



Optimization of Diffusion Bonding of Pure Copper (OFHC) with Stainless Steel 304L

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(Received 10 April 2017; accepted 28 November 2017)

<https://doi.org/10.22153/kej.2018.11.002>

Abstract

This work deals with determination of optimum conditions of direct diffusion bonding welding of austenitic stainless steel type AISI 304L with Oxygen Free High Conductivity (OFHC) pure copper grade (C10200) in vacuum atmosphere of $(1.5 * 10^{-5} \text{ mbr.})$. Mini tab (response surface) was applied for optimizing the influence of diffusion bonding parameters (temperature, time and applied load) on the bonding joints characteristics and the empirical relationship was evaluated which represents the effect of each parameter of the process. The yield strength of diffusion bonded joint was equal to 153 MPa and the efficiency of joint was equal to 66.5% as compared with hard drawn copper. The diffusion zone reveals high microhardness than copper side due to solid solution phase formation of (CuNi). The failure of bonded joints always occurred on the copper side and fracture surface morphologies are characterized by ductile failure mode with dimple structure. Optimum bonding conditions were observed at temperature of 650 °C, duration time of 45 min. and the applied stress of 30 MPa. The maximum depth of diffuse copper in stainless steel side was equal 11.80 μm .

Keywords: Diffusion bonding, Pure copper, Stainless steel, 304 L.

1. Introduction

Joining dissimilar metals demands for increasing importance in many applications to utilize hybrid structures and compounds properties like high strength, thermal conductivity and good corrosion resistance [1]. The applications of dissimilar metals such as austenitic stainless steel- coppers (OFHC) are widely used in traditional and nuclear power plants [2]. However, the welding of austenitic stainless steel with copper alloys by conventional welding processes is not recommended by these methods because of probability of formations of newly intermetallic compounds at the weld pool. Copper and its alloys are majorly utilized for pipelines of heat exchangers; valve and clad plate for steel hulls of small ships, etc. alloys of copper have

enough resistance corroded in seawater [3]. Broadly, the application of Austenitic stainless steel to copper joints is utilized in the heat exchanger which consists of plates of austenitic stainless steel to copper [4]. In vessel apparatuses of the International Thermonuclear Experimental Reactor austenitic stainless steel and copper alloy are considered as a chief structure of materials for the first wall and find out systems. The austenitic stainless steel is also utilized in the nuclear surroundings [5].

Innovative joining operation of similar and dissimilar materials is provided by diffusion bonding welding process without producing macroscopic distortion; with minimum dimensional tolerance and no phase transformation or microstructural change occurred during the welding process [6]. A

diffusion bonding process also permits the production of high- quality joints with little or no need for post weld machining [7]. Design of experimental (DOE) was used in this work to get a suitable number of samples to be tested to characterize the effect of bonding variables (temperature, duration time and applied load) on the diffusion bonding joints. Bilgin (2009) studied the interfacial properties of diffusion bonding of stainless steel type 304L with Ti-6Al-4V using Cu interlayer. This work was applied at temperature range of (820-870) C°, time range of (50-90) min. and load of 1Mpa. The joints were examined using SEM, EDS, shear test and micro hardness test. At different conditions to predict optimum conditions of shear strength at 870 C°, and duration time of 90 min. due to better coalescence [8]. Xiong (2012) studied the diffusion bonding of stainless steel to copper with Tin, Bronze and gold interlayer. The diffusion bonding of two materials was obtained under temperature range of (830-955) C°, load 3Mpa and duration time of 60 min. The optimum conditions observed for this work were at 850 C° to get tensile strength of 228 Mpa for Tb-Au interlayer. The microstructure of joint was examined using SEM and EDS [9]. Sabetghadam (2010) evaluated the microstructure of diffusion bonded joint between stainless steel 410 and copper using Ni as interlayer. The bond joints were applied at temperature range of (800-950) C° load of 12 Mpa and duration time was 60 minutes in vacuum of (1.3×10^{-2} Pa). The microstructure and phases near bonding interface were examined using optical microscope, SEM and EDS. The result indicated an increase in thickness of reaction layer with an increase in temperature [10]. Kaya (2011) worked on the diffusion bonding of stainless steel with copper by two methods Conventional diffusion bonding and non-conventional diffusion bonding by applied external current. The specimens to be bonded were in dimensions of 10mm. dia. and 35mm. length. The bonding conditions were 875 C° duration time 30 min. and applied load 3Mpa, with heating and cooling rate of 20 C°/min. The interface of bonded joints was examined by tensile test and SEM&EDS. The strength of conventional bonding was 159 Mpa and for non-conventional 169 Mpa. The total diffusion of conventional was 6.4% wt. while for non-conventional was 9.1% wt [11]. All previous studies were restricted to the evaluation of the diffusion parameters influence and microstructure on the

