Development of Empirical Relationships to Predict Strength of Powder Metallurgically Produced Pure Aluminium and Pure Copper Diffusion Bonded Bimetallic Joints

A. Murugan[1], T. Senthilvelan[2], V. Balasubramanian[3]

[1,2] Department of Mechanical Engineering, Pondicherry Engineering College, Puducherry 607 402, India,
[3] Centre for Materials Joining and Research (CEMAJOR), Department of Manufacturing Engineering, Annamalai University, Annamalai Nagar, 608 002 Tamil Nadu, India
[1] E-mail: a_murugaa@rediffmail.com

ABSTRACT

In the present study, pure aluminium (Al) and pure copper (Cu) plates prepared by powder metallurgy (P/M) method were bonded by diffusion bonding technique. From the literature, it was identified that the predominant diffusion bonding process parameters such as bonding temperature, holding time and bonding pressure influence the shear and bonding strength of diffusion bonded joints. In this investigation an attempt was made to develop empirical relationships to predict the shear strength and bonding strength of diffusion bonded bimetallic joints of pure Cu/Al incorporating the above parameters using statistical tools such as design of experiments, analysis of variance and regression analysis. The developed empirical relationships can be used to predict the strength of Cu/Al bimetallic joints at 95% confidence level.

Keywords: Pure Copper, Pure Aluminium, Powder metallurgy, Diffusion bonding, Design of experiments, Analysis of variance and Regression analysis.

1.0 INTRODUCTION

Diffusion bonding is a solid state metal joining process that is applied for electronic, aerospace and nuclear applications [1]. Aluminium (Al) has been used in aerospace and automotive industries due to its high strength and good corrosion resistance [2]. Copper (Cu) is used in power industries, electrical appliances, machineries and automobile since it has high electrical and thermal conductivity [3].

Joining these dissimilar materials by fusion welding technique is difficult, since it causes several problems such as thermal cracking, easy formation of brittle itermettalic compounds, high internal stress and distortion in the weld region. These problems can be overcome by diffusion bonding technique. Diffusion bonding process has inherent advantages over fusion welding technique as it does not form the unexpected phases at interface of the bond. The joint by diffusion bonding has many advantages such as good resistance to high temperature and it provides a novel joining operation for similar and dissimilar materials without gross microscopic distortion without minimum dimensional tolerance [4–7]. The diffusion bonding process also permits the production of high quality joints without post weld machining. Therefore, the diffusion bonding technique is preferred to join these dissimilar materials without many difficulties. Diffusion bonding process is dependent on various parameters, such as bonding temperature, holding time, and bonding pressure [8]. These parameters affect the interfacial structure, morphology and the quality of bonds [9]. Diffusion bonding can be achieved by applying a static pressure, certain amount of time at high temperature below the melting temperature of the metals.

Many of research works have been carried out to understand the effect of diffusion bonding parameters on mechanical and metallurgical characteristics of dissimilar joints of wrought alloys [13-19]. All the above mentioned investigations were carried out on trial and other basis to attain optimum welding conditions. But there is no literature available to predict strength of joints on diffusion bonding of powder metallurgically (P/M) produced pure Cu and pure Al bimetallic joints. Hence, in this investigation, an attempt was made to develop empirical relationships to predict shear strength and bonding strength of the Cu/Al bimetallic joints using statistical tools such as design of experiments (DOE), analysis of variance (ANOVA) and regression analysis.

2.0 EXPERIMENTAL WORK

Square shaped specimens (50mm x 50mm) were manufactured from pure Al and pure Cu by powder metallurgical technique. The prepared specimen thickness of Cu was 3mm and Al was 5 mm. The specimens prepared by P/M technique were machined to make flat surfaces by milling and then polished and cleaned in acetone just before diffusion bonding.

The polished and chemically treated specimens were stacked in the die which was made by 316 L stainless steel. The diffusion bonding facility available at Centre for Materials Joining and Research (CEMAJOR), Annamlai University was utilized in this investigation. The diffusion bonding die set up is shown in **Fig.1**. The specimens were heated up to the bonding temperature by induction furnace and the heating rate of furnace was 10°C/minutes. The required pressure was simultaneously applied to the certain time. Thus, the bonding was completed and then the bonding samples were cooled to the room temperature before removal from the chamber of diffusion bonding machine. In this way, joints were fabricated using different combinations of bonding temperature, bonding pressure and holding time and they are displayed **Fig. 2**. The microstructure analysis was carried out to reveal the formation of diffusion layer at the interface of the joints using optical microscope. The copper side was etched by a solution containing ethanol, $FeCl_3$, concentrated HCl, whereas the aluminum side was etched by using Keller's solution to reveal the microstructure. The chemical compositions of base metals are presented in the **Table 1**.

