PAPER • **OPEN ACCESS**

Characterization of Diffusion Bonding of Stainless Steel AISI 316 and Pure Titanium sheets Using Copper Interlayer

To cite this article: Ahmed Ali Akbar Akbar *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **518** 032041

Characterization of Diffusion Bonding of Stainless Steel AISI 316 and Pure Titanium sheets Using Copper Interlayer.

Ahmed Ali Akbar Akbar[1], Bahaa Sami Mahdi[1] and Adnan Jumaa Aluwi[1]
[1]Production and Metallurgy Engineering Department, University of Technology, Baghdad, Iraq

Abstract The present work is focused on diffusion bonding between pure titanium and austenitic stainless steel(AISI 316) using interlayer of pure copper foil in vacuum atmosphere of (1.5 x10-5 mbr.) at different temperatures (850,900,950 and 990) Co for diffusion times of (20,30,40,50 and 60 min.) under different pressures (1.5,2,3 and 4) Mpa. Many tests and inspections: tensile shear and microhardness tests, also SEM EDS and X-ray diffraction techniques were utilized to characterize the bonded joints. Tensile shear test results showed that the maximum shear strength of diffusion bonded joints was (96.34) MPa at 950oC, pressure 3Mpa and 50 min. with an efficiency of joint (75%) and maximum diffusion depth of (88 µm) The maximum micro hardness of (338.6 Hv)was obtained in the copper interlayer metal compared with the base metals(stainless steel and titanium) due to formations of brittle intermetallic compounds . The EDS and XRD analysis results confirmed the formation of different intermetallic compounds [FeTi , (Cu + Ti37Cu63Fex), TiCu , Ti2Cu, Ti3Cu4 ,] .The surface fracture topography showed that fracture is located at the bright brittle intermetallic compound phase(Ti3Cu4) which was 85% of the fractured area and 15% was at the (Ti2Cu)dark region .

1. Introduction:-

Diffusion bonded components consisting of pure titanium and stainless steel have several applications in chemical and nuclear industries .A couple consisting of these two dissimilar materials suffers from poor mechanical properties because brittle intermetallic compounds are formed in the diffusion zone. These brittle intermetallic compounds degrades the mechanical properties of the bond joints. For this reason, researchers have tried a number of soft interlayer materials to enhance the properties of the bonded joint. In joining titanium to stainless steel, using fusion welding techniques, formation of brittle intermetallic compounds is the main risk.

P.R.C.Camargo and et al [1]carried out joining of titanium to stainless steel by vacuum brazing in order to characterize the brazed joints for high mechanical properties. Formation of intermetallic compounds between the base metals and the cu-silver alloy filler metal degraded the mechanical properties. S.Kundu and et al [2] studied the diffusion bonding of commercial titanium and stainless steel AISI304 using pure copper interlayer,. Tey reported that large numbers of CuxTiy and FeTi brittle intermetallic compounds were resulted and participated in decreasing bond strength. Debasis poddar[3]studied the diffusion bonding of commercial titanium and precipitation hardening stainless steel without interlayer, the same problem of intermetallic compound formation was experienced.. Diffusion bonding of titanium was studied by A.H.M.E RAHMAN [4] by applying silver and copper interlayers, maximum bond strength obtained was with copper interlayer.

Experiments on diffusion bonding of titanium and stainless steel using copper interlayer was carried out by Shuying Liu and etal[5] Mechanical properties and interface structure characteristics were studied , maximum joint strength was obtained at bonding pressure of 5Mpa.The diffusion bonding parameters(temperature, pressure and holding time) were of interest of most of researchers to predict the most predominant parameter and the combined effect of them on joint properties. B.Szwed and M.Konieczny [6] studied the effect of bonding parameters on the properties of diffusion bonded joints of titanium and stainless steel. Maximum shear strength obtained was at bonding temperature of 900C° and highest hardness up to 580Hv was achieved at FeTi phase. Microduplex stainless steel and titanium were joined with Ni interlayer at 900C diffusion bonding temperature with varying holding time from15 to 90 min.[7].Most of failures were at NiTi2 layer .Maximum tensile strength520Mpa was obtained in direct diffusion and increased to 640Mpawith Ni interlayer while a maximum Shear strength 405Mpa was obtained indirect bonding and increased to 479Mpa in case of Ni interlayer.Silver was used as interlayer to bond Ti-6Al-4V titanium alloy and stainless steel AISI304 [8].The quality of the diffusion bond was confirmed by the results of shear test and microstructure analysis. Maximum lap shear strength was at pressure of 5Mpa for a holding time of 90 min,

De- feng MO et al [9] have also studied the diffusion bonding of titanium alloy and stainless steel but different interlayers were inserted (Cu,Ni and Ag). Multi-layer interlayer technique prevented the formation of brittle intermetallic compounds. Aluminum alloy was also applied as interlayer in diffusion bonding of stainless steel to titanium alloy by K.Chandrappa and et al[10] . joint strength was improved when aluminum alloy was inserted as interlayer at optimum diffusion bonding conditions. A review of diffusion bonding of titanium and stainless steel was done by Zhenzhen Yuand et al [11] .They reported that diffusion bonding titanium alloys to steels requires a diffusion barrier which is the interlayer material and in all conditions of inserting interlayer materials ,intermetallic compounds are expected. The aim of present work is to study effect of holding time on the microstructure the aim of present work is to study effect of holding time on the microstructure and mechanical properties of diffusion bonding joints of titanium and AISI316L stainless steel using copper as interlayer with different input variables.