interface of the diffusion area. But, there are no studies of the characterization of the influence of the diffusion bonding metallurgical transformation on the corrosion behavior of (AISI 304L/pure copper) bonding joints. The present work makes an effort with a contribution to this challenging dilemma.

2. Experimental

2.1 Preparation of Vacuum Diffusion Bonding Unit

In order to evaluate the sound diffusion bonding of two dissimilar materials of pure copper (OFHC) grade (C10200) and austenitic stainless steel type AISI 304L, the diffusion bonding requires to be applied in vacuum. Joining under vacuum reveals minimum impurity content, even in the case of high reactive metals. Vacuum provides faster and more complete degassing of joined materials and removal of oxides, impurities and contaminants from both surface and bulk materials. The system consists of vacuum diffusion pump, double stage rotary vacuum pump, electrical loading system with capacity of 50 ton, vacuum tube furnace with heating system, vacuum fitting and cooling system for furnace and diffusion as shown in Fig. (1).

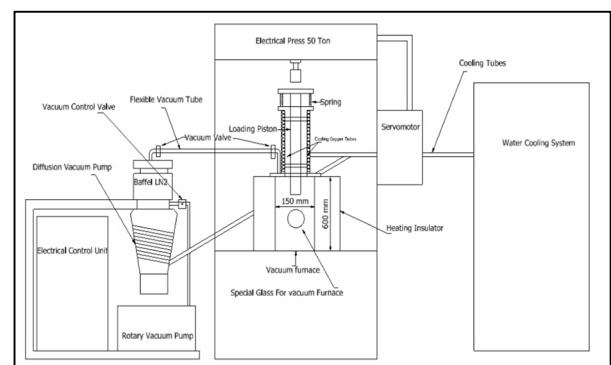


Fig .1. Vacuum Diffusion Bonding Unit.

2.2 Materials

Materials used in this work were wrought austenitic stainless steel AISI 304L according to (ASTM volume 01.01) and hard drawn pure copper (OFHC) grade (C10200) according to (ASTM volume 02.01).

Table 1,
Mechanical properties of two materials used.

Materials	Yield strength(MPa)	Ultimate strength (MPa)	Elongation %	Hardness
Stainless 304L	210	564	58	HRB=85
Copper(OFHC)	230	255	28	HB=65

2.3 Specimens Preparation for Diffusion Bonding Process

The materials to be joined by diffusion bonding were pure copper(OFHC) and stainless steel 304L. Cylindrical samples of 15mm diameter for each lengths were used with 30 mm and 60 mm for copper and stainless steel respectively[12]. The mating sample surface was prepared using convelial grinding on 1200 grade SiC papers followed by polishing with diamond paste using fabricated holder to get flat surface . The specimens were cleaned in ultrasonic bath using acetone for 15 min. to remove adhered contaminations and dried in air before bonding.

2.4 DOE (Design of Experimental)

2.4.1 Selection of process parameter (Surface Response)

The working range for each parameter is given in Table (2). This represents boundary of

optimum conditions to be searched according to (0.5-0.8 melting temperature) [6]; (4-13% σ_y) applied load and duration time range (15-75) min. from previous works. These ranges are selected according to the recommended optimum ranges of temperature, the duration time and applied load of diffusion bonding welding process [14].