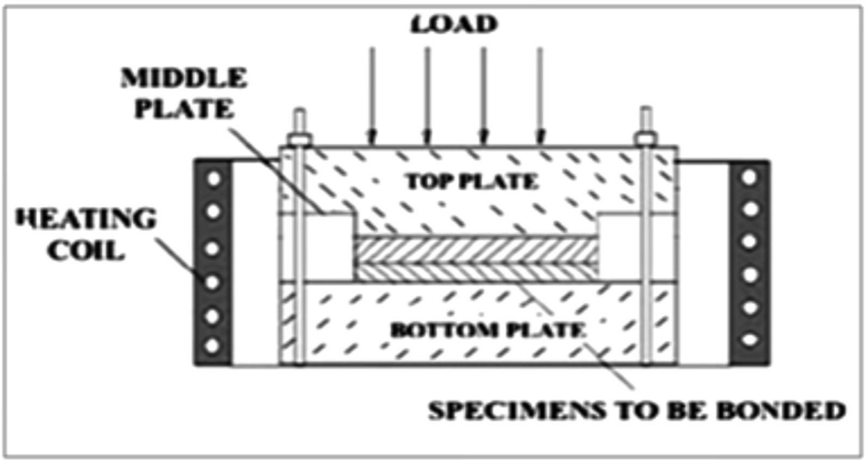


Fig 1: Configuration of the diffusion bonding die set up

Table 1 : Chemical composition of the base metal

Base metal	Si	Fe	Mn	Mg	Al	Bi	Cu	Cr	Ti	Ni
Aluminium	0.053	0.100	0.001	0.003	99.81	–	–	–	–	–
Copper	–	0.010	–	–	–	0.001	99.97	–	–	–
Nickel	–	0.00	–	–	0.178	–	0.048	0.037	0.005	99.02

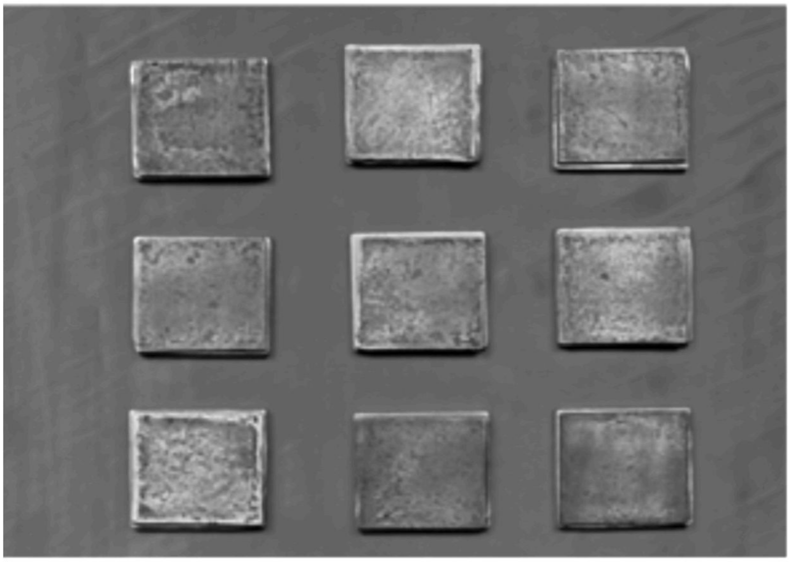


Fig. 2 : Photograph of few of the fabricated diffusion bonds

2.1 Finding the Working Limits of Diffusion Bonding Parameters

From the literature, the predominant parameters influence the diffusion bonding characteristics was identified and they are bonding temperature, bonding time, and bonding pressure. A large numbers of trial experiments were conducted to find the working limits of the above factors by varying one of the parameters and keeping the other factors constant. The working range was fixed in such way that the joints should be free from any visible external defects.

(i) No bonding was occurred between pure Al and Cu, if the bonding temperature was lower than 250 °C and this was due to the insufficient temperature to cause diffusion of atoms between these two materials (**Fig. 3a**).

(ii) If the bonding temperature was higher than 450 °C, then no bonding was occurred between these materials and this leads to the melting of pure Al due to high temperature (**Fig. 3b**).

