

# Joining of Soda Lime Silicate Glass TO Ti6Al4V Alloy in Air by Controlled Heat Treatments

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Soda lime silicate glass was joined to Ti6Al4V alloy in air by heat treatment at 800 °C for 10, 20 and 30 min and by subsequent cooling to room temperature with a controlled cooling rate. Stresses, due to thermal expansion mismatch, have developed at glass-metal interface after high temperature joining and cooling down to room temperature. The finite element calculations, performed using ANSYS 14 software, suggested that the glass-metal interface was experiencing rather low maximal and minimal principal stresses due to joining. Highest maximal principal stress level was about 25 MPa and lowest minimal principal stress level was about -40 MPa for used sandwich sample profiles. Heat treatment duration affected tensile bonding strength and interfacial reaction between the glass and the Ti alloy. Scanning electron microscope analysis of glass-alloy joining interface showed that a secondary K, Ca, Ti rich sodium silicate phase was forming in glass, starting at glass alloy interface and that it was growing with the increase in heat treatment duration. Low joining stress levels at the glass-alloy interface for used sandwich sample profile and good bonding between the soda lime silicate glass and Ti6Al4V alloy resulted in creation of successful soda lime silicate glass-Ti6Al4V joints.

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## 1. Introduction

Soda lime silicate glass is one of the economically available glasses which is used often for outdoor applications such as windows [1]. Ti6Al4V alloy is one of the light weight alloys and it is used in some of applications requiring good corrosion resistance, such as biomedical dental applications [2]. Glass-metal joining is needed for wide variety of applications, but it is especially important for heat collecting units, used in parabolic sunlight collector systems [3]. To lower the heat losses, a glass tube sealing of the alloy tube, carrying heated fluid is required. To achieve this, a good glass-metal alloy joining method is necessary. Borosilicate glass-Kovar alloy (having composition of 54 wt.%Fe – 29 wt.%Ni – 17 wt.%Co) joining is generally used in these applications, due to their closely matched thermal expansion coefficients and good bonding [4]. If soda lime silicate glass-Ti6Al4V alloy joints could be successfully made, they can find uses in such heat collecting tube applications as well. In literature, there are some earlier studies on joining of SiO<sub>2</sub> glass to Ti6Al4V alloys, based on vacuum brazing techniques, employing AgTiCu and AgTi/Ni layers [5, 6]. However, there are not many earlier studies aiming for joining of soda lime silicate glass to Ti6Al4V alloy. This study provides (i) a heat treatment procedure for successful joining of soda lime silicate glass-Ti6Al4V alloy in air, (ii) calculations employing finite element method, of maximal and minimal principal stresses at joining interface, due to thermal expansion coefficient mismatch, (iii) results

of tensile bonding strength measurement of glass-metal sandwich structure sample profiles, as a function of heat treatment duration, and (iv) investigation of the glass-Ti6Al4V joining interface by the scanning electron microscope analysis.

## 2. Experimental procedure

Soda lime silicate glass having chemical composition of 69 wt.% SiO<sub>2</sub>, 1 wt.% B<sub>2</sub>O<sub>3</sub>, 3 wt.% K<sub>2</sub>O, 4 wt.% Al<sub>2</sub>O<sub>3</sub>, 13 wt.% Na<sub>2</sub>O, 2 wt.% BaO, 5 wt.% CaO and 3 wt.% MgO (Schott Ar glass) and Ti6Al4V alloy, having chemical composition of 5.9 wt.% Al, 4 wt.% V, 90.1 wt.% Ti (fulfilling ASTM F136 specification) in its annealed form, were used in the experiments. Samples of required sizes were slowly cut from supplied rods using a diamond saw. The cut pieces were then grinded employing metallographic grinder with a 1000 grade SiC grinding paper to remove any cutting marks. Two sets of joining samples were prepared: Ti6Al4V (having 12 mm diameter and 22 mm height) – soda lime silicate glass (having 10 mm diameter and 2 mm height) – Ti6Al4V (having 12 mm diameter and 21 mm height), for tensile bonding strength measurements and soda lime silicate glass (having 10 mm diameter, 2 mm height) – Ti6Al4V (having 12 mm diameter, 3 mm height) for controlling interfacial reactions. Samples were joined by inserting them directly into the furnace preheated at 800 °C and keeping for the duration of heat treatment of 10, 20 and 30 min in air. The joined samples were then cooled down to 550 °C during 20 min, and kept for 20 min at 550 °C for thermal annealing of glass, to remove any prior developed stress. The joined sample was cooled down from 550 °C to room temperature slowly, during 4 hrs.