Table 2,
Working ranges of selected parameters.

Parameters	Min.	Max.
Temperature (°C)	550	750
Duration time (min)	15	75
Applied pressure (MPa)	10	30

2.4.2 Construction of the experimental design matrix

The value of coded variables and uncoded variables for experimental design is shown in Table (3).

Table 3,
Experimental Design Matrix.

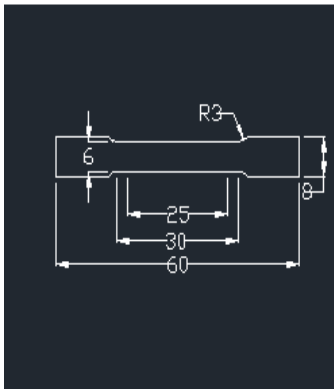
Exp. Numbers	Coded value			Original value		
	Bonding temp.(°C)T1	Bonding Time(min.)T2	Bonding pressure(MPa)T3	Bonding temp.(C°)	Bonding time(min.)	Bonding pressure(Mpa)
1	-1	-1	-1	600	30	15
2	+1	-1	-1	700	30	15
3	-1	+1	-1	600	30	25
4	+1	+1	-1	700	30	25
5	-1	-1	+1	600	60	15
6	+1	-1	+1	700	60	15
7	-1	+1	+1	600	60	25
8	+1	+1	+1	700	60	25
9	-1.682	0	0	550	45	20
10	+1.682	0	0	750	45	20
11	0	-1.682	0	650	45	10
12	0	+1.682	0	650	45	30
13	0	0	-1.682	650	15	20
14	0	0	+1.682	650	75	20
15	0	0	0	650	45	20
16	0	0	0	650	45	20
17	0	0	0	650	45	20
18	0	0	0	650	45	20
19	0	0	0	650	45	20
20	0	0	0	650	45	20

where:

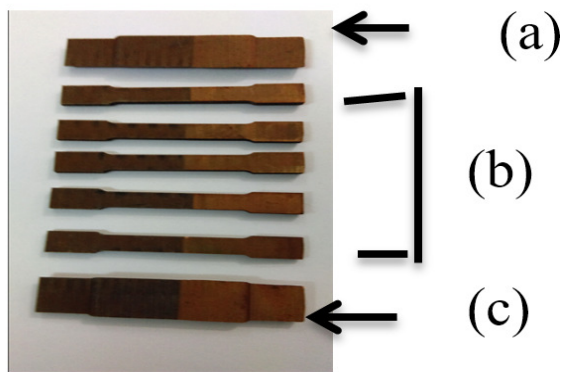
T₁: The code value of temperature; T₂: The code value of duration time; T₃: The code value of applied load.

3.7.1 Tensile Test of Diffusion Bonded Joints

To evaluate the tensile strength of bonded joints after welding, the bonded joint specimens were sectioned into small specimens for microstructure, mechanical properties tensile test. The tensile strength value was obtained by average value for tests. The tensile test specimens were cut by using wire cutting machine from bonded joints according to (ASTM E8-89) , in such a way that the weld zone was positioned at the center of gauge length. The tensile test specimen shown in Fig. (2), was applied by using universal testing machine type WDW 200 E with cross speed 0.1 mm/min.



Dimensions (mm) of tensile test specimen with thickness of (6mm) according to ASTM E8-89



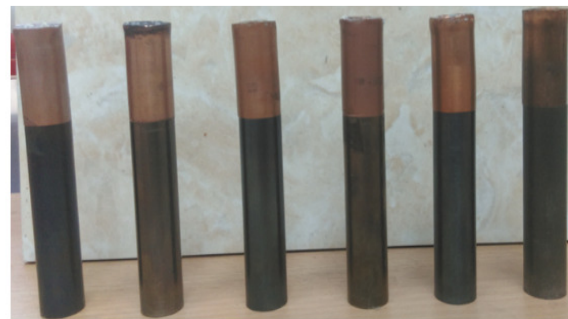
Tensile test specimen

Fig. 2. Specimens for Tensile test.