2. Experimental work:

2.1 Materials

Two base materials, austenitic stainless steel AISI 316 sheet of 2mm in thickness and pure titanium of 1mm in thickness were selected with a pure copper interlayer foil of 0.04mm thickness. The specimens of the two base materials were prepared with dimensions of (12mmx25mm) and the copper interlayer was prepared with (12mmx12mm) to be placed between in an overlap joint between the two base materials. Table (1) shows the chemical compositions of the three selected materials.

Table 1. Chemical analysis (wt%) for the selected materials.

Stainless Steel AISI316L			Pure Titanium			Pure Copper		
Element	Analytical Value (Wt. %)	Standard (Max.)	Element	Analytical Value (Wt. %)	Standard (Max.) ASTM grade 1	Element	Analytical Value (Wt. %)	Standard Cu-OF Grade C1020
C	0.059	0.03	N	-	≤0.03	Cu	99.939	99.96
Cr	16.5	16-18	C	-	≤0.08	Cr	0.001	Traces
Ni	9.75	9.5-14	H	-	≤0.013	Fe	0.006	Traces
Si	0.565	0.75	Fe	0.192	≤0.15	Sn	0.002	Traces
P	0.045	0.045	O	-	≤0.12	Mn	0.001	Traces
Mn	1.37	2	Al	0.015	-	other	-	
S	0.014	0.03	V	0.0037	-	-	-	
Mo	2.01	2-3	Ti	Balance	Balance	-	-	
Fe	Balance	Balance	-	-		-	-	

2.2 Diffusion Bonding Process

The diffusion bonding unit shown in figure 1 was used to join the samples operating at vacuum of (1.5x10-5mbr) during the process where the holding time was changed from 20 to 60 minutes. For different pressure/temperature combinations to study the holding time effect on joint strength for different other variables. After performing joining processes, the specimens were tested by single lap-tensile shear test shown in figure 2, also bonded samples were sliced longitudinally by wire cutting from one side with a depth of 1.5mm. The join samples were mounted and prepared by grinding, polishing and electrochemical etching using 10% oxalic acid in distilled water with 3 voltage DC electrochemical cell to reveal the different joint microstructures. Micro-examination was done using TESCAN(R) Vega3LUM scanning electron microscope assisted with Oxford Max3 Energy Dispersive spectroscopy detector (EDS) to check the diffusion between three materials and determine the optimum diffusion conditions. Fractured surfaces resulted from tensile shear test were examined by SEM to determine the location and fracture mode of diffusion bonded joints.

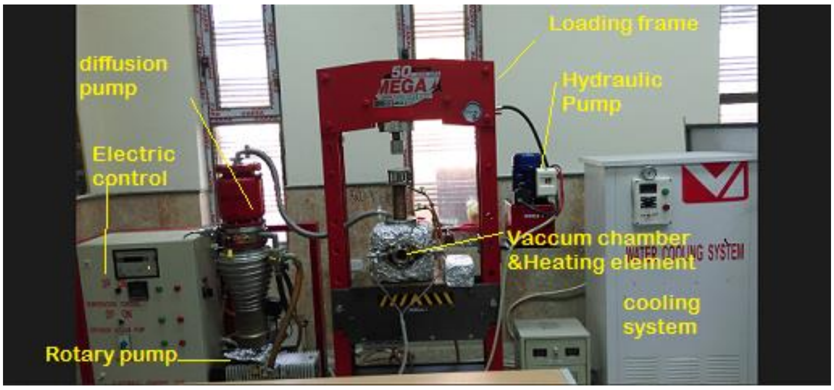


Figure 1. Vacuum Diffusion Bonding System.

Figure 2. diffusion bonded lap joints

3. Results and Discussion

3.1 Shear strength Test results:

The experimental data for the tensile shear strength is reported for all experiments and tabulated with input data given in table 2.

Table 2. Shear strength of diffusion bonding joints using different input variables.

Sample No.	T ºC	P Mpa	t Min.	F N	Real area mm^2	□ Mpa
1	850	1.5	20	1154.954	74.2736	15.55
2	900	2.0	20	2300.205	62.8815	36.58
3	950	3.0	20	5214.59	76.2813	68.36
4	990	4.0	20	4257.316	79.3831	53.63
5	850	1.5	30	1910.379	86.6385	22.05
6	900	2.0	30	1428.969	63.9074	22.36
7	950	3.0	30	8616.329	99.3695	86.71
8	990	4.0	30	7348.14	96.7497	75.95
9	850	1.5	40	2007.954	79.17799	25.36
10	900	2.0	40	3257.31	61.832	52.68
11	950	3.0	40	7001.137	75.8027	92.36
12	990	4.0	40	5602.129	74.3481	75.35
13	850	1.5	50	1834.908	79.0568	23.21
14	900	2.0	50	3913.241	68.8466	56.84
15	950	3.0	50	7914.013	82.1467	96.34
16	990	4.0	50	4168.696	60.7239	68.65
17	850	1.5	60	891.6621	45.1246	19.76
18	900	2.0	60	3763.604	75.8027	49.65
19	950	3.0	60	5582.249	71.2385	78.36
20	990	4.0	60	5270.157	73.8531	71.36

Figure 3 represents the relationship between the diffusion time and shear strength for different temperatures and pressures values. The maximum shear stress of (96MPa) was achieved when the working conditions were (holding time=50mim., pressure=3Mpa, temperature=950Cº) Comparing the maximum shear value with that of the weakest material which is copper, the joint strength represented %75 of the shear strength of copper. Temperature rises up to 950 Cº which increases the joint performance due to increase in the diffusion activity of material atoms motion ,while beyond this value the strength was decreased due to the high diffusion rate and reactions which produces undesirable intermetallic compounds .

