

# Fracture Surface Analysis of Borosilicate Bonded Stainless Steel

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## ABSTRACT

Borosilicate glass was bonded to the stainless steel (st.st.) grade 316 by using a technique of oxides growth considered as active filler. Contact angle of oxide to the stainless steel was found 47°. The bonding strength achieved under ambient condition enriched with oxygen, and is equal to 4 MPa. The good bonding was correlated with new structural phases at the intermediate layer. The bubbles size and their amount were found as important factors play a significant role in bonding borosilicate and stainless steel.

## الخلاصة

تم ربط زجاج البوروسيليكات بالفولاذ صنف 316 باستخدام تقنية انماء الاوكسيد الذي يعتبر بدوره حشوة مألثة فعالة. قيست زاوية التماس والتي تعرف بزواوية يونك بين طبقة الاوكسيد المألثة و سطح الفولاذ وكانت تساوي 47 درجة. تم الحصول على قوة ربط الزجاج بالفولاذ تحت الظروف الاعتيادية والمشبعه بالاكسجين فكانت تساوي 4 ميكا باسكال. عزيت قوة الترابط بين الجزئين الملتحمين الى نشوء اطوار تركيبية جديدة عند منطقة الربط البينية. وقد وجد ان حجوم الفقاعات المتولدة وكمياتها كانت من العوامل المهمة التي لعبت دورا اساسيا في عملية ربط البوروسيليكات بالفولاذ.

## INTRODUCTION

The major reason for joining is to permit dissimilar materials to be used in complex structures or assembly creating so-called hybrid structures, dissimilar materials enable the achievement of functions where design requirements properties unobtainable in single materials.

Joining metal to glass or ceramic is considered as one of the most promising dissimilar materials because of its low-cost and readily availability[1].

Many researchers have sought to modify metal-glass (or ceramic) joint techniques, A. Elrefaey et.al [1] used anodic bonding of glass- titanium and glass-steel with tin based filler, they used share test to investigate the joint strength, they noticed that the fracture happened along the glass-solder interface. Chichiro Iwamoto et.al [2] studied the interface between Al and Mo coated glass substrate by ultrasonic welding, no intermetallic compound were observed, dislocation were produced, Al and Mo lattices were directly conducted at the interface. M.Mazar Atabaki [3] studied the effect of thermal expansion on the joint strength by using metallic foam as buffer layer between metal and ceramic to avoid thermal expansion mismatch between the two materials when bonding together by brazing.

A very large number of metals and alloys such as stainless steel have been bonded into glass such as borosilicate in manufacturing various types of fields apparatus [4]. Each of glasses type should have special thermal expansion characteristics for bonding or sealing to certain metal or alloy [5]. Matched bonds should be used when glasses and alloys of closely matching thermal expansion (10% differences) characteristics are to be bond [6, 7]. The stresses developed in the glass are minimized by the deformation of the metal (elastic or plastic) [1]. Wettability studies the measurement of

contact angles which indicates the degree of wetting when a solid and liquid interacts. Small contact angle less than 90° correspond to high wettability, otherwise large contact angle more than 90° correspond to low wettability [1]. There are many factors affecting on groups of oxides growth that may be found in intermediate layer. The bond in glass (borosilicate) – alloy (stainless steel) bonds is based on the direct borosilicate–stainless steel adhesion.

The problem of glass to metal bond by indirect adhesion is a problem of oxidation growth on the metal surface. The oxidation of metals or alloys follows different rate laws depending on the specific of each of them, properties of oxides growth and the temperature. Accordingly, the dependence of oxidation reaction rate upon temperature is expressed by Arrhenius equation as following [1]:

$$K = A \exp -E_a / RT \quad (1)$$

Where K = oxidation reaction rate constant at temperature T, A = real of oxidation area depends upon K, E<sub>a</sub> = activation energy of the metal or alloy, R = gas constant.

