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# Developing Empirical relationships to predict diffusion layer thickness, hardness and strength of Al-Cu dissimilar joints

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## ABSTRACT

The principal difficulty when joining Aluminium (Al) and commercial grade Copper (Cu) lies in the existence of formation of oxide films and brittle intermetallics in the bond region. However, diffusion bonding can be used to join these alloys without much difficulty. Temperature, pressure and holding time are the three main variables, which govern the integrity of the diffusion bonds. The experiments were conducted based on three factors, five-levels, and central composite rotatable design with full replications technique. Empirical relationships were developed to predict diffusion layer thickness, hardness, strength of Al-Cu joints incorporating process parameters using Response Surface Methodology. The developed relationships can be effectively used to predict the bond properties at 95 % confidence level.

**Keywords:** Diffusion bonding, Aluminium alloy, commercial grade Copper, lap shear tensile strength, ram tensile strength.

## INTRODUCTION

The joining of materials by conventional welding techniques becomes difficult if the physical properties such as melting temperature and thermal expansion coefficients of the two materials differ a lot, as it is necessary to have controlled melting on both sides of weld joints simultaneously. Even if this criterion is met, it may not be possible to have an appropriate joint when the two materials are metallurgically incompatible. This is because metallurgical incompatibility may lead to a weld zone and heat affected zone microstructure without adequate mechanical strength [1]. By means of diffusion bonding, it is possible to bond all of the materials whose chemical and metallurgical properties are appropriate. In particular the bonding of advanced materials is not possible by classical welding methods because of unexpected phase propagation at the bond interface [2]. Hence, diffusion

bonding introduces convenience to the bonding of materials, which are not possible to bond by conventional welding methods. Further, more diffusion bonding is preferred for the materials in which brittle phase formation is unavoidable. To obtain the desired strength, it is essential to have a complete control over the relevant process parameters to maximize the strength on which the quality of a weldment is based [3].

Therefore, it is very important to select and control the welding process parameters for obtaining maximum strength.

In order to achieve this various prediction methods can be applied to define the desired output variables through developing mathematical models to specify the relationship between the input parameters and output variables. It has been proved by several researchers [4-6] that efficient use of statistical design of experimental techniques, allows development of an empirical methodology, to incorporate a scientific approach in the fusion welding procedure. Even though sufficient literature is available on diffusion bonded Al - Cu dissimilar alloys; no systematic study has been reported so

Table 1(a) Chemical composition (wt %) of AA2024 aluminium alloy

Al	O	Fe	Pb	B	S	Cu
0.14	0.092	0.007	0.001	0.018	<0.001	Bal

Table 1(b) Chemical composition (wt %) of commercial grade copper alloy

Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.5	0.5	4.9	0.9	1.8	0.10	0.25	0.15	Bal

far to correlate the process parameters and mechanical properties of diffusion bonding of Al-Cu alloy joints. Hence, in this investigation, an attempt was made to develop empirical relationships to predict the bonding characteristics such as diffusion layer thickness, diffusion layer hardness, shear strength and bonding strength incorporating diffusion bonding process parameters such as bonding temperature, bonding pressure and holding time using response surface methodology.

## EXPERIMENTAL WORK

### Identifying the important process parameters

From the literature [7] and the previous work done [8] in our laboratory, it was found that the independently controllable primary parameters affecting the quality of diffusion bonded joints are bonding temperature, bonding pressure and holding time.

### Finding the working limits of parameters

Square shaped specimens (50 mm x 50 mm) were machined from rolled plates of 5 mm thickness aluminium (AA2024) and commercial grade copper alloys. The chemical composition of the base metal used in this investigation is presented in Table 1. The polished and chemically treated specimens were stacked in a die made up of 316L stainless steel. The entire diffusion bonding setup, shown in Fig. 1, was inserted into a vacuum chamber (vacuum pressure of 10<sup>-3</sup> mm Hg was maintained). The specimens were heated up to the bonding temperature using induction furnace with a heating rate of 25 °C/min; simultaneously the required pressure was applied. After the completion of bonding, the samples were cooled to room temperature before removal from the chamber.

