

# Development and interface characterization of unmatched glass-metal joint

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## ABSTRACT

An unmatched glass-metal joint between borosilicate glass and austenitic stainless steel SS 304 was developed for parabolic trough receiver tube application.  $40\text{SiO}_2\text{-}54\text{B}_2\text{O}_3\text{-}4\text{Na}_2\text{O-}2\text{Al}_2\text{O}_3$  material was used as the glass sealant to join the SS304 metal ring and borosilicate glass tube. The research work reported was carried out with objective of investigating the role of glass sealant, joint design and pre-oxidation conditions on the wettability and adhesion strength of the glass-metal joint. The work also aimed at analysing the types of oxides formed at the glass-metal joint interface. The pre-oxidation behavior of SS304 was studied by varying the pre-oxidation temperature and pre-oxidation time. The oxide formed on the surface of SS304 substrates were examined using x-ray diffraction and energy dispersive x-ray spectroscopy. During the glass-metal sealing process the oxides at the glass-metal interface were characterized using x-ray diffraction and scanning electron microscopy. In-situ wetting behavior of glass sealant  $40\text{SiO}_2\text{-}54\text{B}_2\text{O}_3\text{-}4\text{Na}_2\text{O-}2\text{Al}_2\text{O}_3$  over austenitic stainless steel SS 304 was examined using the in-house developed image acquisition system. The optimum condition for joining  $40\text{SiO}_2\text{-}54\text{B}_2\text{O}_3\text{-}4\text{Na}_2\text{O-}2\text{Al}_2\text{O}_3$  glass sealant with SS304 is obtained by pre-oxidizing metal specimen at  $700^\circ\text{C}$  for 3 min, where optimum oxide layer mass gain of  $5.8666 \times 10^{-04} \text{ g/cm}^2$  and optimum oxide layer thickness of  $87.57 \mu\text{m}$  was achieved. The maximum strength of joint during adhesion test was measured around  $2.57 \text{ N/mm}^2$  (MPa) corresponding to pre-oxidation temperature of  $700^\circ\text{C}$  and 3 min pre-oxidation time. The FeO and  $(\text{SiO}_2, \text{AlNa}(\text{SiO}_4))$  oxide phases were observed at the interface of glass and the metal during joining. During in-situ wettability analysis the contact angle between the glass sealant pallet and SS304 substrate was measured at different temperatures. The contact angle of the glass sealant pallet decreased during heating process and the contact angle increased as the glass sealant pallet cooled from sealing temperature.

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## 1. Introduction

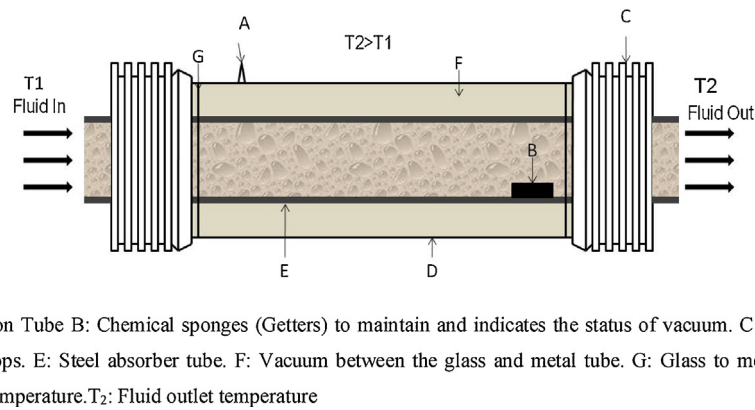
The joining of glass with metal remains a challenge because of the amorphous and crystalline nature of glass and metal respectively. Glass to metal joint has been used for many applications such as vacuum tube, parabolic trough receiver tube, solid oxide fuel cell, and electronic semiconductor applications [1,2]. The glass metal joint can be broadly classified into two categories, viz., matched glass metal joint (the coefficient of thermal expansion is same or closely matched for glass and metal) unmatched glass metal joint (coefficient of thermal expansion is significantly different for glass and the metal). Glass-metal joining is traditionally a fusion tech-

nique, in which glass is melted over the metal surface and reacts to forms an interface. The bonding of glass with the metal mainly depends upon the type of oxide present at the metal surface after pre-oxidation. The oxide formed on the metal surface after pre-oxidation acts as a bonding agent at the interface between the glass and the metal. The properties and the types of oxides formed at the interface affect the bonding of the glass with the metal [1].

It is also recognized that the residual stresses generated due to difference in coefficient of thermal expansion of the glass and the metal must be kept as low as possible [2]. The generated thermal residual stress at glass-metal interface breaks the glass tube at the interface [3–6]. The breaking of the glass at the glass-metal interface leads to the loss of vacuum in the parabolic trough receiver tube. A Parabolic Trough Receiver Tube (PTRT) consists of steel pipe surrounded by transparent glass tube and the space between them consisting of vacuum. The steel pipe is coated with a solar selective coating which is responsible for absorbing maximum solar radia-

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**Fig. 1.** The geometry of parabolic trough receiver tube.

