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# Direct glass-to-metal joining by simultaneous anodic bonding and soldering with activated liquid tin solder



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#### ABSTRACT

Anodically bonded glass/titanium and glass/steel were investigated for applications in a variety of industrial sectors. Residual stresses that build up during the bonding or cooling down of a joint to room temperature represent the main challenge to the joining process since they drastically weaken the mechanical strength of the joint. A layer of liquid tin-based solder in between the glass and metal part of the joint is used to reduce the internal stresses and improve the contact between the surfaces. The microstructural characterization of glass/solder/titanium and glass/solder/steel joints formed from Ni coated metal substrates indicated that Ni<sub>3</sub>Sn<sub>4</sub> was formed for both types of joint but with a different morphology and location depending on the type of metal substrate. The average shear strength of the joints was 24 MPa for glass–titanium and 21 MPa for glass–steel joints. For both types of joint, the fracture crack propagated along the glass–solder interface.

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#### 1. Introduction

Conventional glass-to-metal bonds are formed by mechanically pressing a metal surface against the glass near its glass transition temperature. Relatively strong bonds can be achieved by this process because the glass viscously deforms at this temperature and interdiffusion is fast. However, many applications require the bonding to be achieved at lower temperatures where the glass does not deform. Anodic bonding is one of the most versatile bonding processes to permanently bond glass-to-metal at relatively low temperatures. The anodic bonding of various conductive materials to glass was realized successfully for the first time in 1969 by Wallis and Pomerantz (1969). It was possible to irreversibly bond different metals and semiconductors to anion-containing glass by applying a potential (200–2000 V) between the two samples at temperatures typically 300–500 °C below the glass transition temperature. By attaching a cathode to the backside of the glass and an anode to the metal part, electrostatic attraction between the metal and glass forced the two parts to join.

The application of a voltage potential across the parts for a few minutes, with the glass held at a negative potential, causes mobile positive ions (mostly Na<sup>+</sup>) in the glass to migrate away from the metal–glass interface toward the cathode, leaving behind fixed negative space charges, as evidenced by Maluf and Williams (2004). At the same time, anodic oxidation occurs on the metal surface and the electrostatic attraction between the positively charged metal cations and the negative charges in the glass holds the two materials together and drives the bonding of metal to glass. Additionally, the reported strong dependence of the bond strength on the activating element in the solder (e.g. Al, Mg, Li) indicated that the oxidation of Al from the solder and the diffusion of Al<sup>3+</sup> into the glass were essential for the formation of strong solder-to-glass anodic bonds (El Hawi et al., 2013).

Wallis and Pomerantz (1969) successfully produced anodic bonds of Pyrex-glass to materials with a similar thermal expansion coefficient, such as Kovar, Alloy 42 and silicon. According to Schmidt (1998), the most significant area of application for this technology is in wafer-level packaging despite the remaining

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Fig. 1. Backscatter electron (BSE) image of the steel and titanium substrates coated with nickel.

challenges to convert some of the new, promising technologies into accepted production techniques. However, anodic bonding of metal to glass can also be of great interest for a variety of industrial applications which today are still untapped. One example is the edge seal for vacuum insulated glazing or vacuum solar collectors, for which the long design life prohibits the use of organic glues (Koebel et al., 2010). Another example is the packaging of sensors operating in harsh environments reported by Briand et al. (2004) and for piezoresistive silicon sensors used as pressure or liquid flow sensors investigated by Weber et al. (2002). Additionally, Blom et al. (2001) integrated robust interconnects and sealing of devices in microfluidics. One of the applications of bonding glass to metal, particularly copper, is in the semiconductor roystal is clamped between two copper-clad wires (Hanneborg et al., 1991).

