Joining of SiO₂ glass and 316L stainless steel using Bi–Ag-based active solders

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ABSTRACT

Active brazing is a commonly used method for joining dissimilar materials with at least one non-metallic component. In the present study, joining of SiO₂ glass to 316L stainless steel was performed utilizing Bi–Ag-based solders. Ti up to a concentration of 4 and Mg up to 1 wt.% were added as active elements. Microstructures of the solder alloys in the as-cast state and of cross sections of the joined compounds were analysed using scanning electron microscopy and energy-dispersive X-ray spectroscopy. In the as-cast state of the solder, Ti is found in Bi–Ti intermetallic phases; Mg is partially dissolved in the fcc-(Ag) phase and additionally contained in a ternary Ag–Bi–Mg phase. After soldering, a tight joint was generated using several alloy compositions. Ti leads to the formation of reaction products at the steel/solder and glass/solder interfaces, and Mg is exclusively accumulated at the glass/solder interface.

Introduction

Besides the optical transparency of SiO₂ glass, its thermal stability is one of the reasons for its increasing use, e.g. as packaging material for electronic and optical devices [1, 2] or for viewing and measuring windows in vacuum applications [3, 4]. Many of such applications require joining of the SiO₂ glass and a metallic substrate. Various techniques such as brazing and soldering [5], mechanical [6], adhesive [7], anodic [8] and diffusion [9] bonding are utilized for joining dissimilar materials (e.g. metal/ceramics and metal/glass joints). Each method

exhibits specific advantages and drawbacks [10]. In order to solder/braze non-metallic materials, sufficient wetting of the substrate by the solder alloy is a prerequisite. Wetting may be achieved by metallization of the substrate surfaces prior to the joining process [11], but an alternative, frequently used approach is adding chemically active elements, most commonly Ti, Zr and Hf to the solder alloy [12–15]. Furthermore, the use of Mg [16–18], V [19, 20], Ta [21, 22], Cr [23], Nb [24] and rare earth elements such as La [25, 26] and Lu [27] is also reported in the literature. Wetting with the aid of active elements is associated with their enrichment at the substrate/solder interface, reaction with the substrate

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and consequently the formation of a reaction phase layer [28–31].

In order to ensure a reaction between the active elements and the substrate, activation of the solder alloy is necessary, e.g. by exposure to high temperature or by mechanical agitation utilizing ultrasound [12]. The processes of wetting, interfacial reactions and the formation mechanism of bonding between active solders and SiO₂ surfaces have been investigated by various authors, mainly using Ag–Cu–Ti [32–36] and Sn–Ag–Ti-based alloys at relatively high brazing/soldering temperatures [37] and with ultrasonic agitation of the molten solder [38, 39], respectively.

In the present work, we focus on active solder alloys on the basis of eutectic Ag–Bi, an alloy system with an equilibrium eutectic temperature of 262.5 °C [40]. The potential of Bi–Ag alloys as a substitute material for Pb-containing solders for high soft soldering temperature applications has been demonstrated by various authors [41–51]. Furthermore, Kolečák et al. [25] and Šuryová et al. [52] showed the possibility of joining ceramics (Al₂O₃ and SiC) with Cu using Bi–Ag-based alloys with small additions of La and Ti as active elements; these authors used ultrasound to enhance wetting during the soldering process. In the present study, Ti in combination with Mg were selected as reactive elements. Three Bi–Ag–Ti and Bi–Ag–Ti–Mg alloy compositions were investigated concerning their suitability for joining stainless steel 316L and SiO₂ glass. Interaction with the substrate surfaces was assessed via scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX).

Materials and methods

Based on studies of one of the present authors [41] on mechanical properties, wetting behaviour and thermal conductivity of Bi–Ag alloys as a function of Ag content, a starting composition of the binary alloy of Bi–12 wt.% Ag was selected. Different amounts of Ti are added as active element. The maximum Ti content was below 4 wt.% in order to avoid excessive formation of intermetallic compounds at the substrate/solder interface and to maintain a sufficiently low liquidus temperature of the solder alloy. Additionally, up to 1 wt.% Mg was added due its high reactivity with the SiO₂ substrate [53, 54]. Nominal

compositions of the solder alloys are displayed in Table 1.

