X-Ray Radiography of Alumina to Kovar Joining

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Abstract:

In this work alumina samples were used to be joined with kovar alloy by using different filler types . Fillers were formed with different concentrations of Silver , Copper , and Titanium powdered metals . These fillers where by mixing powders and compacting by hydraulic press , and sintering by tube furnace with argon atmosphere of 1100 ^{o}C and time of 60 min. Alumina/Kovar assemblies were joined in vacuum furnace of pressure of $2x10^{-4}$ torr at temperature of 850 ^{o}C and time of 20 min. .X-ray radiography was used evaluates and detect the defect in contact area Thrtee types of defects were detected namely craqcks , voids and Non uniformity . Shear test was achieved to determine joint strength . Correlation between shear test (destructive test) and X- ray radiography (Non destructive test) was found .

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.   60   (1100°C)

$2x10^{-4}$ /

20   (850°C)

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Introduction:

Joint between ceramic and metals are becoming evermore important in the production of electronic component and other high technique products[1] . The bond of ceramic to metal is a result of chemical and physical interaction in a very thin interfaces , most ceramic to binding is conducted at high temperature where the chemical reaction is to be expected[2,3] . One of the sources of the stress in ceramic to metal joint is the different of thermal expansion of the materials involved even if the materials are chosen to have closely similar expansion coefficient over a wide range of temperature is the case which so called matched seals while if the materials have different expansion coefficient the seal is called unmatched seal[4] . The heat capacity between metal and ceramic makes it practically impossible to obtain a real matched seal , for this reason the following principle are to be observed also expansion matching is good .

-The ceramic part should be designed having it well thick enough to withstand the stress and mechanical actions .

-The expansion coefficient of combined bonding should be kept near to that of ceramic part .

Wetting process:

The wettability of materials by active filler is indicated by the contact angle (θ) defined in fig.(1) contact angle depend on surface roughness ,when (θ) is greater than 90° the liquid is considered to non wetting while (θ) less than 90° are associated with a liquid which wets the surfaces contact [4,5] . The wetting is a physical process which is controlled by interfacial energy acting between the materials surfaces , the body of the melt and ambient gas according to the Young equation as follows :

$$Cos\theta = (\gamma_{sv} - \gamma_{ls})/\gamma_{lv} \quad -----(1)$$

Where $\gamma_{sv}, \gamma_{ls} and \gamma_{lv}$ are the interfacial free energies with respect to the boundaries between the phase solid (s) , liquid (l) and vapour (v) .

Materials selection:
1- Active filler alloys:

The principle requirements of an active alloy used for joining ceramic to metal are those of any type of filler metal as follows :
a-Ability to bonding and wetting the surface .
b-Ductile and have low melting point
c-Low vapour pressure .
d-It is thermal expansion accommodates with ceramic and metal .
e-The filler should have the ability to oxidation and corrosion resistance .
The alloy of Ag-Cu that have low melting point was selected . Copper is highly soluble in silver and the melting point of the alloy is lower ceramic and kovar[6,7] . Titanium is commonly used to join metals and ceramic by active filler , and can take considerable oxygen into solution and forms as a family of oxides whose chemistry depends on activity of oxygen [8,9] .

2-Ceramic:

Ceramic is one of the oldest technologies with history of about 1000 years behind it[10] . The properties of the ceramic are high thermal and electrical resistively ,high mechanical strength , wide range of

thermal expansion , no special annealing required , low vapour pressure , and good chemical stability The pure alumina is common type of ceramics and the increased purity makes alpha (α) type , increased strength and higher electrical resistivity [4] .

3-Kovar alloy:

Kovar alloy contains 18%Cobalt , 29%Nickel and 53% Iron . Kovar is ductile and free from embrittle element under all conditions of ordinary use including annealing and heating in air ,and can be soldered and brazed .A standard specification of kovar sealing alloy was designed by ASTM-F15-83[11]Thermal expansion of kovar alloy is matched seals [8] . These properties have led kovar being the most widely used for sealing technique in industrial applications.