(iii) If the bonding pressure was below 5 MPa, no bonding was occurred because less number of contact points by which diffusion of atoms occur between the materials (**Fig. 3c**).

(iv) When the bonding pressure was higher than 20 MPa, Aluminium plates deformed which causes the reduction in thickness and bulging at the edges (**Fig. 3d**).

(v) If the holding time was below 5 min., no bonding was occurred because of insufficient time which causes to take place the diffusion reaction (**Fig. 3e**).

(vi) When the holding time was greater than 120 min., the grain growth was excessive and lead to the melting of pure Al (**Fig. 3f**).

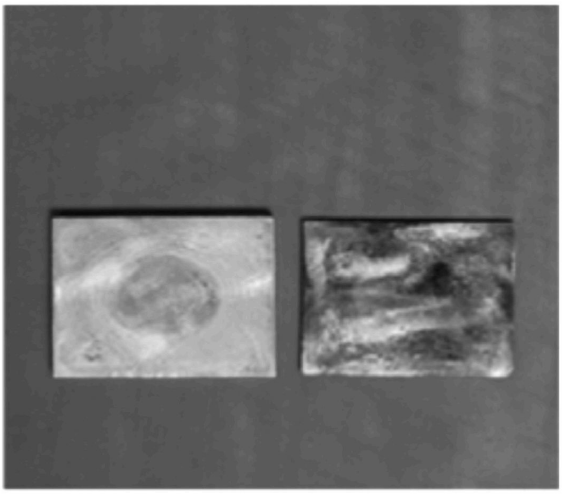
2.2 Developing Experimental Design Matrix

The feasible limits of the process parameters were chosen in such a way that all the combinations of the above materials should be bonded without any defects. As the range of individual factor was wide, a central composite rotatable three-factors, five-level, central composite rotatable design matrix was selected. The chosen welding parameters and the levels are presented in **Table 2**. The experimental design matrix consisting 20 sets of coded condition and comprising a full replication three-factor factorial design of 8 points, 6 star points, and 6 center points was used (**Table 3**). The method of designing such matrix is dealt elsewhere [20]. The upper and lower limits of the parameters were coded as +1.682 and –1.682, respectively. The coded values for intermediate levels can be calculated from the following relationship [20]

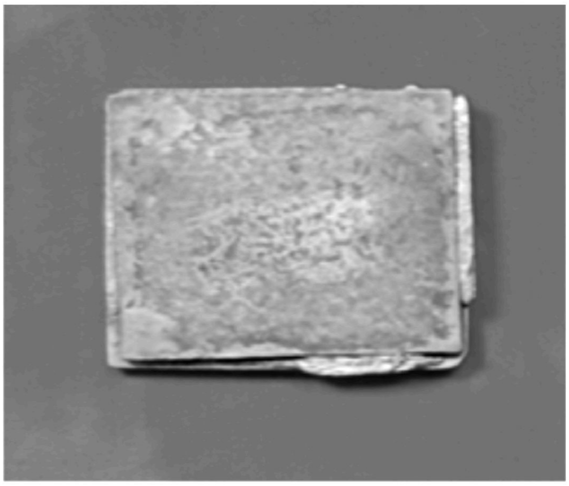
$$X_i = 1.682\,[2X - (X_{max} + X_{min})] / (X_{max} - X_{min}). \quad (1)$$

Where,

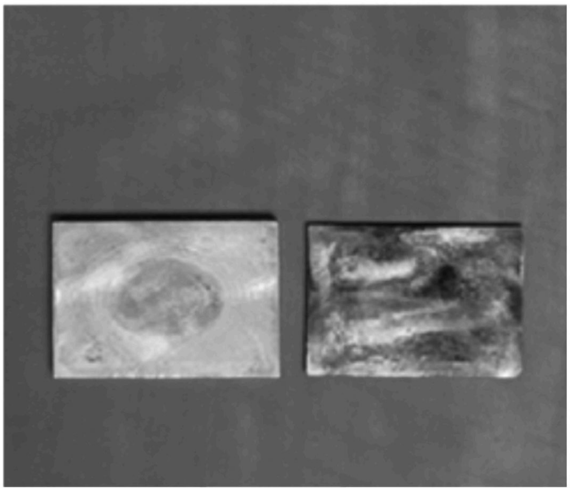
X_i is the required coded value of a variable X and X is any value of the variable from X_{min} to X_{max}. X_{min} is the lower level of the variable and X_{max}; is the highest level of the variable.