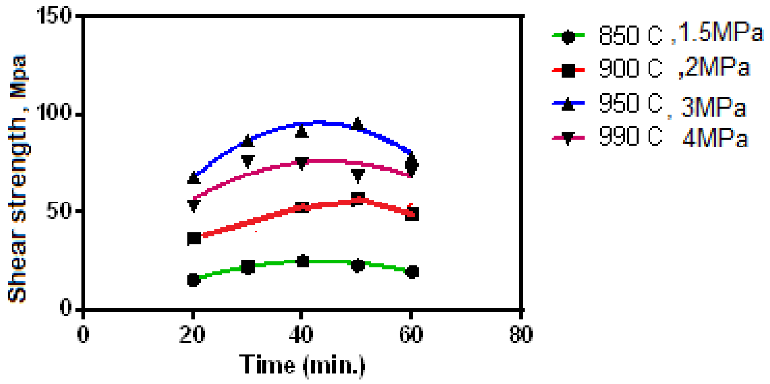
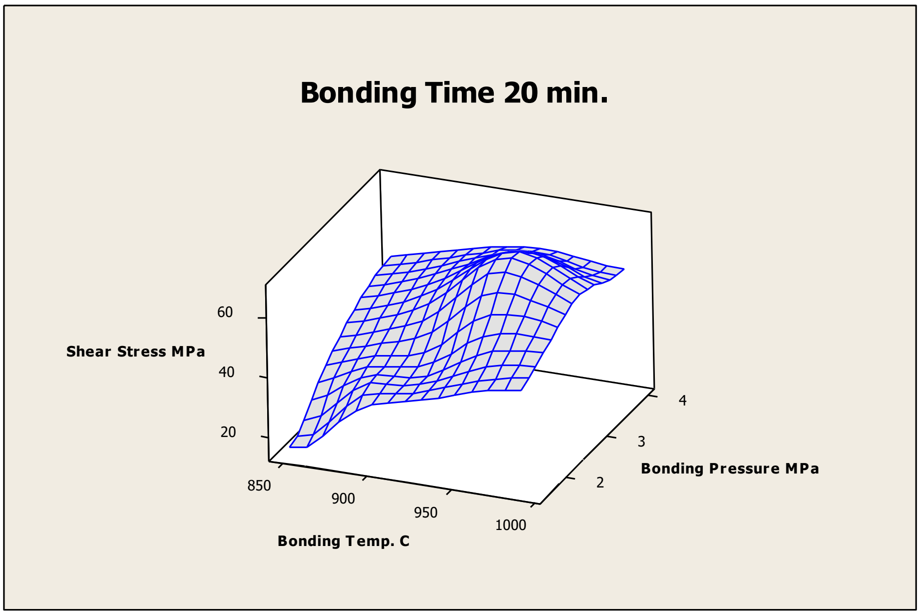
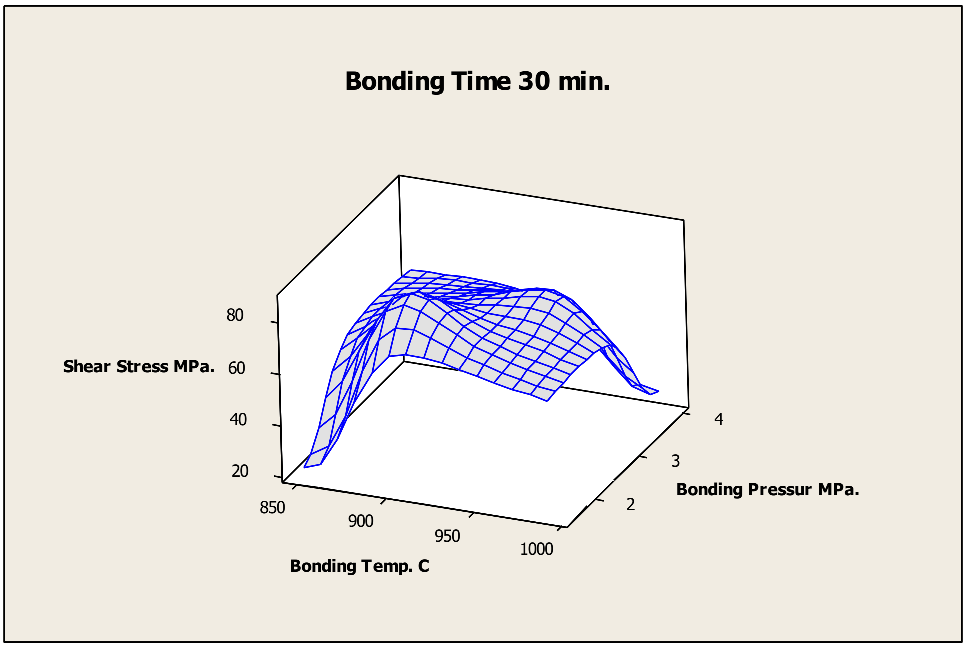


Figure 3. The relationship between holding time and shear strength at different temperatures and pressures.