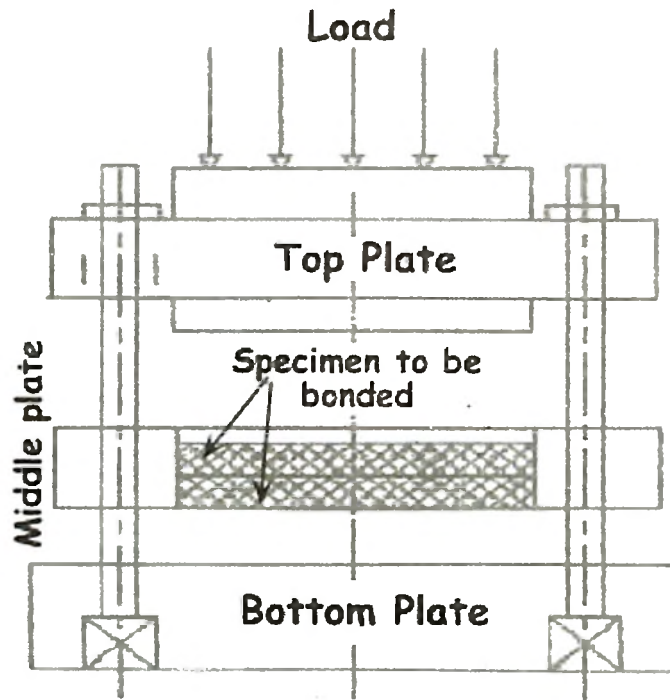


Fig. 1 Configuration of the diffusion bonding setup.