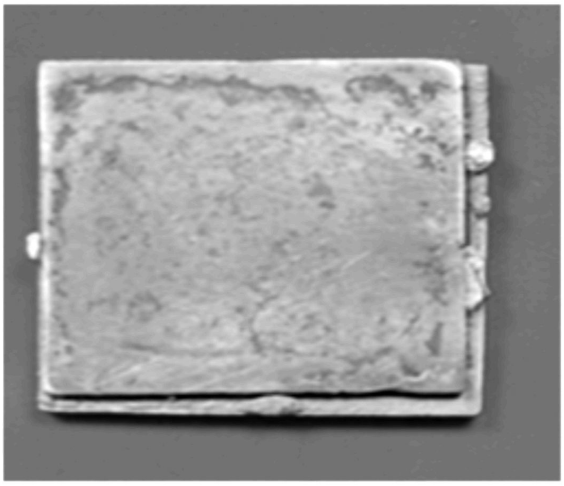
(a): Temperature < 250 ºC



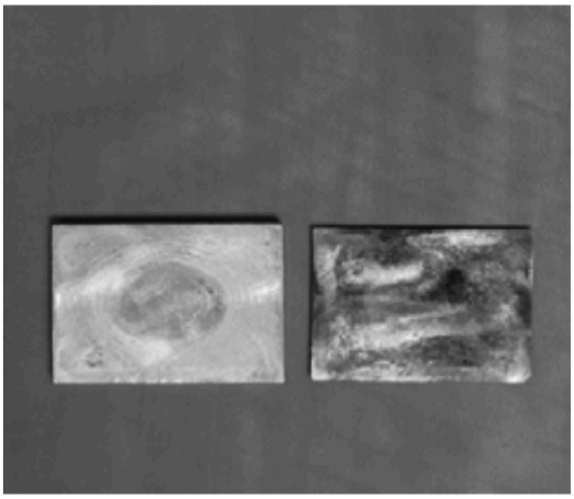
(b): Temperature > 450 ºC



©: Pressure < 5 MPa



(d): Pressure > 20 MPa



(e): Time < 5 Min

(f): Time > 120 Min.

Fig. 3: Photographs of fabricated bonds using lower and upper limit process parameter

Table 2 : Feasible working limits of diffusion bonding parameters of Cu/Al bonds

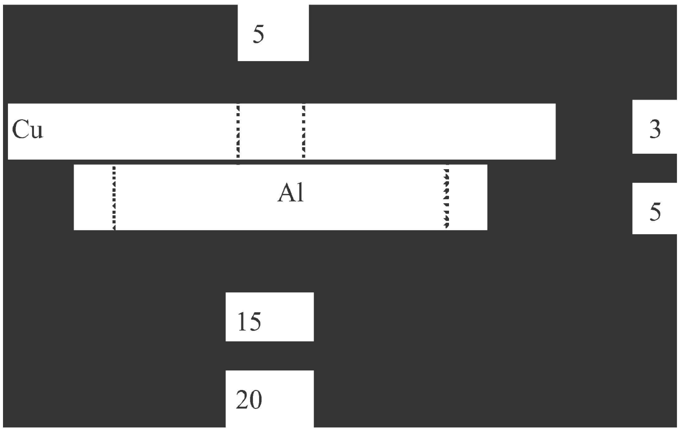
No	Parameters	Notations	Units	Levels				
				-1.682	-1	0	1	1.682
1	Bonding temperature	T	0C	266	300	350	400	434
2	Holding time	H	minutes	30	45	60	75	90
3	Bonding pressure	P	MPa	10	12.5	15	17.5	20

Table 3 : Experimental design matrix and responses of Cu/Al bonds

Exp. No	Coded values			Original values			Shear strength (MPa)	Bonding strength (MPa)
	T	H	P	T	H	P	(SS)	(BS)
1	-1	-1	-1	300	45	13	17	22
2	1	-1	-1	400	45	13	29	33
3	-1	1	-1	300	75	13	23	27
4	1	1	-1	400	75	13	33	33
5	-1	-1	1	300	45	18	20	24
6	1	-1	1	400	45	18	26	30
7	-1	1	1	300	75	18	21	25
8	1	1	1	400	75	18	24	29
9	-2	0	0	266	60	15	12	16
10	2	0	0	434	60	15	23	27
11	0	-2	0	350	35	15	24	28
12	0	2	0	350	85	15	27	32
13	0	0	-2	350	60	11	33	37
14	0	0	2	350	60	19	28	32
15	0	0	0	350	60	15	32	36
16	0	0	0	350	60	15	31	35
17	0	0	0	350	60	15	32	35
18	0	0	0	350	60	15	32	36
19	0	0	0	350	60	15	32	36
20	0	0	0	350	60	15	32	36