In accordance with this equation, activation energy of the metal oxide is determined by measuring the oxidation reaction rate constant at different temperatures. The second parameter that plays an important role in adhesion assembly is the contact angle which is given by the Young equation as following [1]:

$$S_p = \gamma_{SA} - (\gamma_{SL} + \gamma_{LA}) \quad (2)$$

Where S<sub>p</sub> denoted the spreading coefficient, γ<sub>SA</sub> denoted the measure of the tension when a solid air interface is

replaced by solid–liquid ( $\gamma_{SL}$ ) and liquid–air ( $\gamma_{LA}$ ) interface.

The good wetting is that a molten glass be able to dissolve or to diffuse into the metal surface on which it flows.

The objective of the currently work is to study the oxidation behavior at different temperatures and to study the oxidation growth and then followed the bonding strength as a result of good oxidation growth.

**MATERIALS AND METHODS**

Borosilicate glass was selected as the first pieces to be joined to the stainless steel (st.st.) of grade 316. Borosilicate is a corning glass number 7052 designated by ASTM F105 [4]. Borosilicate powdered glass was selected as an active filler corresponding bonds to the base material to be joined. Stainless steel is a commercially available alloy is enriched by the elements Cr, Ni, and Mo. Borosilicate powdered glass obtained by crushing a clean piece of glass in a mill ball machine with different particle sizes (0.38 – 0.75) $\mu\text{m}$ . Decontamination processes were achieved on the pieces to be joined according to the standard reference [1, 7]. Grinding and polishing techniques were performed by using 300 grad metallographic paper with smooth clothes and by treated with the chemical agent as mentioned in reference [8,9]. Oxides growth was carried out in an electrically tube furnace with programmable controller Eurotherm-2116, UK made. This furnace provided with K-type thermo couple. Oxidation temperature was selected in the range (873-1173) °K according to the temperature–time schedule as in Figure 1. Holding time was examined between 30 to 60 minutes. The oxide gain was measured at each experimental case after the specimen was cooled naturally in air. The weight gain per unit area was measured as a function of holding time. Mechanical test was achieved by tensile technique and the measurement deduced from the stress- strain curve to obtain the bonding strength. The XRD technique was used to investigate the structural phases established at the intermediate layer region.

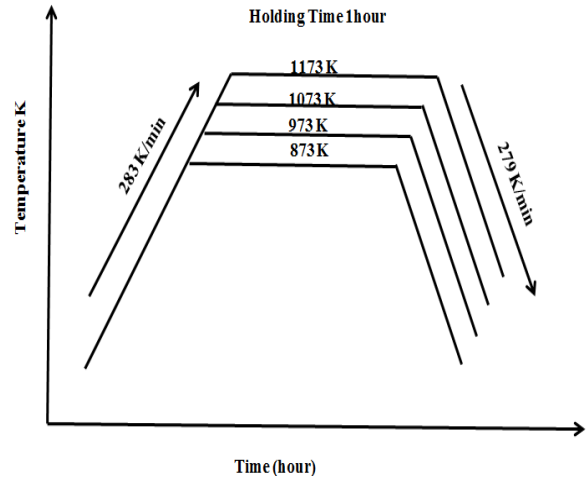


Figure 1: Temperature –Time Schedule.

**RESULTS AND DISCUSSION**

The oxide weight gain per unit area versus the square root of oxidation time is a parabolic plot in the form shown in Figure 2. These results were agreed with the researchers Kai Zhao *et.al.* [8].The best oxidized gain was found when the (st.st.) heated to the 1173 K as noticed in Table 1.

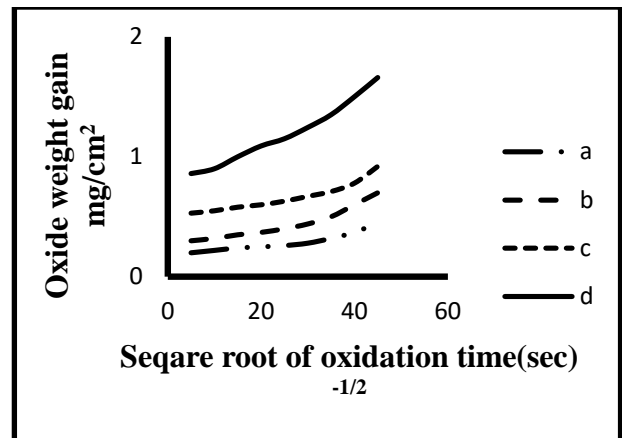


Figure 2: Oxide weight gain at different temperatures (a=873K, b-973K,c-1073K, and d-11073K).