The alloys were manufactured in a two-step process, considering that the high melting point of Ti combined with the large density differences between Ti and the other components may lead to inhomogeneity, oxidation and excessive evaporation. In a first step, an Ag–Ti alloy with a composition yielding the correct final Ti content in the solder was produced via inductive levitation melting using a cold wall crucible under Ar atmosphere. In a second step, the final solder alloys were induction melted in an Al₂O₃ crucible in air atmosphere using the Ag–Ti alloy as master alloy and adding Bi or a combination of Bi and Mg, respectively. The solder alloys in the as-cast state were ground with SiC abrasive paper up to a grit of 4000 # and polished with 3 and 1 µm polycrystalline diamond suspension. After metallographic preparation, microstructures were analysed by SEM in the backscattered electron (BSE) mode and EDX (EVO 40, Carl Zeiss, Germany).

SiO₂ glass and stainless steel 316L (Goodfellow Cambridge Limited, UK) were used as substrates. The composition of the steel substrate as determined by EDX is listed in Table 2.

Prior to the joining process, the substrate materials were laser-cut to a dimension of 10 × 10 × 2 mm, polished with 1 µm polycrystalline diamond suspension, cleaned in an ultrasonic bath with isopropanol, and rinsed with distilled water. The solder alloys were cut in plates with a size of 5 × 5 × 0.3 mm. (The smaller size of the solder plates was chosen so as to get some information of the spreading/wetting behaviour.) After ultrasonic cleaning with isopropanol, the plates were placed between the two substrate materials in an Al₂O₃ crucible and loaded with a piece of C45 steel (mass: 40 g) to ensure contact between the substrates and the molten solder. In order to avoid oxidation,

Table 1 Nominal solder compositions

	Bi (wt.%)	Ag (wt.%)	Ti (wt.%)	Mg (wt.%)
A1	bal	12	0.5	0
A2	bal	12	2	0
A3	bal	12	4	0
B1	bal	13	0.5	0.5
B2	bal	12	0.5	1
B3	bal	12	1	1

Table 2 Composition of 316L stainless steel substrate analysed by EDX measurement

Element	Fe (wt.%)	Cr (wt.%)	Ni (wt.%)	Mo (wt.%)	Mn (wt.%)
	bal	17.3	9.4	2.4	1.8

soldering was conducted under purified Ar atmosphere (< 2 ppm O_2 , H_2O) using a muffle furnace (LM-112, Lynn High Term, Germany) placed in a glove box. The stacked assembly steel/solder/ SiO_2 glass and the heat cycle curve used in the soldering experiments are displayed in Fig. 1. A joining temperature of $780^\circ C$ was chosen to achieve the necessary activity of Ti and Mg in the molten solder and consequently ensure a reaction with the substrates. Further increase in the soldering temperature led to crack formation in the SiO_2 glass after cooling, caused by thermally induced stress.

After the soldering experiments, the samples were mounted in epoxy resin with added Ni filler, and cross sections of the samples were metallographically prepared following the above-mentioned procedure. The interfaces in the SiO_2 glass/solder/steel assembly and the microstructures of the solder were analysed utilizing SEM in the BSE mode. Prior to SEM analysis, the samples were coated with carbon (S150B Sputter Coater, Edwards, UK) to avoid charging effects due to the poor conductivity of the SiO_2 glass. The chemical composition and element distribution were investigated by EDX (EVO 40, Carl Zeiss, Germany) using an acceleration voltage 20 kV.

Results

Solder microstructure in the as-cast state

Exemplary microstructures of the solder alloys are shown in Fig. 2. The different components of the microstructure are labelled with numbers, and the compositions measured by EDX as well as the attributed phases at the marked positions are listed in Table 3.