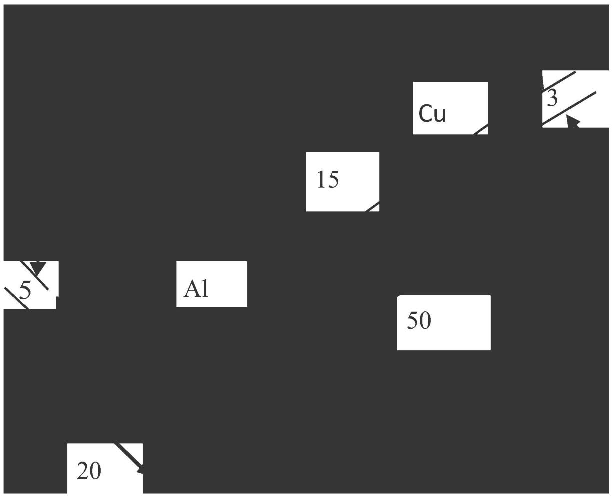
2.3 Recording the Responses (Shear strength and Bonding strength)

As per the design matrix, twenty bimetallic joints were fabricated. The joints were sliced using wire cut electric-discharge machining (WEDM) process. Three specimens were prepared from each joint for lap shear tensile and ram tensile test. Shear strength and bonding strength were evaluated by conducting lap shear test and ram tensile test respectively. The average of three values is presented in **Table 2**. The dimensions of non-standard, sub-size, lap shear tensile specimen and ram tensile specimen are shown in **Figs. (4 -5)**. The test was carried out in 100 kN servo-controlled Universal Testing Machine with a loading rate of 1.5 kN/min.



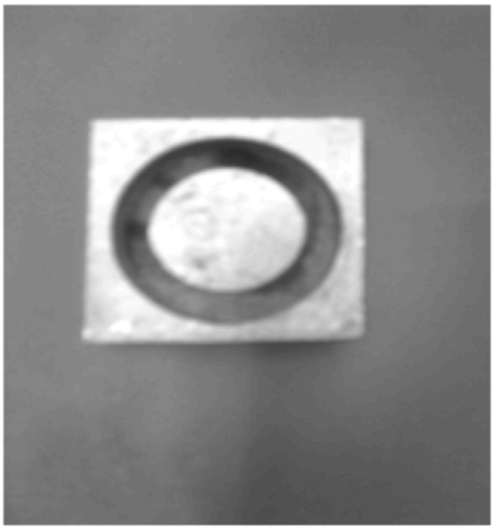
All dimensions are in

(a): Ram tensile test specimen

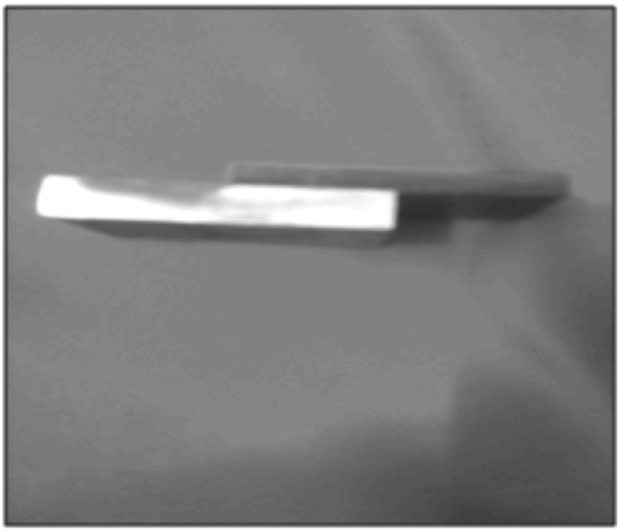


(b): Lap shear tensile test specimen

Fig 4: Dimensions of tensile test specimen



(a): Photograph of Ram tensile test specimen



(b): Photograph of Lap shear tensile test specimen

Fig. 5 : Photograph of Tensile test specimen

3.0 DEVELOPING EMPIRICAL RELATIONSHIPS

The responses functions (Y) are shear strength (SS), bonding strength (BS) and they are function of bonding temperature (T), bonding pressure (P) and holding time (H) and it can be expressed as in the mathematical form as