Calculations of the maximal and minimal principal stresses for the used sandwich sample profile were done

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Material properties used in calculations of joining stress with ANSYS 14 software. TABLE I

Material property	Soda lime silicate glass	Ti6Al4V alloy
Therm. expansion coef.	$9.1 \times 10^{-6}$ [7]	$9.7 \times 10^{-6}$ [8]
Young's modulus ( $E$ ) [Pa]	$7.3 \times 10^{10}$ [7]	$1.14 \times 10^{11}$ [8]
Poisson ratio ( $\nu$ )	0.22 [7]	0.33 [8]

employing ANSYS 14 finite element method software. Table I provides details of the material properties used in the models. Calculations were done assuming that joining stresses were resulted due to cooling down of the bonded glass-alloy joints from the thermal stress relieving annealing step at 550 °C to room temperature.

Tensile bonding strength measurements of Ti6Al4V alloy – soda lime silicate glass – Ti6Al4V alloy sandwich sample profiles were done by attaching joined samples to Instron Satec Tm Series model tensile tester and applying tensile loading to produce 0.1 mm/min extension rate. Maximal tensile stress levels leading to rupture of bonded samples are reported.

Cross section samples of the soda lime silicate glass-Ti6Al4V alloy, disc on disc samples, were prepared by first placing the joined samples into a polymer based mold, setting at room temperature, then by slowly cutting the molded sample using a diamond saw, to reveal interface between glass and alloy, and finally by grinding the cross sectioned profile by employing the metallographic grinder with a 1000 grade SiC grinding paper, to reveal joined interface more clearly. Cross sections of the samples were slightly covered by sputtering of Au to prevent any charging effects prior to scanning electron microscope investigation. Interfacial reactions between the glass and the alloy and the chemical compositions of reaction products were analyzed employing JEOL 6060 model scanning electron microscope with energy dispersive spectrometer with 20 kV acceleration voltage setting.

### 3. Results and discussion

Figure 1 shows the Ti6Al4V – soda lime silicate glass – Ti6Al4V sandwich and Ti6Al4V – soda lime silicate glass disc on disc samples, joined at 800 °C in air for 10, 20 and 30 min. All of the samples were successfully joined and there were no sign of macro cracking in the joined glass layers.

Figure 2 shows maximal and minimal principal stress levels calculated for Ti6Al4V – soda lime silicate glass – Ti6Al4V sandwich sample profiles using ANSYS 14 software. Ti6Al4V side of the glass-alloy joining interface had the highest maximal stress levels of about 25 MPa. The joined glass had a slightly lower maximal principle stress levels of 20 MPa in its outer circumference. The minimal principle stress levels were slightly bigger for the soda lime silicate glass, of about –40 MPa at the circumference close to the joining interface. The Ti6Al4V alloy had lower minimal principal stress levels

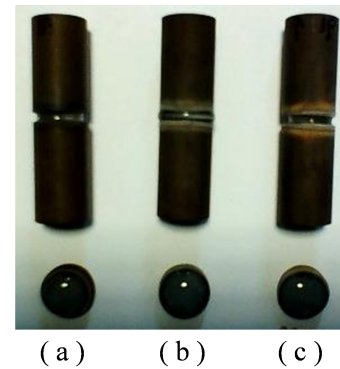


Fig. 1. Ti6Al4V – soda lime silicate glass – Ti6Al4V sandwich samples and soda lime silicate glass disc on disc samples, joined at 800 °C in air, for heat treatment durations of (a) 10, (b) 20 and (c) 30 minutes.

of about –10 MPa close to joining interface. Calculation results suggest that both soda lime silicate glass and Ti6Al4V had relatively low maximal and minimal principle stress levels after the high temperature joining and the cooling down to room temperature.

