally, some of the sputtered and anodically bonded Al film/glass samples were immersed in a NaOH:H_2O (1:1) solution until the Al layer in the samples was completely removed, and the bending strength of the remaining glass substrate was tested. The samples were machined into 10×20 mm^2 specimens using a dicing saw and were placed in the jig with the loading pin placed opposite the bonding surface. The pressing speed was 1 mm/min, and the force [N] and loading-pin displacement [mm] were recorded during the tests.

3. Results and discussion

3.1. Al sheet/glass interface

Fig. 3a and b presents cross-sectional TEM images of the Al sheet/glass interface bonded at 300 °C and 900 V. A sodium-depleted layer was formed with a thickness of 700 nm as shown in Fig. 3a. Fig. 3b is the image taken at the rectangular area in Fig. 3a. Inside the layer, treelike or dendritic nanostructures are visible. The interval, which was measured from a total of 6 images taken at different locations, was 0.23 μm on average. The interface

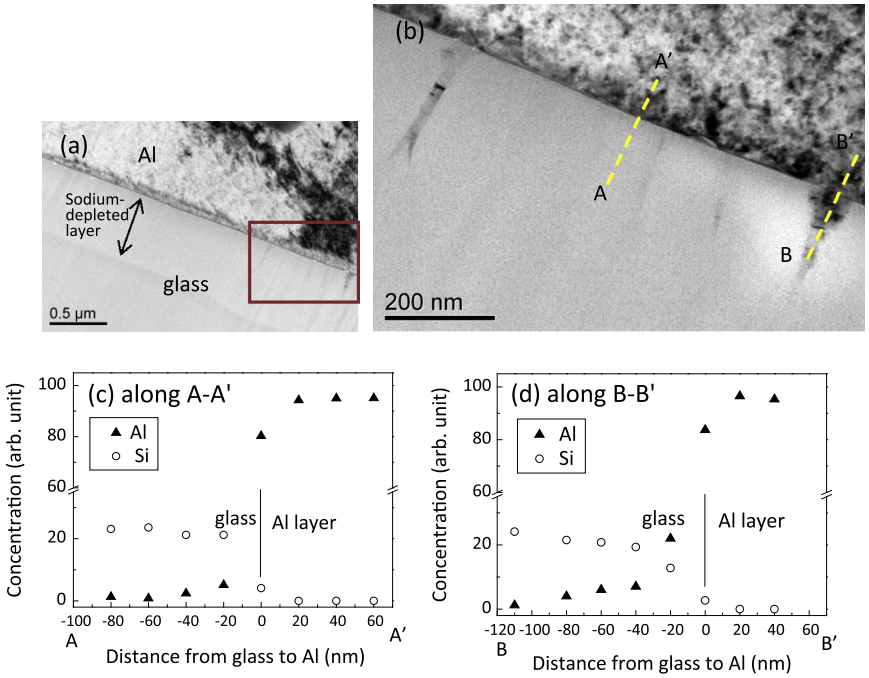

Fig. 3. TEM images (a and b) showing the cross-section of the Al sheet/glass interface that was anodically bonded at 300 °C/900 V. (b) High magnification image taken at the rectangular area in the image (a). The EDS data in (c) and (d) were analyzed along the lines A–A′ and B–B′ in (b), respectively. Line B–B′ was positioned along the dendrite region in the glass. The vertical lines in (c) and (d) indicate the Al/glass interface.