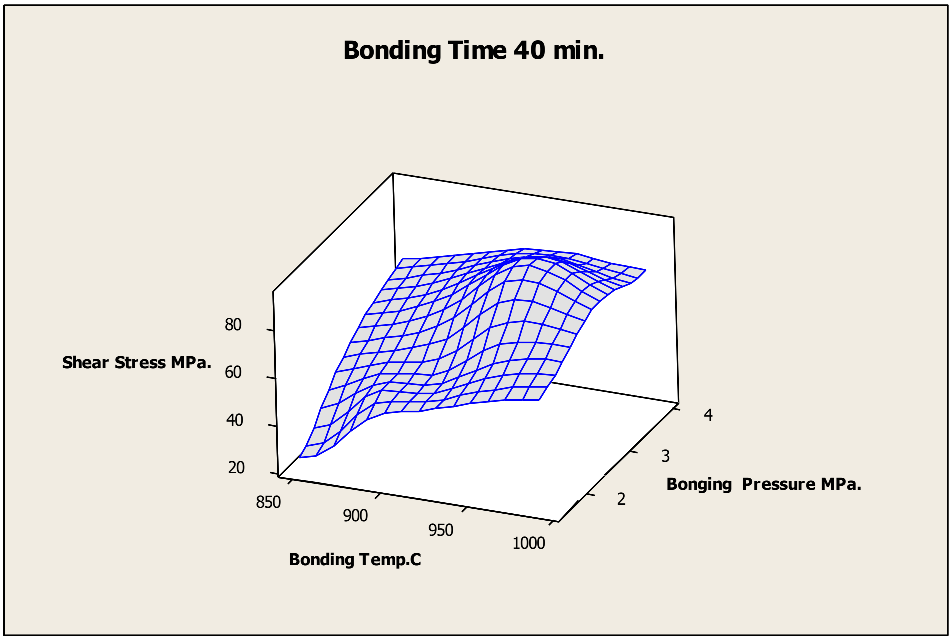
The data obtained for the effects of temperature and pressure variation on the shear strength of the bonded joints for each holding time is presented in figure 4(a, b, c, d and e) for periods of 20,30,40,50, and 60 minutes. Surface response plots are made to predict the interaction effect of both bonding temperature and pressure on the shear strength and to confirm the values of the best bonding conditions (temperature, pressure and holding time). All the 3D –plots show a peak value of shear strength located on the surface but each of certain values of bonding conditions. In figure (4a) for a holding time of 20 minutes maximum shear strength attained was (68.36Mpa) at 950C^o and pressure of 3Mpa . For 30 minutes a strength of(86.71Mpa) at 950 C^o and 3Mpa while at 40 minutes a strength of (92.36Mpa.). The maximum value of shear strength of (96.34Mpa) has attained as shown in figure(4d) at holding time of 50 minutes at 950 C^o and pressure of 3Mpa. The surface response plots also indicate that no combination of diffusion bonding variables rather than the above-mentioned values revealed the enhancement of the shear strength at low values of temperature, pressure and holding time.



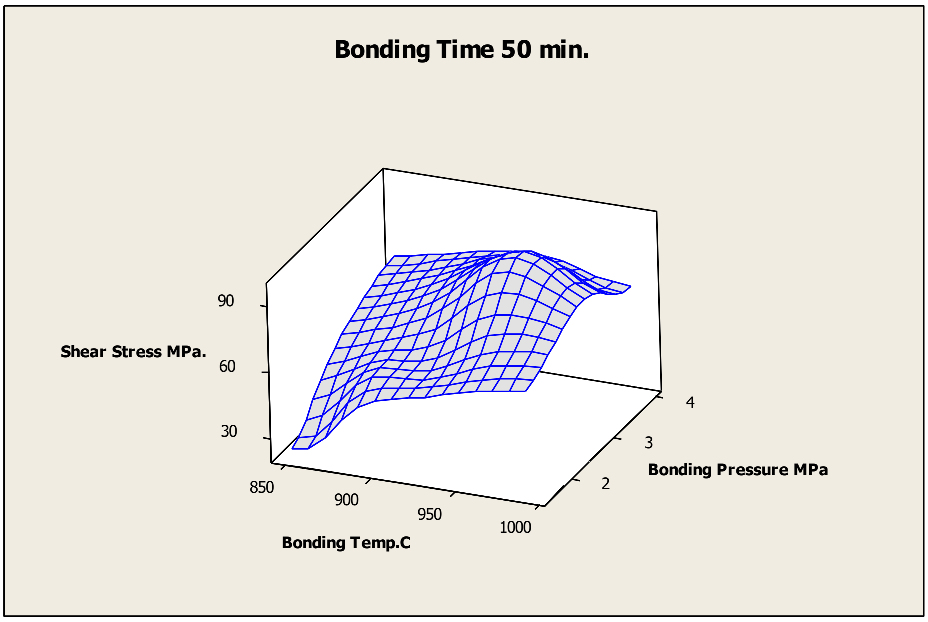
(a)



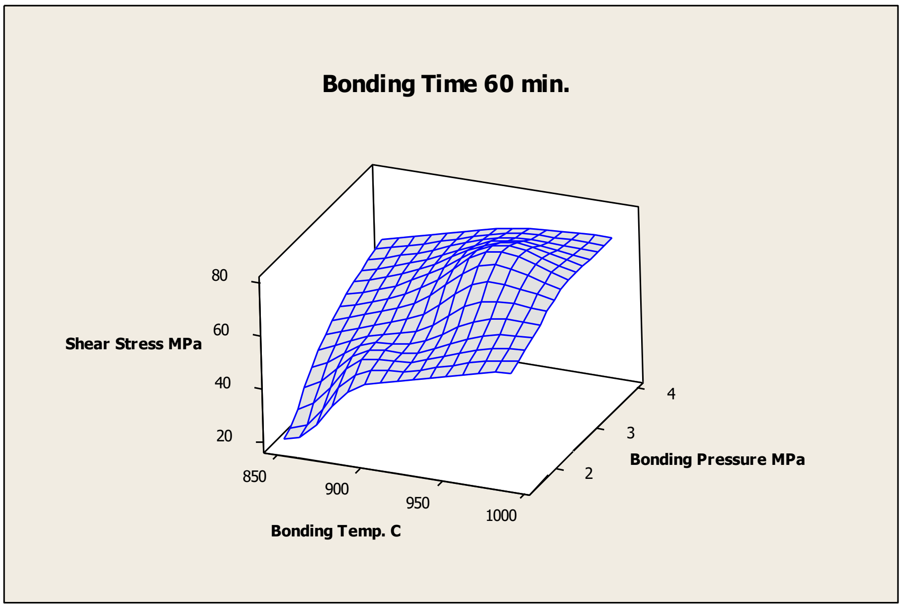
(b)


Bonding Time 40 min.
Shear Stress MPa.
80
60
40
20
850
900
950
1000
Bonding Temp.C
2
3
4
Bonging Pressure MPa.