$$SS = f\{T,H,P\} ; BS = f\{T,H,P\}; \quad (2)$$

The second order polynomial (regression) equation used to represent the response surface 'Y' and the selected polynomial could be expressed as;

$$Y = b_0+b_1(T)+b_2(H)+b_3(P)+b_{11}(T^2)+b_{22}(H^2)+b_{33}(P^2)+ b_{12}(TH)+b_{13}(TP)+b_{23}(HP) \quad (3)$$

where b_0 is the average of responses and b_1, b_2, b_3...b_{23} are regression coefficients that depend on respective linear, interaction, and squared terms of factors. The value of the coefficient was calculated using Design Expert Software .After determining the significant coefficients at the (95% confidence level), the final relationships were developed using only these coefficients and the final empirical relationship to estimate shear strength and bonding strength of diffusion bonded Cu/Al bimetallic joints are given below:

$$SS = \{-401.37177+1.71480(T) +2.06328(H) +8.07366 (P) - 004(T*H) -0.013000 (T* P) - 0.036667(H * P) -003 (T^2) - 003 (H^2) - 0.063071(P^2)\} \text{ MPa} \quad (4)$$

$$BS = \{-360.71073+1.66575(T)+1.82541(H) +5.35037(P)- 003(T*P) -003 (T*P)-0.016667(H*P) -003(T^2) - 003(H^2) - 0.078390(P^2) \}\text{Mpa} \quad (5)$$

Table 4 : ANOVA test results for shear strength of Cu/Al bonds

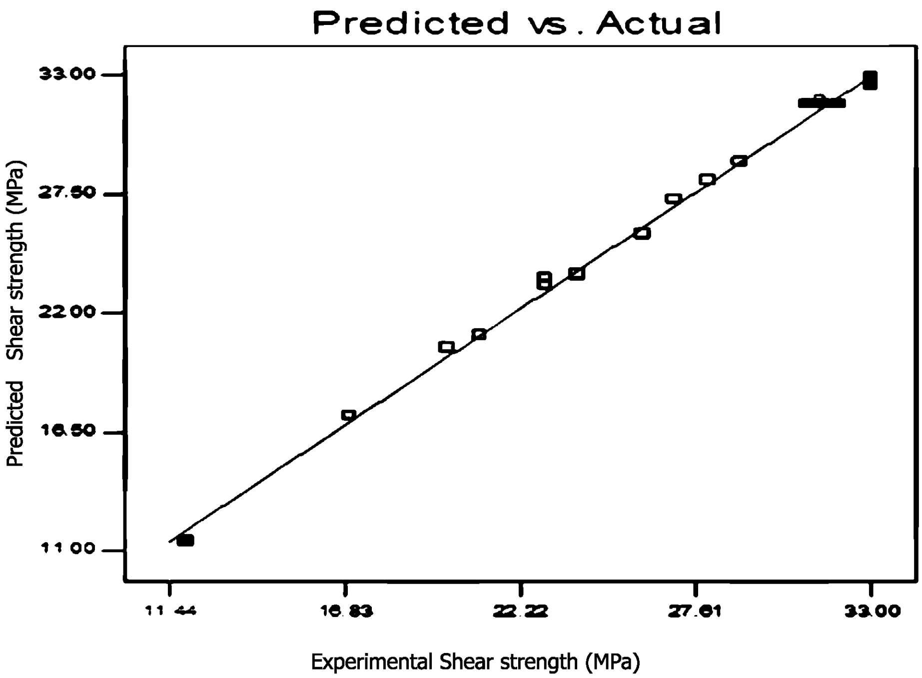
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	661.07	9	73.45	300.55	< 0.0001	significant
T	179.41	1	179.41	734.13	< 0.0001	
H	14.44	1	14.44	59.11	< 0.0001	
P	27.58	1	27.58	112.87	< 0.0001	
TH	3.12	1	3.12	12.79	0.0050	
TP	21.12	1	21.12	86.44	< 0.0001	
HP	15.12	1	15.12	61.89	< 0.0001	
T^2	358.90	1	358.90	1468.56	< 0.0001	
H^2	67.36	1	67.36	275.63	< 0.0001	
P^2	2.24	1	2.24	9.16	0.0127	
Residual	2.44	10	0.24			
Lack of Fit	1.57	5	0.31	1.79	0.2686	not significant
Pure Error	0.87	5	0.17			
Cor Total	663.51	19				
Std. Dev.	0.49	R^2		0.9963		
Mean	26.49	Adj R^2		0.9930		
C.V. %	1.87	Pred R^2		0.9798		
PRESS	13.37	Adeq Precision		61.465		
df-dgrees of freedom; F-Fisher's ratio; P- probability						