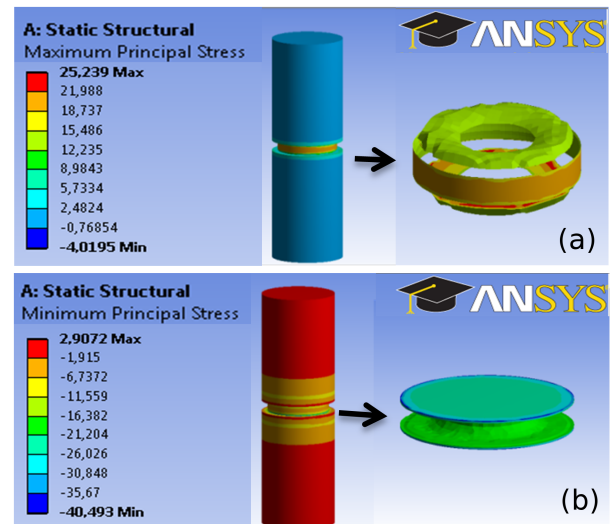


Fig. 2. Results of finite element method calculation of maximal and minimal principal stress levels for Ti6Al4V – soda lime silicate glass – Ti6Al4V sandwich sample profiles. (a) Maximal principal stress levels for whole sample and regions having stress levels higher than 15 MPa. (b) Minimal principal stress levels for whole sample and regions having stress levels lower than –20 MPa.

Table II shows tensile bonding strength observed for sandwich sample profiles as a function of heat treatment duration at 800 °C in air. Tensile bonding strength was 19 MPa for sample heat treated for 10 min and has increased slightly to a level of 27 MPa for sample heat treated for 20 min and remained at a similar level of 28 MPa for 30 min heat treatment. Increase in heat treatment duration affected both the fracture modes and the bonding strength of the joined samples. While the

sample heat treated for 10 min had fractured mostly on the Ti6Al4V – soda lime silicate glass bonding interface, the sample heat treated for 20 min had fractured within glass layer and the sample heat treated for 30 min had fractured in a complex manner, having regions within both the glass and Ti6Al4V interface. A slight increase in bonding strength with the duration of heat treatment suggested that some interfacial reaction was needed for better bonding between soda lime silicate glass and Ti6Al4V alloy.

TABLE II  
Tensile bonding strengths of samples heat treated at 800 °C.

Treatment time [min]	Tensile bonding strength [MPa]
10	19
20	27
30	28

Figures 3 and 4 show scanning electron microscope images and energy dispersive spectroscopic analysis (EDS) of sample heat treated for 10 min at 800 °C. Low magnification secondary electron SEM image (100×) suggested that soda lime silicate glass bonded to Ti6Al4V alloy without any micro cracking at the glass-alloy interface. Higher magnification (1500×) revealed that a secondary phase was forming in glass region starting from the Ti6Al4V – glass interface. EDS spot analysis taken from interior of glass (spot 1) and the new forming phase (spot 2) suggested that the secondary phase was richer in K, Ca and Ti content compared to used soda lime silicate glass.

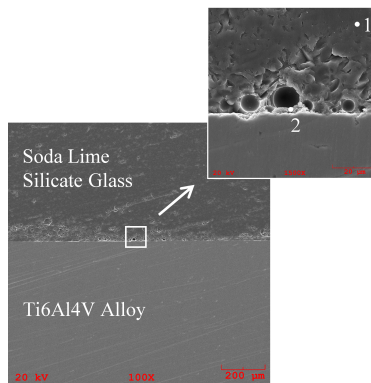


Fig. 3. Scanning electron microscope images of soda lime silicate glass – Ti6Al4V alloy junction sample, heat treated for 10 min at 800 °C. 100× and 1500× magnified images were taken using secondary electron mode at 20 kV acceleration voltage. 1500× magnification image also shows spot positions of EDS analysis.

Figure 5 and 6 show scanning electron microscope images of 20 min and 30 min heat treated samples at 800 °C. Increase in heat treatment duration lead to more interfacial reactions and further growth of secondary phase. Spot EDS analysis of 30 min heat treated sample (Fig. 7) suggested that chemical composition of secondary phase remained richer in K, Ca and Ti content during its growth.

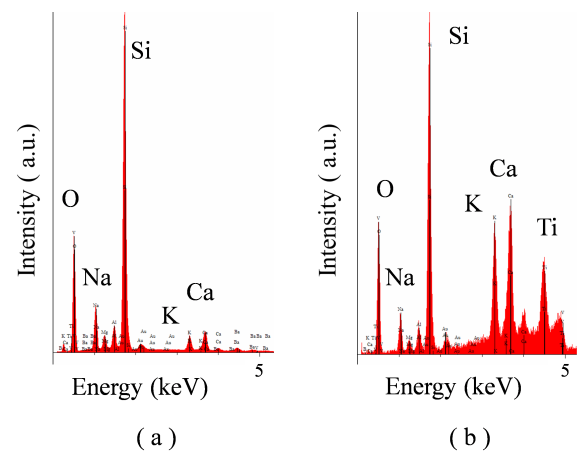


Fig. 4. Energy dispersive spectroscopic analysis of sample heat treated for 10 min at 800 °C: (a) interior of soda lime silicate glass (spot position 1, shown in figure 3), (b) secondary phase, formed at glass-alloy interface (spot position 2, shown in figure 3).