(c)


Bonding Time 50 min.
Shear Stress MPa.
90
60
30
850
900
950
1000
Bonding Temp.C
2
3
4
Bonding Pressure MPa

(d)



(e)

Figure 4. surface response plots represent effect of diffusion bonding temperature and pressure at different bonding times(20,30,40,50,and 60minutes) of AISI316 and pure Titanium using pure copper interlayer foil

3.2 Microhardness results analysis

Microhardness test was carried out for the bonded joints at different conditions. Figure (5A, B, C and D) show the micro hardness variation at two base metals sides and copper interlayer region. Joints performed at a temperature of 850 Cº showed minimum microhardness values in three regions. These regions exhibited little difference in hardness values is noticed while increasing bonding temperature resulted in an increase in micro hardness of the interlayer region which was explained to be resulted due to the formation of brittle intermetallic compounds. Maximum values were obtained at bonding time (40minutes) and the highest value measured was at temperature of 950 Cº to be 343.6 HV. for holding time of 40 minutes. More or less than this time resulted lower harness for all four conditions. Holding time longer than 40 minutes at these diffusion temperatures causes dissolution of these intermetallic compounds.

IOP Conf. Series: Materials Science and Engineering **518** (2019) 032041 doi:10.1088/1757-899X/518/3/032041

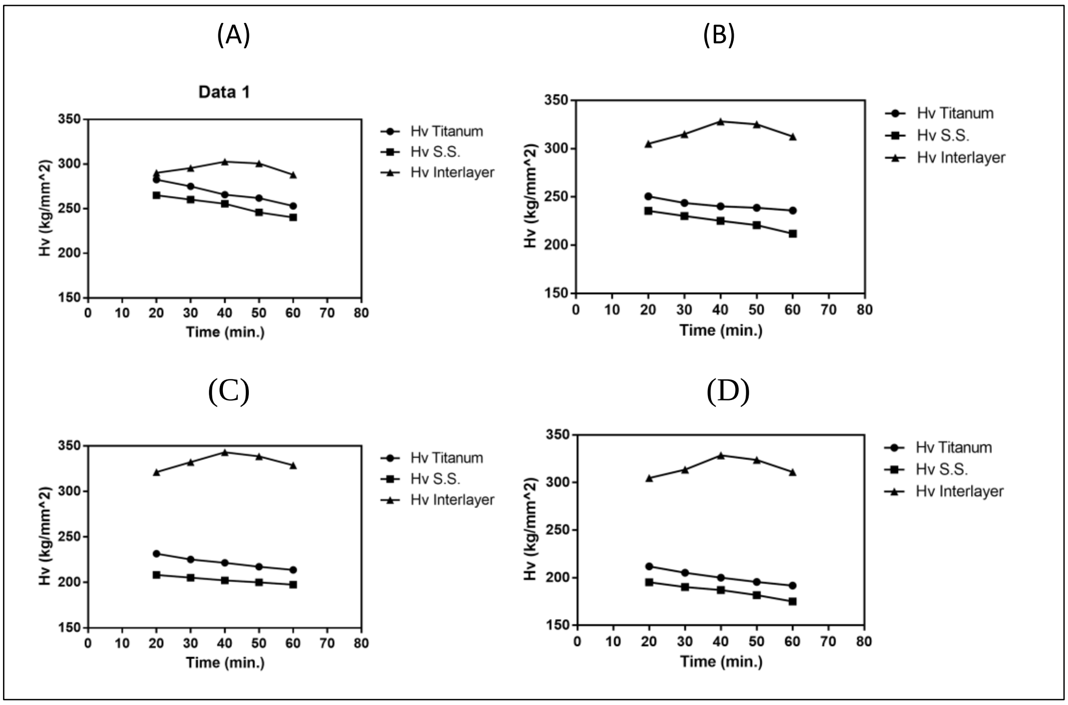


Figure 5. Effect of diffusion holding time on the micro hardness of bonded joints at bonding pressure of 3Mpa and temperatures 1-850 C^o 2-900 C^o 3-950 C^o 4-990 C^o

3.3 SEM, EDS and XRD Results Analysis

To reveal produced phases in the diffusion joint as metallurgical microstructure it is necessary to inspect micro-test at different points across the joint. The microanalysis (SEM) is the best device that can be used with assisted of Energy Dispersive Spectroscopy (EDS) analyzer. TESCAN VEGA3-LM Scanning electron microscope assisted with Oxford MAX3 EDS analyzer used in Production and Metallurgy Engineering department in UOT-Baghdad to resolve the produced phases across after etching the polishing the cross-section of diffusion joints. The results were projected on the ternary (Fe-Ti-Cu) phase diagram as shown in figure 6. Minimum shear strength of 15 MPa was obtained using a pressure of 1.5MPa and heating at 850oC for 20 minutes in vacuum.