Table 5 : ANOVA test results for bonding strength of Cu/Al bonds

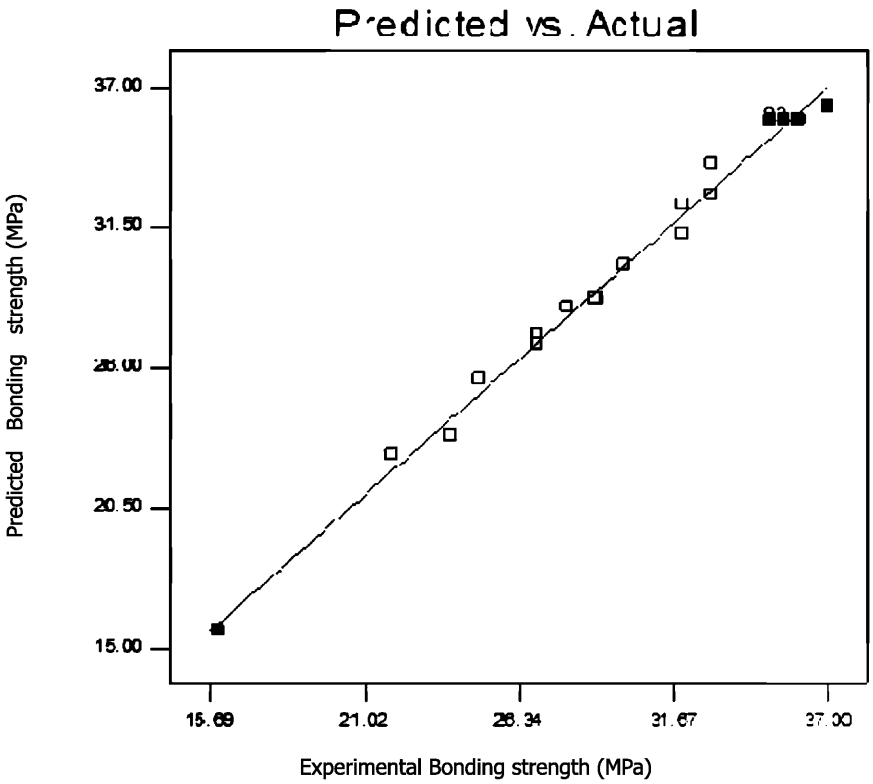
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	603.73	9	67.08	142.15	< 0.0001	significant
T	151.59	1	151.59	321.24	< 0.0001	
H	10.07	1	10.07	21.34	0.0010	
P	17.39	1	17.39	36.84	0.0001	
TH	6.12	1	6.12	12.98	0.0048	
TP	6.12	1	6.12	12.98	0.0048	
HP	3.12	1	3.12	6.62	0.0277	
A^2	372.80	1	372.80	790.02	< 0.0001	
B^2	62.40	1	62.40	132.24	< 0.0001	
C^2	3.46	1	3.46	7.33	0.0220	
Residual	4.72	10	0.47			
Lack of Fit	3.89	5	0.78	4.66	0.0582	not significant
Pure Error	0.83	5	0.17			
Cor Total	608.45	19				
Std. Dev.	0.69	R^2		0.9922		
Mean	30.45	Adj R^2		0.9853		
C.V. %	2.26	Pred R^2		0.9466		
PRESS	32.47	Adeq Precision		42.205		
df-dgrees of freedom; F-Fisher's ratio; P- probablity						

4.0 CHECKING ADEQUACY OF THE DEVELOPED RELATIONSHIPS

The adequacy of the developed relationships was tested using the analysis of variance (ANOVA) technique and the results of second order response surface model fitting in the form of analysis of variance are given in **Tables 3-4**. The determination coefficient (R^2) indicates the goodness of fit for the model. In this case, the values of the determination coefficient (R^2) indicate that the model does not explain only less than 5% of the total variations. The values of adjusted determination coefficient (adjusted R^2) are also high, which indicates a high significance of the model. Predicted R^2 is also made a good agreement with the adjusted R^2. Adequate precision compares the range of predicted values at the design points to the average prediction error. The relationships between actual and predicted responses and each observed responses of Cu/Al bonds are compared with the predicted responses calculated from the model and their respective correlation graphs are shown in **Fig. 6**. From the ANOVA **Table 4-5**, the higher F ratio value implies that the respective term is more significant and vice versa. From the F ratio values it can be concluded that the bonding temperature is contributing more on shear strength and bonding strength and it is followed by bonding pressure and holding time.