Primary fcc-(Ag) phase and Ag-Bi eutectic are observed in all alloys. All alloys also show Bi-Ti phases, the amount of which increases with increasing Ti content. XRD structural analysis was not carried out, but according to their composition these phases can be safely assumed to be Bi_2Ti and Bi_3Ti_2 , respectively, i.e. the two most Bi-rich equilibrium phases in the Bi-Ti system [55]. The presence of the Bi_3Ti_2 phase is only observed in alloys with a nominal Ti content of > 1 wt.% (alloys A2, A3 and B3).

Mg in the as-cast microstructure is completely (alloy B1) or partially (alloys B2 and B3) dissolved in the Ag phase. A maximum concentration of 11 at.% Mg was found in the fcc-(Ag) phase. This is in accordance with the large solubility of Mg in Ag [56]. Alloys B2 and B3 also exhibit the formation of an Ag, Mg and Bi containing phase. This phase does not correspond to a known phase and could not be identified.

Figure 1 Experimental set-up a, and exemplary heat cycle curve used in the soldering experiments b.

Figure 2 SEM images of Bi-12 wt.% Ag- x Ti ($x = 0.5, 2.0$ and 4.0 wt.%) (a, b, c), and Bi-12 wt.% Ag- x Ti- y Mg ($x = 0.5$ and 1.0 wt.%; $y = 0.5$ and 1.0 wt.%) in the as-cast state.

Interfaces of the SiO₂ glass/solder/steel

Except for alloy A3, a solid bond between SiO₂ and steel was achieved at the selected soldering temperature and time. Figures 3 and 4 display overview SEM images of cross sections of assemblies using alloys A2 and B3 as example. In addition, detailed images of the regions at the steel/solder and solder/SiO₂ glass interfaces and element distributions across the boundary measured by EDX are shown.

In alloy A2, a reaction of Ti and Mg and the formation of new phases at the glass/solder and solder/

steel interfaces was detected regardless of the solder composition. At the steel/solder interface, a discontinuous intermetallic layer is formed. A Ti concentration of 1 wt.% leads to a distinctly thicker intermetallic layer ($4.5\ \mu\text{m}$) as compared to that in the alloy with 0.5 wt.% ($1\ \mu\text{m}$). A further increase in the Ti concentration has only a minor influence on the thickness of the interface layer, but rather on the fraction of Bi-Ti phases in the solder. Irrespective of the Mg content of the alloy, at the steel/solder interfaces EDX analysis reveals no accumulation of Mg. An exemplary EDX line scan across the solder/

Table 3 Compositions of the solder alloys in the as-cast state as analysed by EDX: mean compositions of the marked areas of the microstructures (Fig. 2) and assigned phase(s)

Alloy	Position	Bi (at.%)	Ag (at.%)	Ti (at.%)	Mg (at.%)	Phase(s)
A1	1	94	6	0	0	Ag-Bi eutectic
	2	0	100	0	0	fcc-(Ag)
	3	74	0	26	0	Bi ₂ Ti
A2	1	94	6	0	0	Ag-Bi eutectic
	2	0	100	0	0	fcc-(Ag)
	3	58	0	42	0	Bi ₃ Ti ₂
A3	1	93	7	0	0	Ag-Bi eutectic
	2	0	100	0	0	fcc-(Ag)
	3	56	0	44	0	Bi ₃ Ti ₂
B1	1	94	6	0	0	Ag-Bi eutectic
	2	71	0	29	0	Bi ₂ Ti
	3	0	93	0	7	fcc-(Ag)
B2	1	95	5	0	0	Ag-Bi eutectic
	2	0	89	0	11	fcc-(Ag)
	3	71	0	29	0	Bi ₂ Ti
	4	33	47	0	20	unknown
B3	1	94	6	0	0	Ag-Bi eutectic
	2	0	89	0	11	fcc-(Ag)
	3	32	44	0	24	unknown
	4	56	0	44	0	Bi ₃ Ti ₂

Figure 3 SEM images and EDX profiles of the joint assembly using alloy A2, overview image **a**, high magnification image of the steel/solder interface **b** and high magnification image of the solder/

SiO₂ glass interface **c**; element distributions along the lines A-B **d**, and C-D **e**.

steel interface using alloy B3 is shown in Fig. 4d. All six solder alloys display an intermetallic phase at the steel/solder interface containing Bi, Fe and Ti (Figs. 3b and 4b) with concentrations, measured by EDX, of 33, 12 and 55 at.%, respectively. Occasionally,

the occurrence of additional Ti-containing phases near the interface was observed.