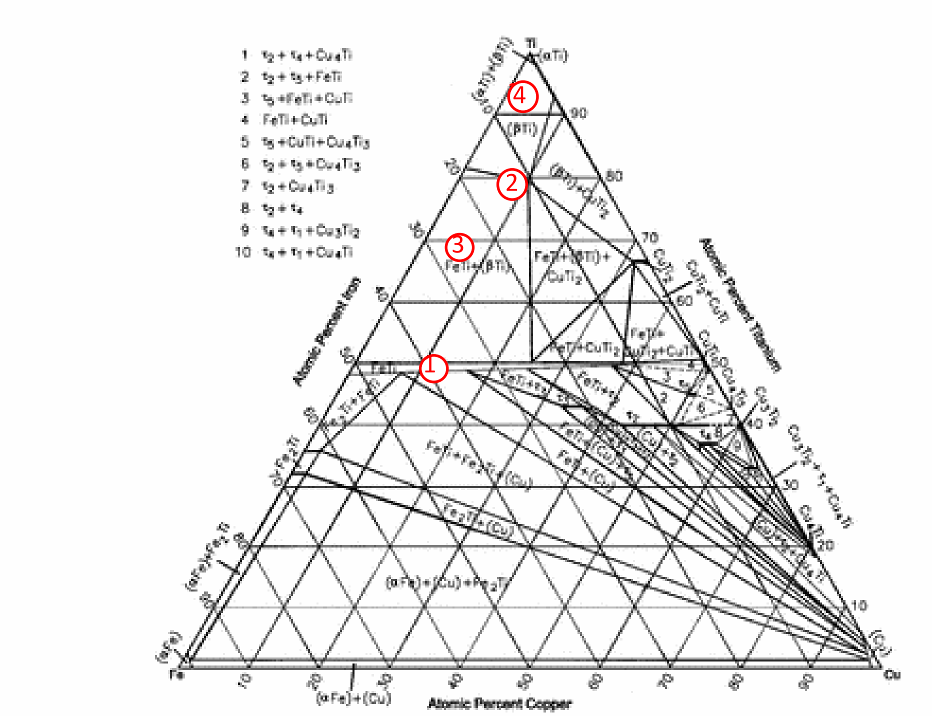
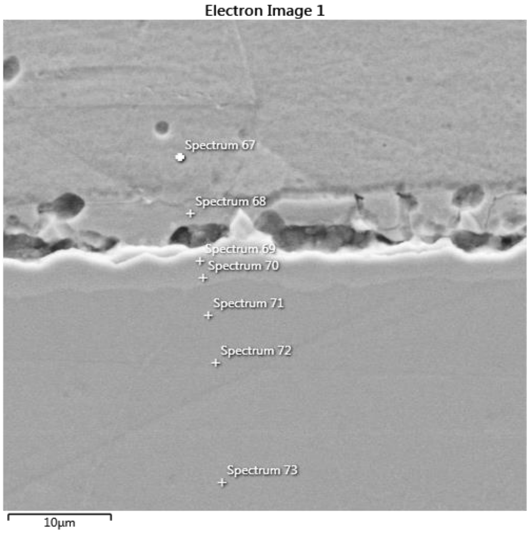


Figure 6. Ternary Fe-Ti-Cu diagram. [12]

Figure 7 shows the joint microstructure where Point 67 which located 5 µm from the joint in stainless steel side where no change was shown in base alloy. While point 68 located in the stainless-steel joint side shows high chromium content with low copper content of (1.1%). Therefore, the diffusion ability of copper in stainless steel is very low while Nickel diffused toward copper. Some spaces and vacant have existed because the low pressure cannot force the metals together to form and give sound joint.



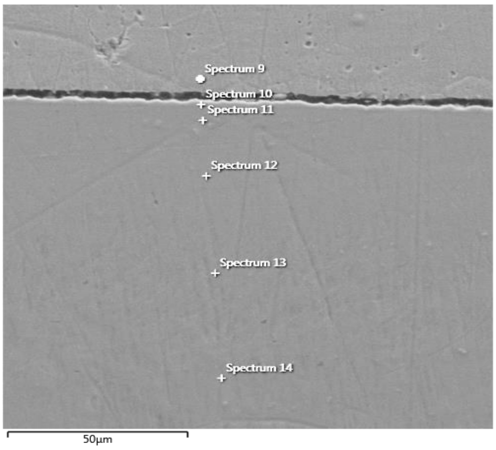
No.	Fe	Cr	Ni	Cu	Ti
67	72.7	17.9	9.4	---	---
68	69.7	27.5	1.7	1.1	---
69	33.9	5.7	4.9	10.3	45.2
70	29.9	4.3	---	10.2	55.6
71	11.3	4.2	---	8	76.4
72	10.7	3.5	---	8.7	77.1
73	11.8	3.6	---	9.5	75.2

Figure 7. The elements distribution along the diffusion bonding joint for sample (1) joined at 1.5MPa and at 850 Cº for 20 minutes

Poinst 69 and 70 are located at mid-point of the joint show high content of Titanium and Iron and by projecting these points on the ternary diagram the resulted phases are [FeTi + βTi] as indicated as (1) number in figure 6. These two phases are responsible for bonding the joint. Point 71 is indicated as number (2) on figure 6 and located 3.2 µm from joint in the Titanium side where the higher content of αTi and the remainder is FeTi compounds. Increasing the distance from the joint zone where point 72 is at 8.4 µm from joint and point 73 is 21 µm from joint zone. A decrease in FeTi phase is noticed due to relatively high copper and Iron diffusion ability in Titanium.

It can be concluded that the response phase effect on bonding strength at temperature of 850oC and the shorter diffusion time of (20 min.) is FeTi which gave low bonding shear strength. The best diffusion bonded joint strength (specimen 15) is of shear strength of 96 MPa, bonded by 3MPa at 950oC for 50 minutes at vacuum, is shown in figure 8 and analyzed for micro-chemical analysis.

Point 3 located in figure 6 represents the spectrum of points (10 and 11) in figure (8) where the dominant phases are [FeTi + βTi] but its concentration at point (1) differs from point (2) in figure 5, where the concentrations are equal or nearly equal of the two intermetallic phases.