The main advantage of anodic bonding is that it produces a strong, permanent bond without the need for pre-metallization, adhesives or excessively high temperatures, as it would be required for conventional bonding at the glass transition temperatures. The main disadvantage of anodic bonding is related to the limitations on the combinations of materials because of the need for similar coefficients of thermal expansion (CTE). Glass typically has a lower CTE than metals, so there is a mismatch between the coefficients of thermal expansion. After the bond is formed, the joint is cooled down to room temperature and the different CTEs result in thermal residual stresses that weaken the mechanical strength of the joint.

In this study, an intermediate layer of tin-based solder was used to anodically bond materials with different CTEs. The tin solder was anodically bonded to glass and at the same time soldered to metal. This method allowed the bonding of materials with different CTEs, because tin has an intermediate CTE and a low Young's modulus and therefore decreases the residual stresses. An important advantage of carrying out the anodic bonding in the liquid state is that it offers good contact of the liquid metal sealant to the glass. A uniform contact of the tin-based filler was ensured by the interaction of the liquid solder with the base materials prior to the actual bonding which was earmarked by an early stage partial wetting (Koebel et al., 2011). The electric field applied during the anodic bonding, and also the presence of an activating element in the solder further enhances wetting of materials by the liquid solder (El Hawi et al., 2013). When carried out in the absence of oxygen (vacuum or a protective inert gas atmosphere), glass metal joints can be formed at low melting temperatures without flux. It should be mentioned that because of the poor intrinsic wettability of the investigated metals by molten solder, a fine nickel layer was used to improve wetting at the metal side of the joint. In summary, the objective of this investigation was to demonstrate the feasibility of anodic bonding of glass to dissimilar metals such as titanium and steel using tin-based solders. This was achieved by carrying out anodic bonding of glass coupled with a soldering process to metal. The focus of this study was on the interfacial microstructure and the mechanical strength of the joints.

#### 2. Experimental work

#### 2.1. Materials

The base materials used in this work were 3 mm thick soda–lime float glass, 2 mm thick CP titanium (ASTM Grade 2), and 2 mm thick low carbon steel (AISI 1008). The glass was cut into  $20 \times 20$  mm squares and the metal into  $10 \times 10$  mm squares. Metal surfaces were polished to a 1  $\mu$ m finish and a 3  $\mu$ m thick nickel layer was deposited on the titanium and steel surfaces by arc physical vapor deposition (Arc-PVD). The activated solder alloy, Sn0.994Al0.006 (wt%) was prepared by repeated melting of a mixture of pure tin and a Sn0.98Al0.02 parent alloy at 900 °C under vacuum as reported in detail by El Hawi et al. (2013).

Cross sections of the steel and titanium samples after the Arc-PVD coating are shown in Fig. 1. The deposited nickel layer is continuous but inhomogeneous in thickness. This is typical for the applied arc-PVD process because of the droplet formation during the deposition. The average layer thickness was adjusted to be ca. 3  $\mu$ m. The interface showed a smooth diffusion layer and transition of elements between the coating and the substrate.

#### 2.2. Coupled anodic bonding/soldering process

Fig. 2 shows a schematic of anodic bonding/soldering process used to produce the glass-metal joints. The solder was introduced between the glass and metal as a  $30\,\mu m$  thick foil. The glass side of the joint was connected to the ground while the metal side was attached to the positive lead of a programmable HPx20157 high voltage power supply which can deliver up to 150 mA of current at a maximum voltage of 2000 V. PC interface was used to control the high voltage power supply which also recorded the voltage-current data. The experiment was set up inside a rectangular vacuum chamber with a transparent glass door that enabled the close observation of the experiment. First, the chamber was evacuated to approximately  $5 \times 10^{-4}$  mbar by means of a Pfeiffer/Balzers turbomolecular pump. Then, the sample was heated by a pair of copper heaters to 250–350 °C within 15 min. The solder alloy melted within the 30 min thermal equilibration period. Next, a positive voltage of 600, 800 or 1000 V was applied to the metal



**Fig.2.** Schematic illustration of the experimental setup for the coupled anodic bonding/soldering process. The electrical current is sustained by electron conduction in the metallic parts of the circuit and by ionic conduction in the soda-lime glass.

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