2.3 Radiography inspection :

Radiography is a well established NDT method for obtaining information about discontinuities through the specimen [12] . The Radiography is performed by placing photograph film amount in light tight holder as a close to joint as a possible and then irradiating the assembly from the opposite side with either X-ray source . Radiography method is generally used successful detection of internal flows that are located well below the surface , and its interact with any substance . This interaction enable part to be inspected by differential attenuation of radiation enable difference in the intensity of radiation varies exponentially with thickness of sample through which it passes , this behavior is expressed according to the following equation[13] :

$$\frac{I}{I_O} = EXP(-\mu\chi) \quad \text{------(2.1)}$$

Where I and I_o is a function of exposure time (t) and operation current (i) , and since t and I fixed then ; $I\alpha D$ and $I_O\alpha D_O$ where D is the optical film density at the radiograph , and D_o is the optical film density at the background .

Then $\frac{I}{I_O} = \frac{D}{D_O}$ -------(2.2)

The radiographic interpreter is lookinf for the change may be caused one of three factors , namely a change of the thickness of the test piece , internal defect , and density change with may be induced by faulty processing and important that the interpreter can asses the nature and the cause of each density difference observed . The radiograph should be viewed in darkened room so that there will be light reflection from the surface film . Radiograph reveal three types of the flows [14-16] as shown in table (2.1) .

3-Experimental part:
3.1 Sample preparation:
A-Kovar samples:

Each kovar samples was first edge freed from burrs followed by polishing with metallographic paper and then by chemical cleaning [4] .

B-Alumina samples:

The samples should be free from any contamination . Chemical is done by immersing the samples in acetone for 20 minutes ,followed by immersing in dilute nitric acid for 5 minutes , and rinse in distilled water . Finally the samples fired in air at $1100 \,^{O}C$ for one hour .

C-Active filler samples:

Active filler samples formed from Ag , Cu , and Ti of the specification illustrated in table (3.1) .The active filler were formed by using a stainless

steel die of inner diameter (0.5cm) to compact a green pellets using hydraulic press (Stueres type) of Denemark product with (8KN) force and the sintering were carried out with tube furnace (Storhleln type) of Denemark product in argon atmosphere at 1100 ^{o}C for 60 minutes

D- Joining samples :

Joining of kovar to alumina using different active filler alloys mentioned in table (3.1) have been carried out in vacuum furnace (Honwell type) of England product with joining temperature of 850 ^{o}C , joining time of 20 minutes , and vacuum pressure of 2×10^{-4} torr .

E- X-ray radiography :

This method was performed by placing x-ray film amounted in light tight holder close to joint region , and irradiating it from opposite side . The X-ray system of (Balteau type) of Germany product combined with control unit . Figure (3.1) illustrated this system ,and the main characteristics of this system as follows :
Tube voltage: 160KV
Tube current: 5mA
Anode type: Tungsten
Focal spot size: 1.5mm
Exposure time: 5minutes

F- Joint strength measurements:

Shear tests were conducted as destructive testing to joining samples . This was designed to asses the strength of joining between kovar and alumina according to [17] . Tensile machine (servopulser type) of Shemadzu company product was used to measure the fracture stress at constant crosshead speed of 3mm/min The joint strength in (pa) was conducted from the force at fracture

point in (N) divided by fracture area (m^2) .

4- Results and discussion :
4.1 Defect interpretation :

Three types of defect were noticed namely cracks , voids ,and non uniformity of filler distribution in contact area . Typical radiograph are shown in Fig.(4.1A-C) which reveal these defect for active filler alloys . Table (4.1) summarized the observed defects in all joined samples . Voids density was determined as total voids presented in contact area . Voids depend on Ti concentration in fillers , higher voids density were found in fillers with higher Ti concentration (Ti wt%>2) .Fig.(4.2) shows the behavior density of voids with Ti concentration .