No.	Fe	Cr	Ni	Cu	Ti
9	71.2	18.0	10.8	_	_
10	23.5	5.2	3.3	6.5	61.6
11	11.7	3.7	2.0	5.9	76.7
12	7.8	2.7	1	6.2	74.2
13	3.1	_	_	3.4	93.5
14	_	_	_	_	100

Figure 8. SEM and EDS analysis for diffusion joint cross section for sample 15, joined by 3MPa at 950oC for 50 minutes

Spectrums (12 and 13) away from the joint zone by distances of (14 and 45 µm) respectively had 7.8 and 3.1% wt. of Iron and 6.2 and 3.4% wt. of copper respectively. The dominant phases are αTi and βTi which can be considered as commercially pure titanium. The longer diffusion time leads to deeper diffusion distance of iron and copper. Also, higher pressure during diffusion process eliminated asperities of the surface and smoothened adjacent surfaces and intimate contact area was obtained which lead to higher diffusion rates and well bonded joints. Figure 9 shows a magnified region of the joint for the interface between copper and titanium. The structure of this region contains a (Ti3Cu4) Phase where its concentration
(40%Ti and 60%Cu) by projecting on the equilibrium phase diagram of (Ti-Cu).

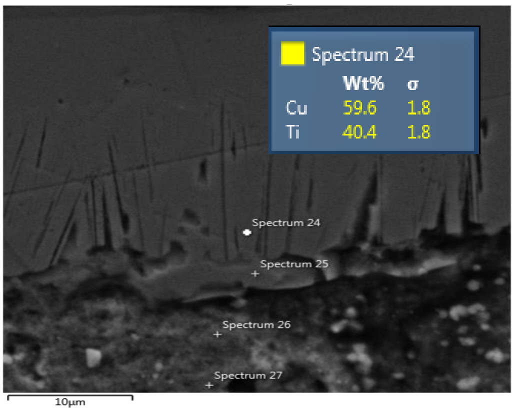


Figure 9. SEM Image (BSE) and EDS analysis for Copper-Titanium side of a Diffusion joint.

The most area of the fracture is located at the layer of (Ti3Cu4) phase. Where figure 10 shows the general fracture area of the tested joint. The fracture surface topography given in figure 11 shows two discrete regions by color contrast. When the two regions are magnified, the bright region showed a thin fractured layer as shown. By comparing the shape of this layer with the cross section of the joint, it can be seen in this region to be the same as indicated by spectrum 24 in figure 8.

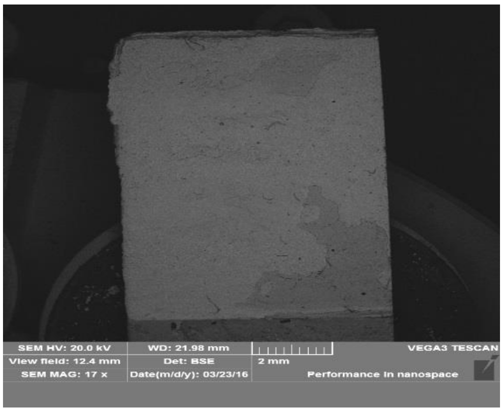


Figure 10. SEM Image (BSE) for fractured specimen (low magnification)

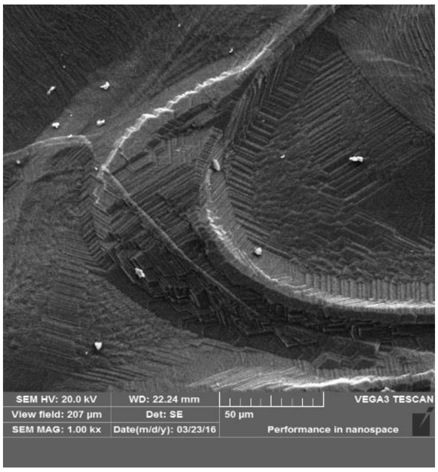
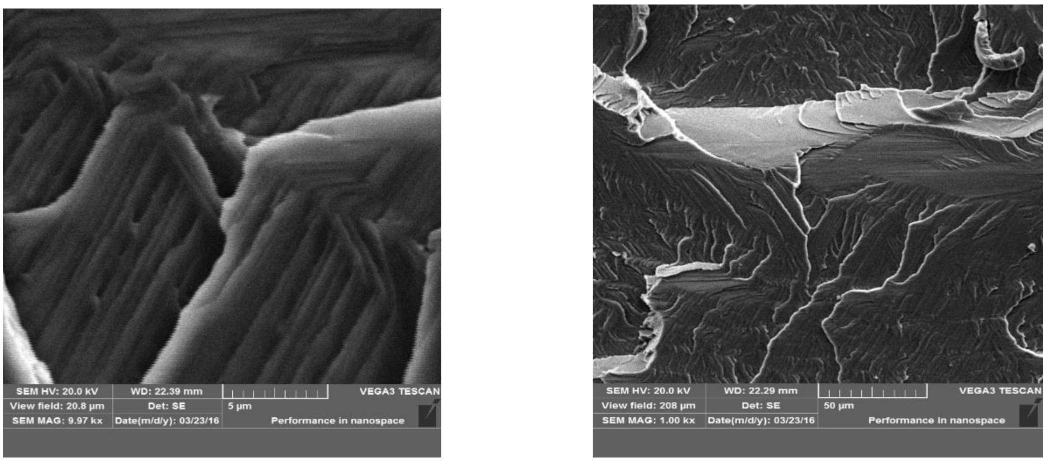


Figure 11. SEM Image for fractured bright region (Ti3Cu4).

IOP Conf. Series: Materials Science and Engineering **518** (2019) 032041 doi:10.1088/1757-899X/518/3/032041

The (Ti3Cu4) phase contains small voids (typically 0-200 nm) between these layers (crystals) which caused a decrease in the mechanical properties. As shown the fracture in figure 11 and (12A) is a brittle mode, since the (Ti3Cu4) phase is a brittle intermetallic compound. This fractured area represented (85%) of the total bonded area. The remaining fractured area was (15%). Also figure (12B) shows a fine laminar structure (Typically 200nm) in a high magnification image for the fractured surface.