4. Results and Discussions

4.1 Tensile Test of Diffusion Bonding Joints

The bond strength of the bonding joints represents the response for the program Min Tab16. As shown in Fig.3 (A). The results from tensile tests are shown in Table (3). The fracture of most tensile test specimens was occurred in the copper side not at the bond line as shown in Fig.3 (B). This means the bond area is much stronger than copper side; this due to the used of vacuum atmosphere conditions which lead to better coalescence. This results in complete mating of two surfaces, and diffusion of copper atoms leads to good bonding.



T ₁ °C	650	600	650	650	600	650
T ₂ MPa	30	25	20	20	15	20
T ₃ min.	45	30	45	45	30	75

Fig. (A)

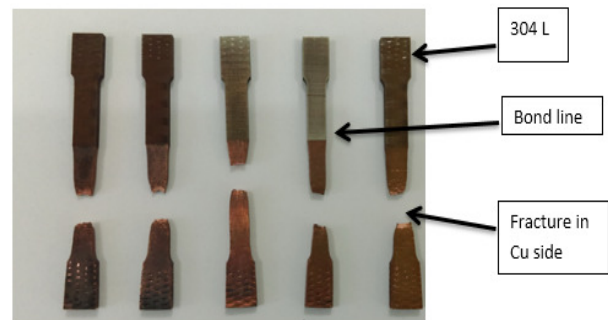


Fig. (B)

Fig. 3. (A) A set of diffusion bonding joints at different bonding conditions. (B) Fracture of diffusion bonding joints.

Table 3,
Results of tensile tests of bonding joints.

T1 Temp. (°C)	T2 Time (Min.)	T3 stress (MPa)	Fracture stress (MPa)	Fracture location	Ultimate Strength(MPa)	Elongation %
600	30	15	128.0	at copper	178	26
700	30	15	30.0	at interface	113	8
600	30	25	123.0	at copper	210	44.5
700	30	25	45.0	at interface	76	5.5
600	60	15	38.0	at interface	49	3.5
700	60	15	58.0	at copper	142	11.5
600	60	25	80.0	at copper	193	22.5
700	60	25	113.0	at copper	191	24
550	45	20	53.0	at copper	120	12.5
750	45	20	4.8	at interface	56	4.5
650	45	10	57.0	at copper	138	16
650	45	30	153.0	at copper	210	40.5
650	15	20	128.0	at copper	200	27
650	75	20	127.0	at copper	200	40.5
650	45	20	110.0	at copper	200	38.5
650	45	20	49	at interface	82	3.5
650	45	20	105.0	at copper	210	42.5
650	45	20	112.0	at copper	184	44.5
650	45	20	107.0	at copper	173	24
650	45	20	112.5	at copper	225	44.5

4-2 Parametric Analysis of Bond Strength

The main effect of plot of each variable of (T1, T2 and T3) on the bond strength is shown in Figure (4). The bond strength increases with an increase in temperature (T1) until reaching maximum value at temperature of 650 °C and then decreases until reached a temperature of 750 °C. This indicates maximum strength at 650 °C with higher bond strength than other temperatures values of bonding. The effect of a second variable that is, duration time of bonding (T2) on the bond strength of 15min. gets on the acceptable bond strength, but when time increases fluctuation in bond strength can be seen until reaches the maximum value of 75min. Finally, the effect of third variable (T3) is applied load on the bond strength at low values of load. This indicates low bond strength, but the maximum value of bond strength was observed at applied stress of (30 MPa) due to complete mating surface between two bonding metals used.