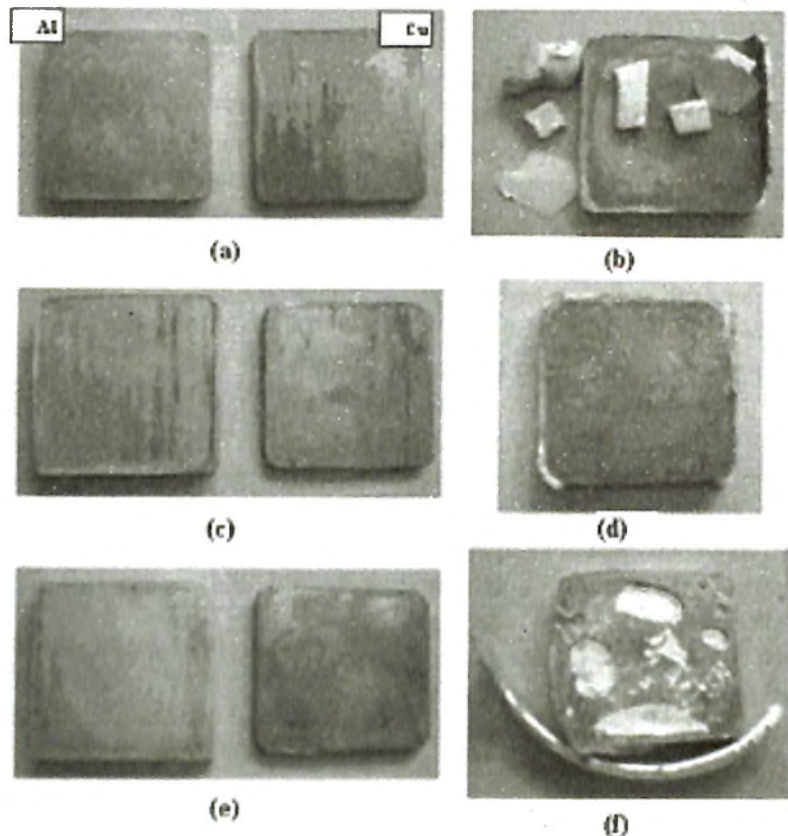


Fig. 2 Some of the joints fabricated during trial experiments

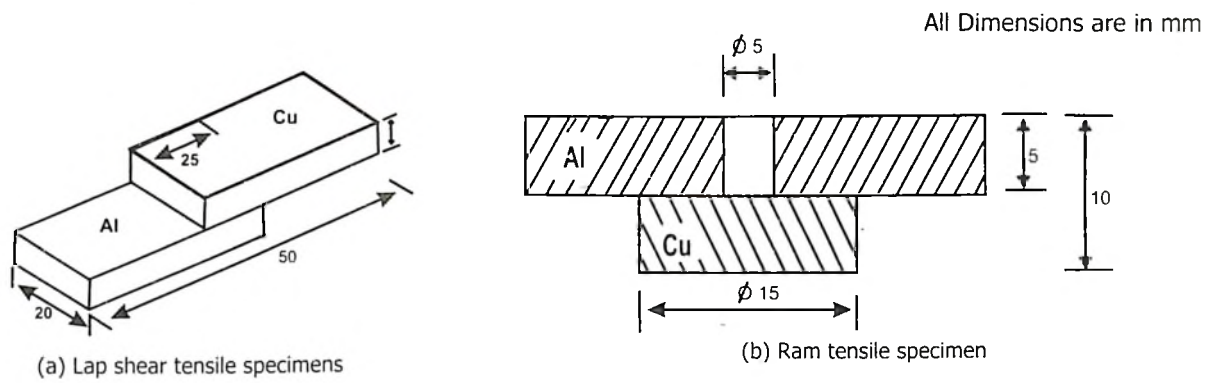


Fig. 3 Dimensions of test specimens

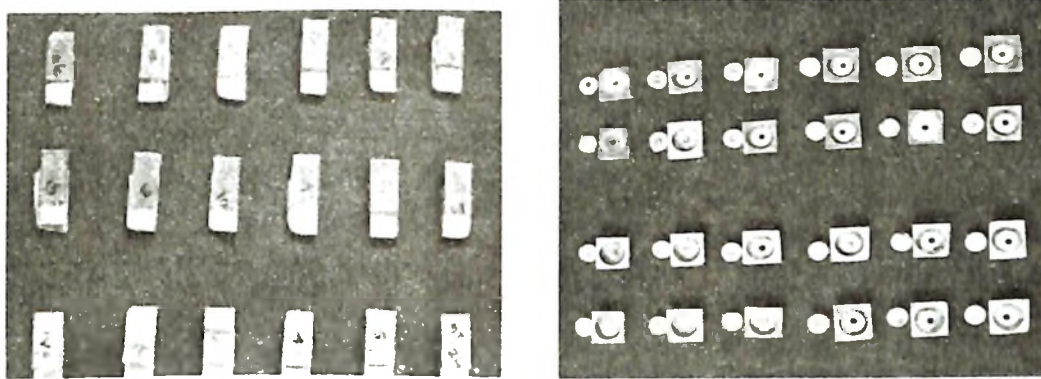


Fig. 4 Photographs of test specimens

A large number of trial experiments were carried out using the above procedures and from the experimental results the following inferences were obtained:

- (i) If the bonding temperature was lower than 450 °C, then no bonding was occurred between Al and Cu alloys and this was due to the insufficient temperature to cause diffusion of atoms (Fig. 2a).
- (ii) If the bonding temperature was greater than 550 °C, then the bonding pressure decreased automatically after few minutes and this was due to the melting of Al alloy due to higher

temperature (Fig. 2b);

- (iii) If the bonding pressure was lower than 5 MPa, then no bonding was occurred and this was due to less number of contacting points (between surface asperities) through which diffusion of atoms generally should occur (Fig. 2c);
- (iv) If the bonding pressure was greater than 20 MPa, then the plates were deformed plastically causing reduction in thickness and bulging at the outer edges (Fig. 2d);
- (v) If the holding time was less than 5 minutes, then no bonding was occurred and this was due to the

insufficient time allowed for the diffusion reaction to take place (Fig. 2e);

- (vi) If the holding time was higher than 75 minutes, then excessive grain growth followed by melting of Al alloy was observed (Fig. 2f). The bonding temperature of 450 to 550 °C, bonding pressure of 4 MPa to 20 MPa and holding time of 10 to 90 minutes yielded diffusion bonding between Al and Cu alloys. This was validated by conducting few more experiments. The process parameters and its range are presented in Table 2.

## Conducting the experiments

The design matrix chosen to conduct the experiments was a three level five factors central composite rotatable design, which is shown in Table 3.

Twenty joints were fabricated as per the conditions dictated by the design matrix. As the joints were not large enough for normal lap shear testing, a non-standard test was devised to measure the shear strength of the bonds. The dimensions of lap shear tensile and ram tensile test specimens are shown in Fig.3. Specimens were prepared from the Al/Cu diffusion bonded joints by a line cutting machine (electric spark cutting) and the photographs of the prepared specimens were depicted in Fig. 4. Lap shear test and Ram tensile test were carried out in 100 kN capacity servo controlled Universal Testing Machine (Make: FIE-Bluestar, India; Model: UNITEK-94100) and the results are presented in Table 3. Vicker's microhardness testing machine (Make: Shimadzu, Japan and Model: HMV-2T) was used for measuring the diffusion layer hardness with 0.05 kg load. Microstructural examination was carried out using a light optical microscope (Make: MEJI, Japan; Model: MIL-7100) incorporated with an image analyzing software (Metal Vision). Diffusion layer thickness was measured using the metal vision image analyzing software. The microstructures of diffusion bonded joints are presented in Fig.5.