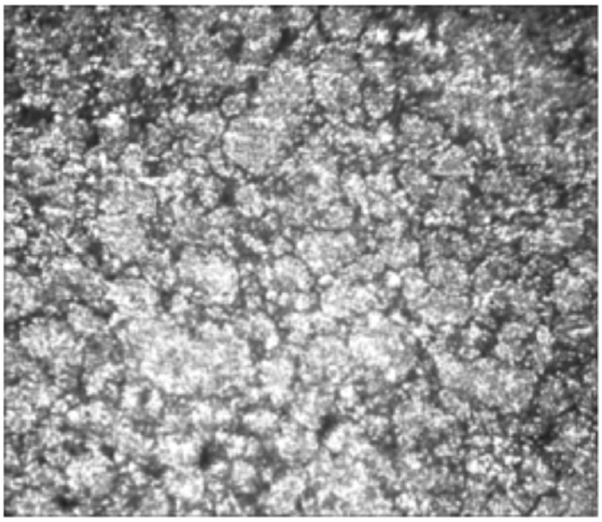


(a):Shear strength



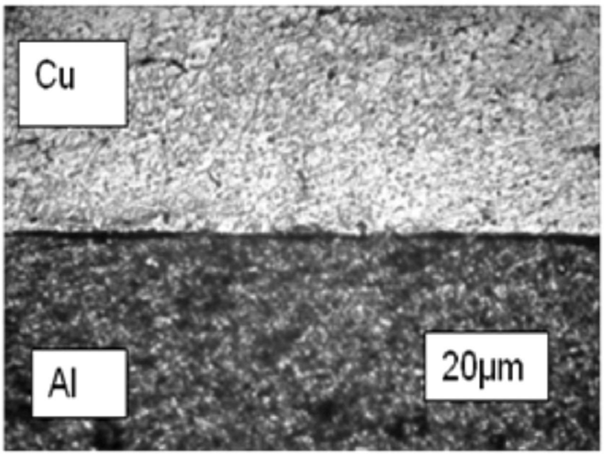
(b):Bonding strength

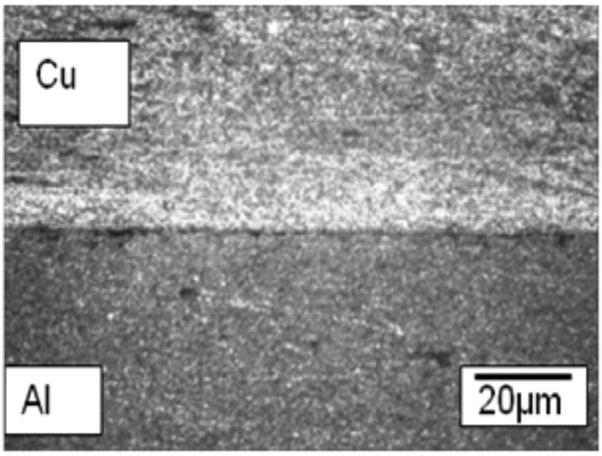
Fig. 6 : Correlation graphs



(a) Aluminium (a) Copper

Fig. 7 : Optical micrograph of base metals





(c)T=266 ^{0}C; t= 60 min; P=15 MPa (d)T=350 ^{0}C; t= 60 min; P=11 MPa

Fig. 8 : Optical micrographs of diffusion bonded of Al/Cu joints

5.0 CONCLUSIONS

(i) Empirical relationships were developed to predict the shear strength and bonding strength of diffusion bonded bimetallic joints of pure Cu/Al incorporating important parameters such as bonding temperature, holding time and bonding pressure. The developed empirical relationships can be effectively used to predict the shear strength and bonding strength of the above bimetallic joints at 95% confidence level.

(ii) Bonding temperature was found to have greater influence on shear strength and bonding strength of bimetallic joints of pure Cu/Al followed by bonding pressure and holding time.

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