A: Evacuation Tube B: Chemical sponges (Getters) to maintain and indicates the status of vacuum. C: Bellow. D: Glass envelops. E: Steel absorber tube. F: Vacuum between the glass and metal tube. G: Glass to metal joint. T<sub>1</sub>: Fluid inlet temperature. T<sub>2</sub>: Fluid outlet temperature.

tion and minimizing reflection and radiation losses as suggested by Barriga et al. [7]. The working fluid temperature is around 265 °C as reported in the literature [8,9]. The geometry of a parabolic trough receiver tube is shown in Fig. 1.

From the literature [4–6,10,11–13], it has been noted that kovar alloy (ASTM F-15 alloy) having a coefficient of thermal expansion  $5.0 \times 10^{-6}$  m/m°C and kodial glass with a coefficient of thermal expansion  $3.3 \times 10^{-6}$  m/m°C has been used for the development of a matched glass-metal joint for parabolic trough receiver tube [10]. The pre-oxidation behavior of kovar alloy has been studied by varying the pre-oxidation time and the pre-oxidation temperature along with different environment by Cheng-hsien and Pi-ying Cheng [11]. Wettability study of different glasses over kovar alloy has been studied using image capturing system by Chenghsien Kuo et al. [12]. The experimental results indicate that the pre-oxidation of kovar alloy for about 10 min at 700 °C provides better adherence of glass with kovar [12]. FeO and Fe<sub>3</sub>O<sub>4</sub> types of oxides were formed after pre-oxidation of kovar alloy as reported by D.W. Luo and Z.S. Shen [10].

Studies on glass-metal joining for parabolic trough receiver tube have been conducted using kovar alloy and kodial glass by researchers [4–6]. Wettability study of glass over kovar alloy has also been carried out at joining temperature of 980 °C by Chenghsien Kuo et al. [12].

The aim of the present work was to fabricate unmatched (coefficient of thermal expansion of both glass and metal differ significantly) glass-metal joint between SS304 alloy having coefficient of thermal expansion ( $17.3 \times 10^{-6}$  m/m°C) and borosilicate glass with coefficient of thermal expansion ( $3.3 \times 10^{-6}$  m/m°C). The selection of SS 304 alloy and the borosilicate glass for the present study is based on their ease of availability and their lesser cost as compared to the kovar alloy and the kodial glass. Glass sealant was used to fill the space between borosilicate glass tube and the SS304 metal ring. Glass sealant composition was selected on the basis of the shear strength tests. Localized heating was used through induction heating process to the join glass-metal assembly. Pre-oxidation behavior of the austenitic stainless steel SS304 alloy specimen was studied by varying pre-oxidation temperature and pre-oxidation time. The types of oxides formed after pre-oxidation were examined using x-ray diffraction (XRD) technique. The morphology of the glass-metal joint interface was analyzed with the help of scanning electron microscope and the X-ray dot mapping technique. In situ wetting behavior of glass sealant over the austenitic stainless steel alloy SS304 was analysed at different temperatures by using an image capturing system. The adhesive strength of the glass with

the metal was measured using specially designed specimen holding fixture used for testing under compression loading condition.

## 2. Experimental detail

### 2.1. Material selection

In the past, researchers [4–6] have used kovar alloy and kodial glass combination for glass-metal sealing application because of their closely matched coefficient of thermal expansion. In the present work borosilicate glass tube and SS304 metal rings were joined. The major issue involving the development of glass metal joint is the formation of suitable bonding between the glass sealant and the glass tube on one side and glass sealant and metal ring on the other side. The problem is complicated due to the differential expansion of the glass sealant and the metal ring during heating due to a significant gap is formed between the metal ring and the glass sealant. This gap closes during cooling as metal ring shrinks much more as compared to glass sealant when cooled to room temperature. The gap between the metal ring and glass tube (where the glass sealant is filled) depends upon the initial design selection (geometric dimensions) of the metal ring and the glass tube dimensions. The larger initial gap would result in debonding of glass sealant with the metal ring as the metal ring would expand much more in heated condition forcing the glass sealant to leak out. The smaller initial gap would limit the strength of the joint and can cause breakage of the glass tube. Hence the selection of the suitable glass tube-metal ring joint design is very important issue to obtain a suitable compression joint. The selection of the suitable glass sealant is also a major issue as the coefficient of thermal expansion mismatch between the glass sealant and the metal ring helps in increasing the development of compressive stresses on the glass sealant and prevents the breakage of glass tube due to absence of any direct compressive stresses on the glass tube. It is necessary to have a glass sealant with lower melting point than glass tube and have high wettability with the metal ring. The compressive strength of glass is larger than its tensile strength therefore compressive stresses generated on glass tube and glass sealant after fabrication of glass-metal joint helps to resist high mechanical stresses in glass-metal joint.

#### 2.1.1. Selection of glass-metal joint design

The experimental procedure for adopting a suitable glass-metal joint design is shown in Fig. 2. Initially the glass tube inner surface was placed over the outer surface of metal ring as shown in Fig. 2(a). The complete assembly was then heated locally to 980 °C temperature with the help of an induction heating set-up.

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