At the solder/SiO₂ glass interface, the solder alloys without Mg addition (Fig. 3c, e), show the formation of phases containing Ti, Si, O and Bi. The layer thickness is < 1 μm in all cases.

Figure 4 SEM images and EDX profiles of the joint assembly using alloy B3, overview image **a**, high magnification image of the steel/solder interface **b** and high magnification image of the solder/

SiO₂ glass interface **c**; element distributions along the lines A-B **d**, and C-D **e**.

The Mg containing solder alloys show a significantly different microstructure in the vicinity of the solder/SiO₂ glass interface (Fig. 4c, e) as compared to their Mg-free counterparts. Next to the glass substrate, a distinct increase in the Mg concentration is detected. In the adjacent region (up to a distance of 5 μm from the SiO₂ glass), the occurrence of a Ti-rich phase was detected. This phase does not form a continuous layer, but is rather present as small, partially interconnected particles in a Bi-Matrix. The amount of this scattered phase increases with increasing Ti concentration.

Discussion

Bonding of SiO₂ glass to 316L stainless steel could be achieved with the alloys A1, A2, B1, B2, B3 at a soldering temperature of 780 °C. At the steel/solder interface, the formation of a new phase was observed. No enrichment of Mg at the steel/solder interface was detected, and mainly Ti is responsible for the bonding with the steel. The evolution of the microstructure in the interfacial region can be attributed to a multi-stage process: after melting, in a first step interaction of Ti with the steel (e.g. adsorption of Ti at the interface [28]) leads to a concentration gradient in the solder and diffusion of Ti

towards the interface, changing the activity [57] and the chemical potential of all species in the vicinity of the interface. In the second step, when a sufficient amount of Ti is present at the interface, new phases form. EDX measurements indicate the formation of Bi₂FeTi₄, one of the ternary equilibrium phases in the Bi-Fe-Ti system [58, 59]. Bi₂FeTi₄ was found in the solder mainly close to the steel/solder interface. Bi₂FeTi₄ particles may break off the reaction layer and are also found inside the solder. Considering that there is no enrichment in Mg at the steel/solder interface, the addition of Mg does not appear to play a role for the bonding with the steel. Prior to the soldering process, the Ti in the as-cast state is mainly found in the Bi-Ti phases. For Ti contents > 1 wt.%, large particles of Bi-Ti phases are found in the cross section of the joint, indicating incomplete melting of these intermetallic phases during the soldering. Thus, a part of the Ti is not available to enhance the bonding. Similar results have been reported by Li et al. [60] concerning the reaction layer thickness during brazing of SiC using a Sn–Ag–Cu–Ti alloy. The authors observed a decrease in the reaction layer thickness with Ti contents > 1.5 wt.%, which is attributed to the presence of Ti–Sn phases. An increase in solid fraction during soldering causes an increase in the apparent viscosity of the melt, which counteracts good wetting behaviour [60, 61]. The lack

of bonding for alloy A3 may be attributed to this effect. In addition, coarse particles of intermetallic phases in the solder and a thick reaction layer at the steel/solder interface are expected to be disadvantageous for the mechanical properties of the assembly. The results in the present work indicate that the maximum Ti content in the Bi-Ag solder should be in the order of 0.5 wt.%.