4.2 Joint strength results :

The average fracture stress obtained for all joint samples are shown in table (4.2) These results are plotted versus with Ti concentration as shown in Fig,(4.3) . Kovar was joined strongly to alumina this may be due to the effect of matching in thermal expansion coefficient of these system with all type of fillers . The effect of Ti concentration appears to very critical only about 2wt% for joint to acquire the maximum fracture stress at different filler type , since fillers with low Ti concentration shows voids density as showed in Fig.(4.2) , this may be due to the reduction in oxidation of kovar and filler surfaces . The result obtained for destructive tests and for non destructive tests (as non uniformity and voids density) were correlated in Fig.(4.4) and (4.5) . Joining strength decrease   exponentially   with

increasing non uniformity and voids density . It seems to be that the maximum allowable non uniformity and voids density in this type of joined system (lap joined) this equal to 25% since they will produce a suitable joint strength . This maximum allowable non uniformity and voids density .

Conclusions :

- The X-ray radiography technique is suitable non destructive testing for alumina to kovar lap joining system .
- Kovar to alumina joined samples contain three types of defects with different percentage
- The effect of defect type crack and voids density on joint strength is higher the effect of non uniformity .
- Samples joined with active filler type (Ag63%+Cu53%+Ti2%) reveal minimum defect in joined contact .

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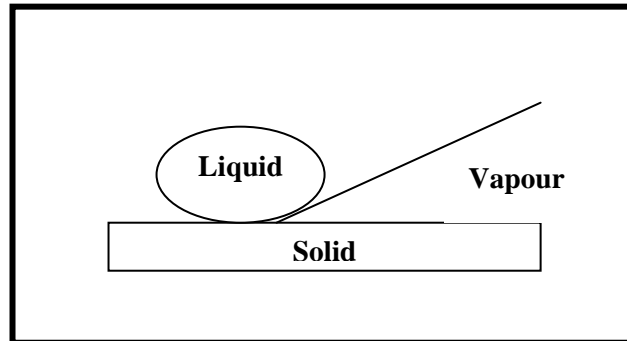16- ASTM Desgnation E390-75 .
17- ASTM-F-19-61-CLM-15 .

Fig.(1) Schematic diagram of contact angle .

Table (2.1) The main defect type revealed in radiograph .

Defect	Description	Radiographic appearance
Cracks	Discontinuity by fracture in the specimen	Fine dark line straight or wandering in direction
Voids	Small holes	Rounded or elongated dark area
Non uniformity	Density variation due to geometric regularities	Dark and bright area

Table (3.1) Active filler specification .

Sample code	Composition (wt%)	Dimension (cm)
F1	Ag(72%)+Cu(28%)+Ti(0%)	0.5x0.05
F2	Ag(63%)+Cu(35%)+Ti(2%)	0.5x0.05
F3	Ag(68%)+Cu(28%)+Ti(4%)	0.5x0.05
F4	Ag(55%)+Cu(37%)+Ti(8%)	0.5x0.05

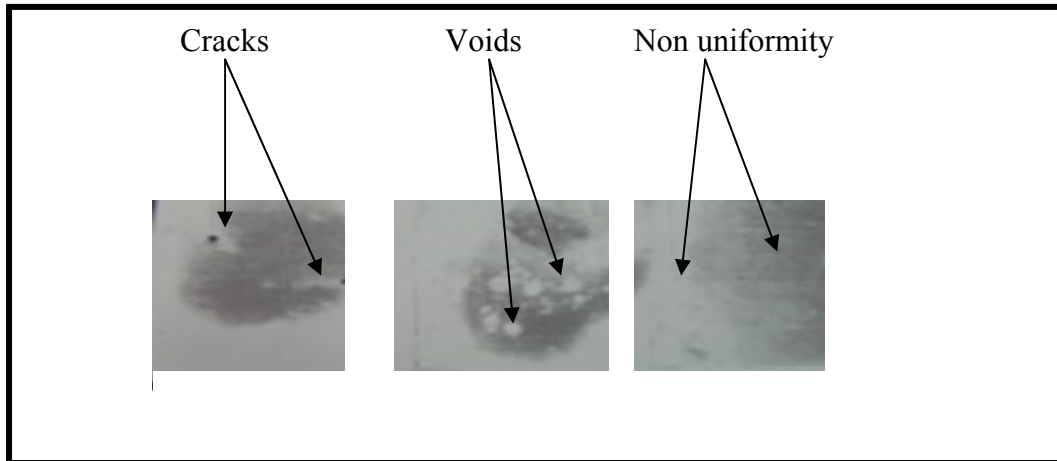Fig.(3.1) Photographic of Balteau X-ray system .