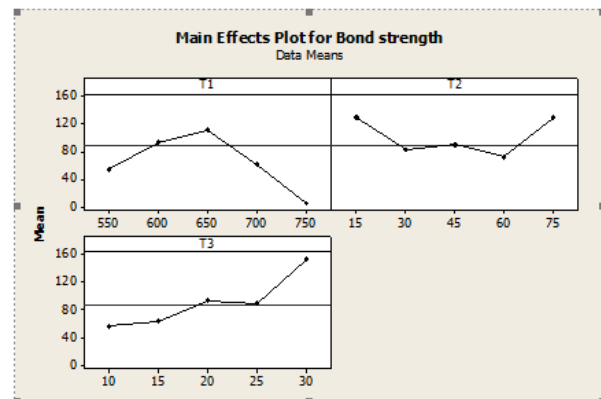


Fig. 5. Main effects on fracture stress.

4.2.1 Response Surface Regression

The analysis of results of bond strength versus T1 (Temperature), T2 (Duration time) and T3 (Applied stress) was done using uncoded units of input data. Regression was estimated coefficients for bond strength in order to obtain the significance by calculating (P-level) for each coefficient to determine which of these significant factors have effect on the bond strength. Depending on the significance level ($\alpha=0.05$), the coefficient with p- level value is greater than 0.05 which is not significant like (T3, T22, T33, T13 and T23), coefficients which have p-level less than 0.05 like (To, T1, T2, T11 and T12) represent the effective values on the bond strength of

diffusion bonding joints of two materials, therefore the mathematical model of significant parameters may be written as shown in equation (1).

$$Y = -1711.34 + 8.19T_1 - 29.85T_2 - 0.01 T_1^2 + 0.04T_1 * T_2 \dots(1)$$

4.2.2. ANOVA Results of Bond Strength

The analysis of variance (ANOVA) for the bond strength is shown in Table (4); it is made by using Fisher test (F-Test) and results. It can be

observed from this table that, the coefficient of R.sq=92.80% but the R.sq (Adj.) =86.32%. The difference between two values of R is small therefore, this means the three variables (T1, T2 and T3) qualified in variation of bond strength by (86.32%) and about of (13.68 %) due to random error and noise or the effects of other variables. Depending on significance value of (0.05), observed from the table, the total of P-value is less than 0.05 therefore the test is significant.

Table 4, ANOVA for bond strength with significant level (0.05).

Source	DF	Seq. (SS)	Adj. SS	Adj.(MS)	F	P
Regression	9	28882.0	28882.0	3209.1	14.32	0.000
Linear	3	8649.8	18994.4	6331.5	28.26	0.000
Square	3	12451.0	12451.0	4150.3	18.52	0.000
Interaction	3	7781.2	7781.2	2593.7	11.58	0.001
Residual Error	10	2240.6	2240.6	224.1	-----	-----
Lack of Fit	5	1588.2	1588.2	317.6	2.43	0.176
Pure Error	5	652.4	652.4	130.5	-----	-----
Total	19	31122.7	-----	-----	-----	-----
R-Sq. =92.80% R-Sq.(pred)=55.86% R-Sq. (Adj)=86.32%						

4.2.3 Optimization of Surface Response (Bond Strength)

The optimum value of bonding conditions is calculated using the equation (2), to predict the maximum value of bond strength of diffusion bonding joints for various bonding conditions.

$$D_i = \left(\frac{y_i - L_i}{T_i - L_i} \right)^r \dots(2)$$

where:

Di= Individual desirability.

yi = Response.

Ti= Target.

Li= Lower limit values of the response.

r= Unit weight factor (usually = 1).

The calculated optimum results are shown in Table (4); optimum bonding conditions was observed at experiment number 12, with bonding conditions of 650 °C, 45 min. and 30 MPa applied stress. The high value of Di equal to (1) represents the maximum value of bond strength and the value of Di=0 represents the minimum value of bond strength [14]. The maximum tensile strength

of bonding joint was obtained at the bonding conditions of temperature at 650 °C, duration time of bonding at 45 Min. and applied load of complete coalescence at 30 MPa. Joint efficiency is attributed to the quality of the joints. Therefore, joint efficiency was estimated. [15] as follow:

Joint efficiency = 153 MPa/ 230 MPa (for copper) % = 66.5%

Joint efficiency= 153 MPa/550 MPa (for stainless steel) %= 28%

4.3 Surface and Counter Plots

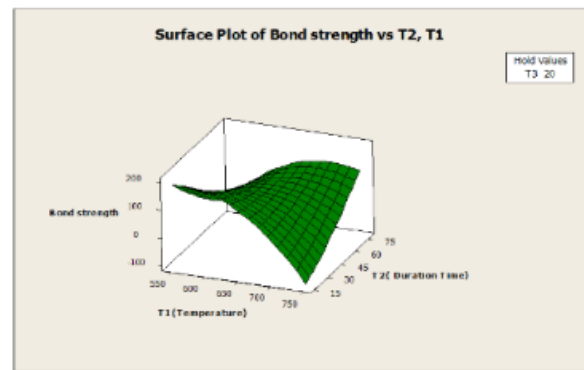
The response surface analysis which has two types of plots, the first is three dimensions (3D) representing the surface plot. The second is the two dimensions (2D) indicating the contour plot. The two plots were studied the combination effects of bonding parameters (T1, T2 and T3) on the bond strength of joints.

Table Ranks of response surface of results.

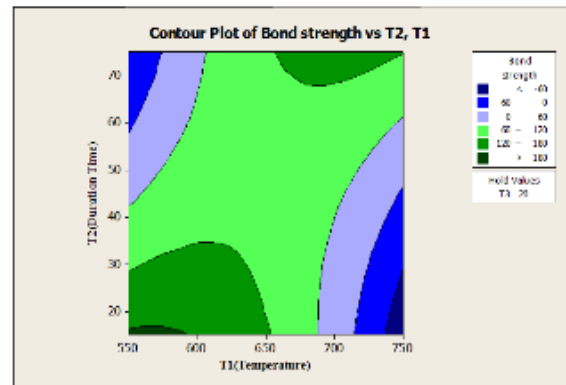
No.	T1 (°C)	T2 (Min.)	T3 (MPa.)	Fracture stress. (MPa.)	Di	Rank
1	600	30	15	128.0	0.8313	2
2	700	30	15	30.0	0.1700	18
3	600	30	25	123.0	0.7975	4
4	700	30	25	45.0	0.2712	16
5	600	60	15	38.0	0.2240	17
6	700	60	15	58.0	0.3589	13
7	600	60	25	80.0	0.5074	12
8	700	60	25	113.0	0.7300	5
9	550	45	20	53.0	0.3252	15
10	750	45	20	4.8	0.000	20
11	650	45	10	57.0	0.3522	14
12	650	45	30	153.0	1	1
13	650	15	20	128.0	0.8313	2
14	650	75	20	127.0	0.8245	3
15	650	45	20	110.0	0.7098	8
16	650	45	20	49	0.5209	11
17	650	45	20	105.0	0.6761	10
18	650	45	20	112.0	0.7233	7
19	650	45	20	107.0	0.6896	9
20	650	45	20	112.5	0.7267	6

4.3.1 Effect of Temperature and Duration Time on Bond Strength

Figures (5 a and b) indicate 3D surface and 2D contour plots which show combination effect of T1 (temperature) and T2 (duration time) at constant level (middle value) of T3 (applied stress) equal to (20 MPa.) on the bond strength. Figure. (5 a) shows the effect of temperature and duration time on the surface response bond strength. It can be observed from this, the bond strength increases with an increase in temperature and duration time but the effect of temperature is more effective than duration time. The optimal bonding conditions are temperature of 650 °C and duration time at 45 min. The increase in temperature above 650 °C leads to rapid grain growth which leads to low bond strength [16]. Fig. (5 b) represents the 2D contour plot for temperature with duration time. It indicates that the maximum bond strength observed at minimum temperature and duration time but it cannot be result in sound diffusion bonding joints. The optimum bonding condition at the range of 120-180 MPa as shown in Fig. (5 b). The combinations effect of temperature and duration time in contour was observed at maximum temperature of 650 °C and maximum duration time of 45 min., due to complete coalescence between two coupling surface and high diffusion rate.