The mechanism of bonding was by pre-oxidized of (st.st.), due to oxides growth carefully, the borosilicate tube glass could easily bonded to the(st.st.) .The fractured strength as a function of joint temperature were determined and were listed in Table 1, where the fracture mode found at the intermediate layer.

Table 1: Oxide weight gain and fracture strength as a function of joint temperature.

Sample	Joint Temperature K	Oxide weight gain mg/cm <sup>2</sup>	Fracture strength MPa
A	873	0.123	0.15
B	973	0.173	0.24
C	1073	0.234	2.5
D	1173	0.272	4

Also it can be cleared that the contact angle at 11073K was found equal to  $47^\circ$  as shown in Figure 3. The contact angle was measured by transfer two dimensions images to three dimensions images by using a suitable computer program.

Accordingly, the Young equation stated, when the contact angle much smaller than  $90^\circ$ , then the good wettability is achieved and hence good bonding between the pieces is achieving.

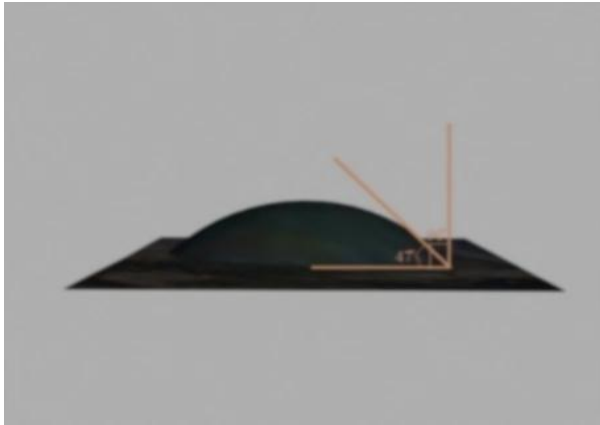


Figure 3: Contact angle of oxide to the stainless steel.

The good bonding may be due to the following: i/good wettability of powdered glass to (st.st.), ii/ the ambient condition of bonding process such furnace temperature and holding time, and iii/ particle size of powdered glass. Due to above parameters, the good bonding obtained because a reliably bonds have been joined the pieces together. This results were verified by the new structural phases such  $MgO_3Si$ ,  $AlCu_2Mn$ , and  $Mn_6NaO_4P$ , found at the intermediate layer between (st.st.) and borosilicate as shown in Figure 4.

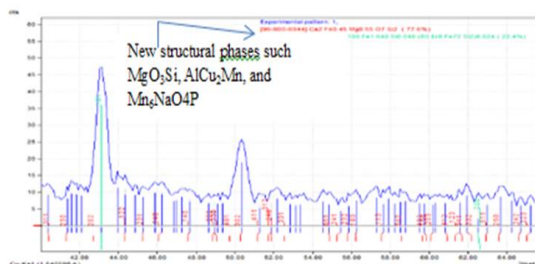


Figure 4: XRD pattern of (st.st.) surface.

Many researchers are agreements with this work results [8, 9]. Thickness of the adherent oxides growth is classified as an active bonding filler attributed the joint reliability. The optical microscope image shows different sizes of bubbles into intermediate layer as shown in Figure 5. The bubbles may cause cavities during bonding mechanism which reduce the bonding strength.



Figure 5: Optical view of bubble x100.

## CONCLUSIONS

From the test results it is concluded that:

- 1- The nature of the joint and the measured contact angle in addition to the room temperature behavior indicates that the coefficient of thermal expansion of both borosilicate powdered glass and stainless steel are perfectly matched.
- 2- New structural phases established at the intermediate layer were found enhanced the bonding reliability between borosilicate and stainless steel.
- 3- Number of bubbles per unit area and its diameter play an important role in the mechanism of joining glass to metal, where increases bubbles means reduces the bonding strength.

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