Fig.(4.1) Typical radiograph shows three types of defect .

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Table(4.1) Non destructive results of Kovar/Alumina joined samples

Joined modes	Cracks length		Voids density (%)
	Transverse cracks	Longitudinal cracks	
K(F1)A	Nil	0.2	21.0
K(F2)A	Nil	Nil	15.0
K(F3)A	0.1	0.1	24.0
K(F4)A	0.3	0.1	44.0
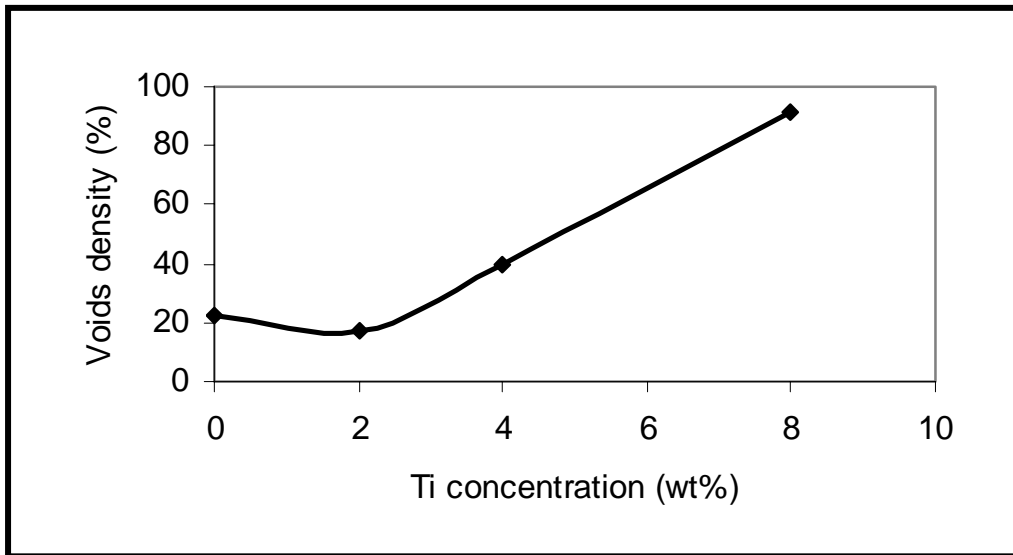
Fig.(4.2) Voids density variation with Ti concentration(%) .

Table(4.2)Joint strength of joined samples

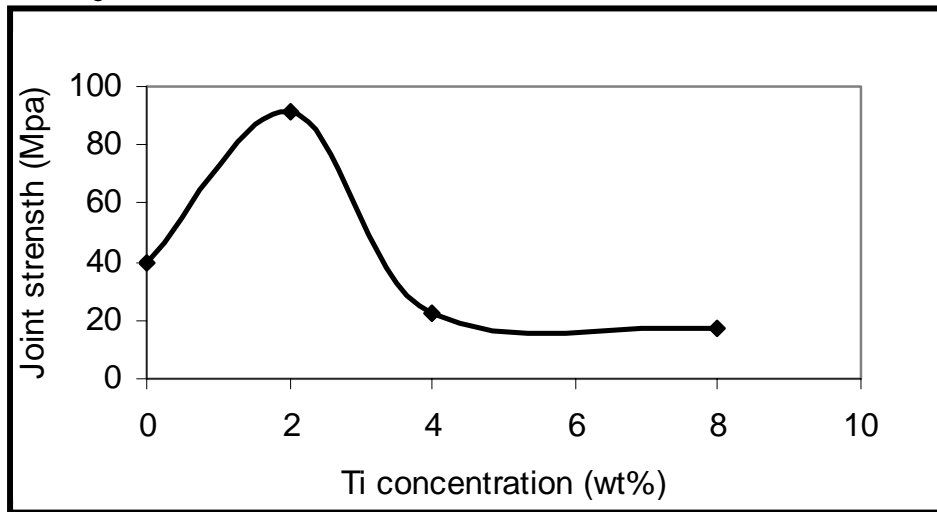| Samples code	Joint strength (Mpa)
K(F1)A	42
K(F2)A	91
K(F3)A	22
K(F4)A	17