(a)

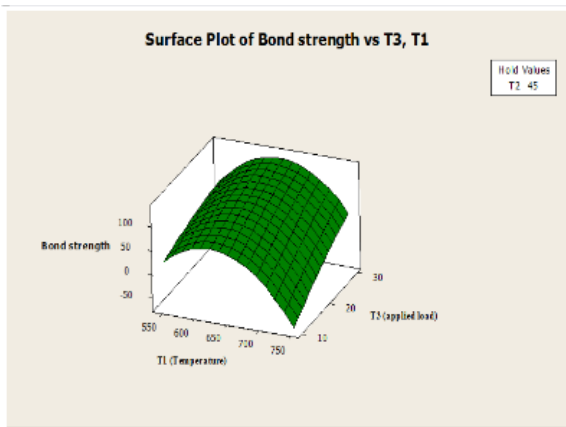


(b)

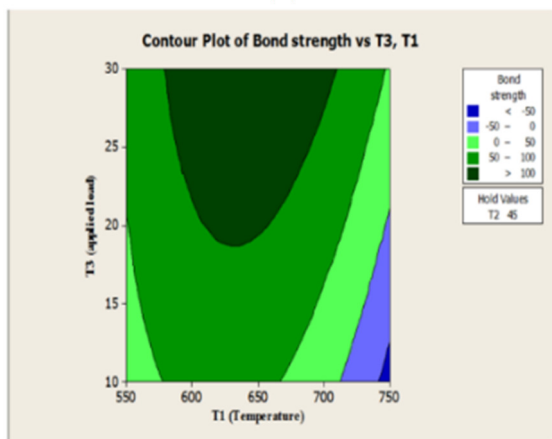
Fig. 5. Effect of temperature and time on bond strength (a) 3D surface plot (b) 2D contour.

4.3.2 Effect of Temperature and Applied Stress on Bond Strength.

Figures (6 a and b) illustrate combination effects of temperature and applied stress on the bond strength. Figure (6 a) shows gradual increase in bond strength until reached maximum value at load of 30 MPa. The bonding load was applied in order to secure a tight contact between the bonding surface and a vital condition for the interdiffusion atoms of metals jointed. If the applied stress is less than the optimum value, then the bond strength decreases. The optimum bonding temperature can be seen at 650 °C and at maximum applied load of 30 MPa. The effect of temperature on bond strength is more effective than the effect of applied load and the higher bond strength is 30 Mpa.



(a)

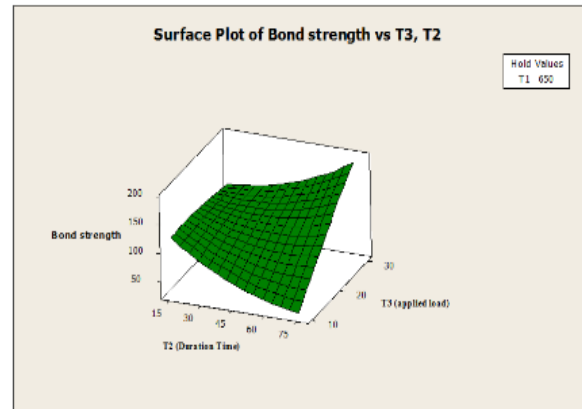


(b)

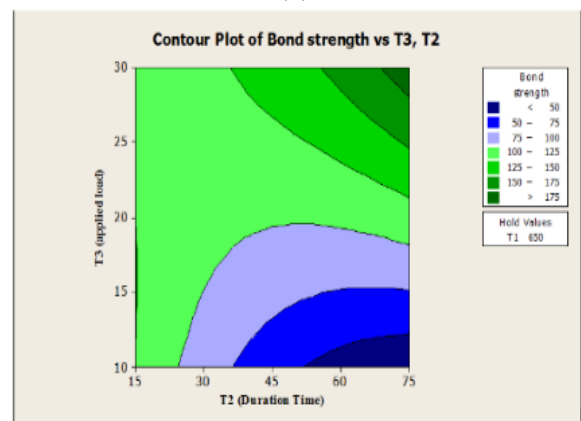
Fig. 6. Effects of temperature and applied stress on bond strength (a) 3D surface plot (b) 2D contour plot.