In contrast to the steel/solder interface, at the solder/SiO₂ glass interface a clear accumulation of Mg is observed, and an influence of the addition of Mg on the microstructure in the vicinity of the interface is evident. Without Mg additions, the reaction of Ti with the SiO₂ glass substrate could also lead to bonding. According to the literature on the reaction of Ti containing solder and brazing alloys on SiO₂ substrates, different reaction products may form. For Ag–Cu–Ti brazes, the formation of TiSi₂ and Ti₄O₇ [33], Ti₂O and Ti₅Si₃ [32] and TiSi₂, Ti₃O₅ and Ti₂O₃ [34] has been reported. Similarly, Ti₅Si₃ and TiO at have been observed using a Sn–Ag–Cu–Ti solder alloy [37]. It is assumed that the Ti containing reaction phases at the solder/SiO₂ glass interface in the present work can be assigned to one or more of these phases. A conclusive determination was not attempted due to their small size and due to the fact that Ti appears not to be responsible for the bonding.

The reaction of solid and liquid Mg with SiO₂ glass has been investigated by several authors, documenting the formation of MgO and Mg₂Si [54, 62–65]. Again, the formation of the new phase may occur in a two-step process [65]: in the first step, SiO₂ and Mg react to a MgO layer at the interface, and the released Si reacts with Mg to Mg₂Si.

Depending on the amount of added Mg, the released Si may also react with the Ti in the solder to form Si–Ti intermetallic phases. However, also in this case a reliable phase identification is not possible with reasonable effort. Fu et al. [66] investigated the wetting of Sn–Ti alloys on SiC and reported the formation of TiC and Ti₅Si₃ with a similar dispersion, attributed to a sequential reaction process. These authors also noted an increased fraction of the intermetallic phases with increasing Ti content.

Considering the properties of the assembly, the formation of reaction layers at both interfaces is crucial for generating strong bonding. An increase in shear strength with a larger fraction of reaction products at the interface has been observed [38, 67]. However, due to the brittle nature of the intermetallic

phases and due to differences in the coefficient of thermal expansion between SiO₂ glass and the reaction products, residual stresses at the interface are intensified with increasing layer thickness, potentially leading to crack formation and delamination. The addition of Mg up to 1 wt.% to the solder alloy leads to a thin reaction layer with the SiO₂ and appropriate bonding.

Based on the present experiments, it can be concluded that the combination of the active elements Ti and Mg to the Bi-Ag solder alloy is suitable for joining SiO₂ glass and 316L stainless steel, with Ti reacting primarily at the steel/solder interface and Mg at the solder/SiO₂ glass interface.

Conclusion

Joining stainless steel 316L to SiO₂ glass was realized via active soldering utilizing Bi-Ag-Ti and Bi-Ag-Ti-Mg alloys with varying Ti and Mg contents. The as-cast microstructure of the solder alloys and the microstructures of cross sections of the steel/solder/SiO₂ glass assembly after soldering at 780 °C for 10 min in a high purity Ar atmosphere were assessed by SEM and EDX analysis, leading to the following statements:

1. Ti is present in Bi-Ti intermetallic phases in the as-cast alloys, and Mg is dissolved in the fcc-(Ag) phase. For Mg concentrations > 1 wt.%, additionally an Ag-Bi-Mg containing phase is formed.
2. The formation of a Fe-Ti-Bi-containing reaction layer is observed at the steel/solder interface. An increasing Ti content from 0.5 to 1 wt.% leads to an increase in the layer thickness. Further Ti addition has no significant influence on layer thickness. An influence of Mg at the solder/steel interface is not visible.
3. At the solder/SiO₂ glass interface, the formation of Ti-rich reaction products was observed. Mg accumulates at the solder/SiO₂ glass interface and forms intermetallic phases. The formation of finely dispersed Ti-containing phases was also observed.

Adding both Ti and Mg as active elements to Bi-Ag-solders allows joining of challenging material combinations such as 316L stainless steel and SiO₂ glass without the need for flux and prior metallization. Thus, a post-joining cleaning process is not

necessary, and contamination of the base materials resulting from the fluxes is avoided.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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