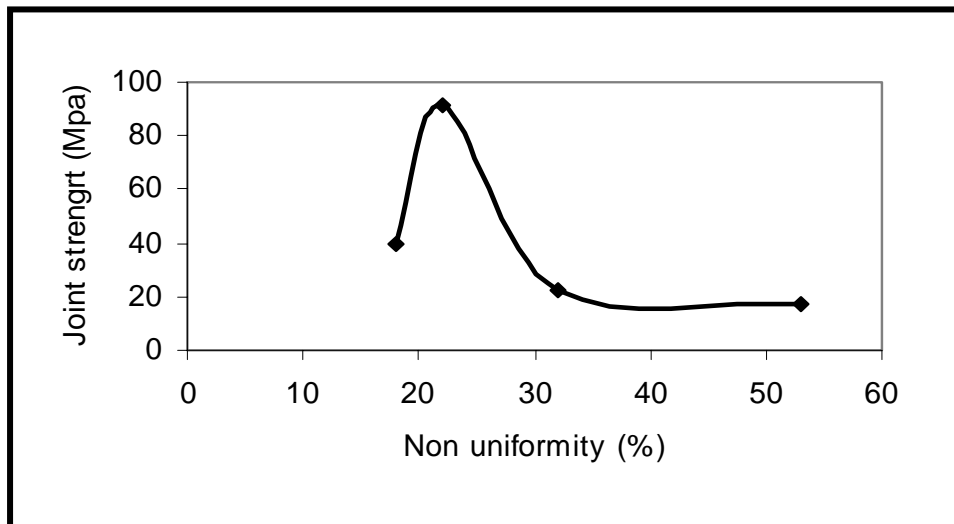Fig.(4.3) Joint strength(Mpa) versus with Ti concentration (wt%) .
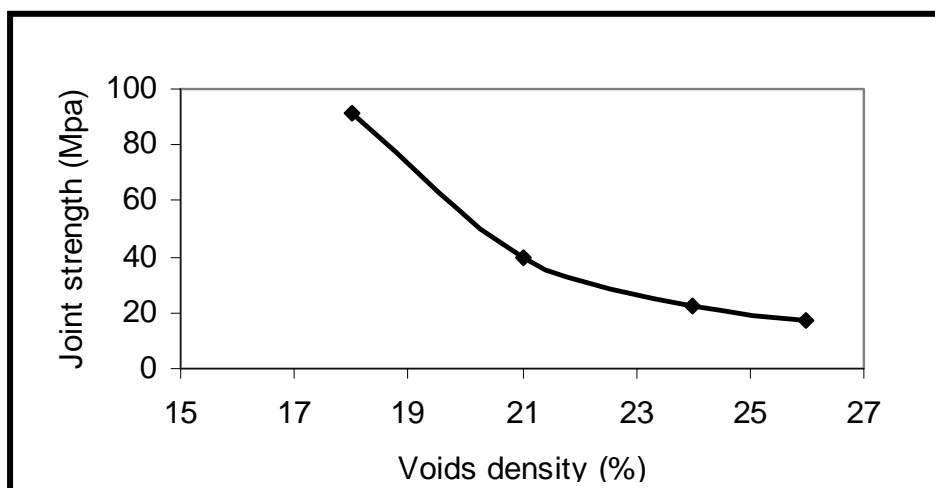

Fig.(4.4) Joint strength(Mpa) versus with non uniformity (%) .

Fig.(4.5) Joint strength(Mpa) versus with voids density (%) .