4.3.3 Effect of Time and Applied Stress on Bond Strength.

The effect of the two variables of duration time (T2) and applied stress (T3) on the surface response bond strength is shown in Figures (7) a and b. Fig. (7 a), shows higher effect of duration time than applied load. The right region of plot shows high change in applied stress up to 30 MPa. These results are combined with the effect of duration time on the bond strength, when time increases until reached 45 min. with an increase in applied stress to reach 30 MPa. At this point maximum value of bond strength is observed. Fig. (7 b) shows combination effects of high applied stress of 30 MPa and high duration time of 45 min. to get optimum value of maximum value of bond strength. Mating surfaces are expanding almost instantaneously. When the applied load increases more than the increase in temperature this leads to plastic deformation and doesn't get sound bonding joint between the two materials used [17].



(a)



(b)

Fig. 7. Effects of time and applied stress on bond strength (a) 3D surface plot (b) 2D contour plot.

5. Conclusions

1. The dissimilar diffusion bonding joints show lower tensile strength than the base material of hard drawn pure copper (OFHC) side and the maximum bond strength is 153 MPa.
2. Vacuum diffusion bonding unit has been successfully prepared and reached vacuum approximately of $(1.5 * 10^{-5} \text{ mbr})$.
3. The maximum tensile strength of diffusion bonding joint was observed for diffusion bonding joint at optimum bonding conditions of 650 °C, 45 min., and applied stress of 30 MPa, annealed at temperature of 800 °C, for 30 min.
4. The equation represent the effect of bonding conditions between hard drawn copper and austenitic stainless steel 304L with significant effects of bonding parameters was:

$$Y = -1711.34 + 8.19T1 - 29.85T2 - 0.01 T12 + 0.04T1*T2$$
5. The strong effects of diffusion bonding parameters are temperature > duration time > applied load.
6. The efficiency of optimum bonding joint was higher as compared with hard drawn copper and lower as compared with austenitic stainless steel.

6. References

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ثلية وصلات الربط الانتشاري للنحاس خالي الأوكسجين عالي الموصلية مع الفولاذ المقاوم 304L للصدأ

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الخلاصة

يتضمن البحث تحديد الظروف المثالية للحام الانتشاري المباشر للنحاس خالي الأوكسجين_ عالي الموصلية مع الصلب المقاوم للصدأ الاوستنايتي نوع(304L) مختبريا. وقد تم ربط المعدنين في جو مفرغ من الهواء يصل الى 10^{-5} mbr) وقد تم استخدام برنامج (Minitab16) لمعرفة الظروف المثلى لربط المعدنين. ومن خلال استخدام البرنامج تم دراسة التأثير المشترك لعوامل الربط على قوة الربط. وقد اجريت فحوصات البنية المجهرية لمنطقة الربط الانتشاري باستخدام المجهر الالكتروني الماسح (SEM-EDS) لمعرفة التركيب المجهري وعمق الانتشار في منطقة الربط والصلادة المايكروية والتحليل الطيفي (XRD) لمعرفة الأطوار التي تكونت في منطقة الربط ومن خلال الفحوصات تبين تكوّن محلول جامد بين النحاس والنيكل (CuNi) بوصفه طوراً احادياً يعطي صفات جيدة. تم ايجاد الظروف المثلى للربط هي عند درجة حرارة 650°C وزمن ربط 45 دقيقة والجهد المسلط كان 30 ميكاباسكال. ولمعرفة قوة الربط تم اجراء فحص الشد لمنطقة الربط حيث كانت متانة الربط عند ظروف الربط المثلى تساوي 103 ميكاباسكال وذات كفاءة ربط 66,5% مقارنة بالنحاس وان عمق الانتشار الذي تم الحصول عليه في منطقة الربط كان $11.80\ \mu\text{m}$.