A **B**

Figure 12. SEM Image for fractured Dark region (Ti_2Cu)

spectrum 33 in figure 13 shows a composition included the presence of the three metals but with another form of a complex intermetallic compound (Cu + Ti37Cu63Fex) revealed by projecting point 33 compositions on the ternary phase diagram in figure 5. This phase is different from the bright (Ti3Cu4) phase in shape and has finer structure so leads to a stronger structure than the former and possessed small fracture area percentage.

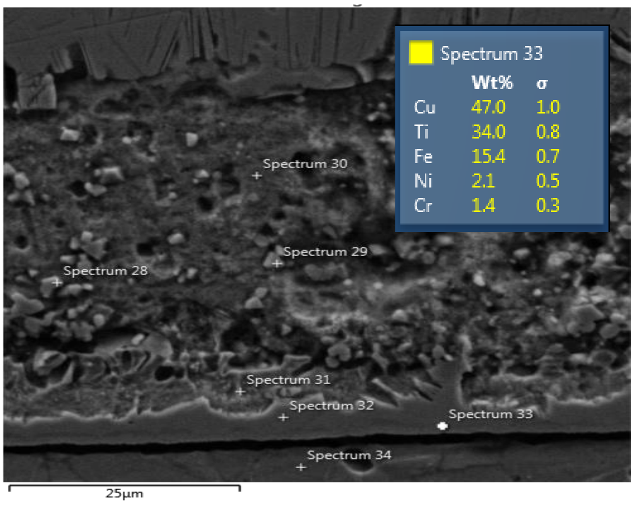


Figure 13. SEM Image (BSE) and EDS analysis of a low strength bonding joint.

4. Conclusions

From the previous discussions the following conclusions can be obtained:

1-In diffusion bonding of Stainless steel AISI 316L to pure Titanium with pure copper interlayer was efficient to give satisfied bonding joints

2- best diffusion bonding conditions were achieved to be 96MPa tensile shear strength at a bonding temperature of 950 Cº, bonding pressure of 3MPa and holding time of 50 Minutes.
3-The maximum joint hardness of 343.6 HV has achieved at 950Cº for a bonding time of 40 Minutes
4-EDS and XRD tests for the various joint zone at various bonding conditions (high and low conditions values) on projection in the ternary phase diagram of (Fe-Ti-Cu) revealed the formation of FeTi, TiCu, Ti2Cu, Ti3Cu4,αTi, βTi and(Cu + Ti37Cu63Fex) intermetallic compounds.
5-Fracture surfaces of the shear tested joints showed that the major part of the fractured area was at the Ti3Cu4 intermetallic compound due to its high hardness and brittleness. This area represented 85% of the fractured cross section area while 15% was at the Ti2Cu intermetallic compound.

References

[1] Camargo P R C, Trevisan R E and Liu S 1993 Microstructural Characterization of Titanium to 304 Stainless Steel Brazed Joints *Welding Journal* **12** s-537.
[2] Kundu S, Ghosh M, Laik A, Bhanumurthy K, Kale G B, Chatterjee S 2005 Diffusion bonding of commercially pure titanium to 304 stainless steel using copper interlayer *Materials Science and Engineering A* **407** 154–160.
[3] Debasis Poddar 2009 Solid-State Diffusion Bonding of Commercially pure Titanium and Precipitation Hardening Stainless steel *International Journal of Recent Trends in Engineering* **1**.
[4] Rahman A H M E , Cavalli M N 2010 Strength and microstructure of diffusion bonded titanium using silver and copper inter layers *Materials Science and Engineering A* **527** 5189–5193.
[5] Shuying Liu, Kuan Xu, Guangbao Liu, Congcong Cao and Zhonghao Heng 2013 The Evaluation characteristics and Numerical Analysis of Diffusion Bonding Interface structure of Titanium alloy/Cu/Stainless Steel *Adv. Mater. Sci* **33** 0224-231.
[6] Szwed B, Konieczny M 2014 Influence of diffusion bonding parameters on the structure and properties of titanium and stainless steel joints with copper interlayer *journal of Achievements in materials and manufacturing engineering* **67**.
[7] Kundu S, Sam S, Mishra B and Chatterjee S 2014 Diffusion Bonding of Microduplex Stainless Steel and Ti Alloy with and without Interlayer Interface Microstructure and Strength Properties *Metallurgical and Materials Transactions A* **45A** 373.
[8] Balasubramanian M 2015 Development of processing windows for diffusion bonding of Ti−6Al−4V titanium alloy and 304 stainless steel with silver as intermediate layer *Transactions of Nonferrous Metals Society of china* **25** 2932−2938
[9] De-feng M, Ting-feng S, Yong-jian F, Xiao-song J, Charles Q L, Machael D S and Zhi-ping L 2018 A Review on Diffusion Bonding between Titanium Alloys and Stainless Steels *Advances in Materials Science and Engineering* Volume 2018, Article ID 8701890, 15 pages.
[10] Chandrappa K, Amit K and Kumar S 2018 Diffusion bonding of a titanium alloy to a stainless steel with an aluminium alloy interlayer *IOP Conf. Series: Materials Science and Engineering* 402 012124.
[11] Zhenzhen Y, Sarah M G and Stephen L 2018 Review of Ti to Steel Dissimilar Joining *Research and Development in Material Science* **5** Issue 5.
[12] Section II phase Diagram Evaluations 2002 Cu-Fe-Ti (Copper-Iron-Titanium) *journal of phase Equilibria.* **23** 173.