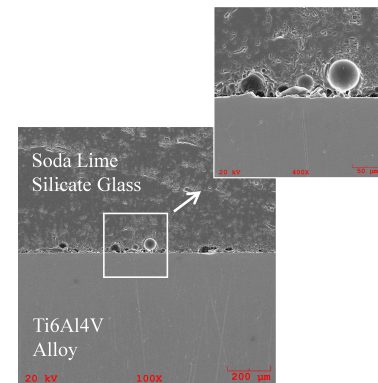


Fig. 5. Scanning electron microscope images of soda lime silicate glass – Ti6Al4V alloy junction sample heat treated for 20 min at 800 °C. 100× and 400× magnified images were taken using secondary electron mode at 20 kV acceleration voltage.

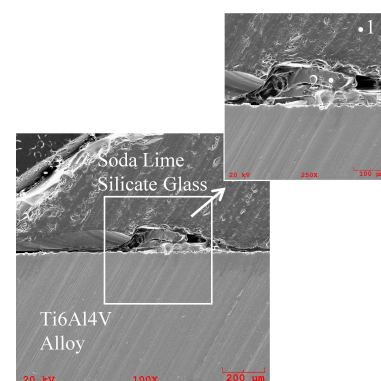


Fig. 6. Scanning electron microscope images of soda lime silicate glass – Ti6Al4V alloy junction sample heat treated for 30 min at 800 °C. 100× and 250× magnified images were taken using secondary electron mode at 20 kV acceleration voltage. 250× magnification image also shows spot positions of EDS analysis.

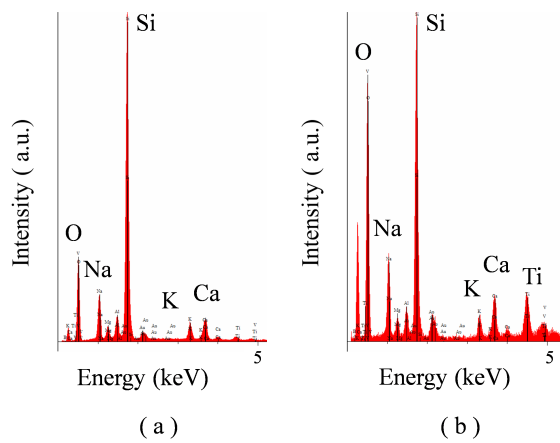


Fig. 7. Energy dispersive spectroscopic analysis of sample heat treated for 30 min at 800 °C: (a) interior of soda lime silicate glass (spot position 1, shown in figure 6), (b) secondary phase formed at glass-alloy interface (spot position 2, shown in figure 6).

Formation and growth of the K, Ca, Ti rich sodium silicate-based secondary phase at soda lime silicate glass – Ti6Al4V interface thought to be related to initial oxide layer formation of Ti6Al4V alloy in air at 800 °C, Ti diffusion into the glass from Ti6Al4V alloy, nucleation of secondary phase from initially formed oxide layer of Ti6Al4V alloy and its growth within the glass with the increase in heat treatment duration. More studies are needed to analyze all these effects clearly in more detail but not scope of this study.

#### 4. Conclusions

Soda lime silicate glass was joined successfully to Ti6Al4V alloy by heat treating at 800 °C in air for 10, 20 and 30 minutes. Having low joining stresses and good interfacial bonding were found to be producing successful joints. Based on finite element method calculations, joined parts were subjected to low maximal and minimal principle stress levels. For the used sandwich sample profile, highest maximal principal stress of about 25 MPa was calculated for

Ti6Al4V side of the glass – alloy joining interface and lowest minimal principal stress about –40 MPa was calculated for the soda lime silicate glass at its circumference, close to joining interface. Tensile bonding strength of glass – Ti alloy joint was dependent on heat treatment duration and increased from 20 MPa for sample heat treated for 10 min, to 30 MPa for samples heat treated for 20 and 30 min, suggesting some interfacial reaction was needed to improve bonding strength. Scanning electron microscope investigation showed that a secondary phase rich in K, Ca and Ti content, compared to the content of the used glass, was forming at glass – Ti alloy interface for sample heat treated for 10 min and growing further with the increase in heat treatment duration to 20 and 30 min. Low joining stress levels due to similar thermal expansion coefficients and good interfacial bonding was concluded to be causing successful soda lime silicate glass – Ti6Al4V alloy joints in air.

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