## DEVELOPING EMPIRICAL RELATIONSHIPS

The responses diffusion layer thickness (DLT), interface hardness (IH), shear strength (SS), bonding strength (BS), are function of bonding temperature (T) bonding pressure (P) and holding time (t) and it can be expressed as;

$$DLT = f \{T, P, T\}; IH = f \{T, P, T\}; SS = f \{T, P, T\}; BS = f \{T, P, T\}; \quad (1)$$

In this present investigation, response surface methodology (RSM) was applied to develop the empirical relationships in the form of multiple regression equations to characterize diffusion bonded Al-Cu dissimilar joints. In applying the RSM, the independent variable was viewed as a surface to which a mathematical model is fitted [9]. The second order polynomial (regression) equation used to represent the response surface 'Y' is given by

$$Y = b_0 + \sum b_i x_i + \sum b_{ii} x_i^2 + \sum b_{ij} x_i x_j + e, \quad (2)$$

and for three factors, the selected polynomial could be expressed as

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

In order to estimate the regression coefficients, a number of experimental design techniques are available. In this work, central composite face centered design (Table 3) was used which fits the second order response surfaces very accurately. Central composite rotational design matrix with the star points are at the center of each face of factorial space was used, so  $\alpha = \pm 1.682$ . This variety requires 5 levels of each factor. The upper limit of a factor was coded as +1.682, and the lower limit was coded as -1.682. All the coefficients were obtained applying central composite rotatable design using the Design Expert statistical software package. After determining the significant coefficients (at 95% confidence level), the relationships were developed using only these coefficients. The developed empirical relationships to estimate diffusion layer thickness, hardness, shear strength and bonding strength are given below

For diffusion layer thickness

$$DL = \{7.83 + 2.99 * T + 0.80 * P + 1.47 * t\} \quad \mu m$$

For interface hardness

$$IH = \{74.45 + 14.11 * T + 3.4 * P + 7.70 * t - 2.16 * P^2 + 2.25 * t^2\} Hv \quad (5)$$

For shear strength

$$SS = \{58.01 + 2.37 * T - 0.612 * P + 1.152 * t - 0.875 * Tt + 1.62 * Pt - 9.61 * T^2 - 11.37 * P^2 - 8.54 * t^2\} MPa \quad (6)$$

For bonding strength

$$BS = \{97.49 + 4.2899 * T - 1.57 * P + 3.33 * t - 2.63 * Tt - 6.13 * Pt - 14.27 * T^2 - 8.09 * P^2 - 18.34 * t^2\} MPa \quad (7)$$

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 (ANOVA) are given in Tables 4-7. The determination coefficient (R2) indicates the goodness of fit for the model. In this case, the value of the determination coefficient (R2 = 0.965482) indicates that the model does not explain only less than 3% of the total variations. The value of adjusted determination coefficient (adjusted R2 = 0.934416) is also high, which indicates a high significance of the model. Predicted R2 is also made a good agreement with the adjusted R2. Adequate precision compares the range of predicted values at the design points to the average prediction error. The relationships between actual and predicted responses are shown in Fig.6.

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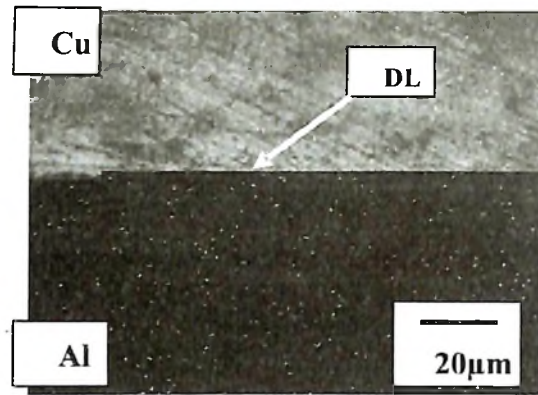


Table 3 : Experimental design matrix and results

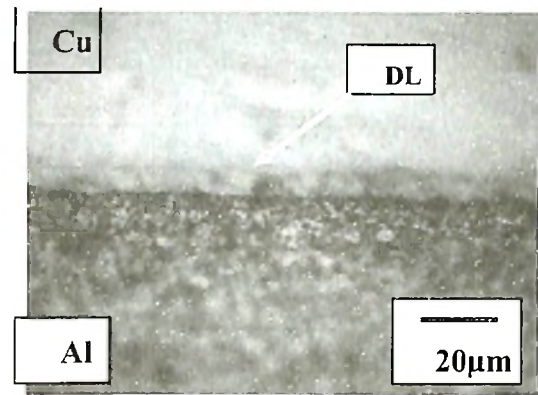
Std	Coded values			Real values			Shear strength (MPa)	Bonding strength (MPa)	Diffusion layer thickness (m)	Interface Hardness (Hv)
	T	P	t	Bonding Temperature (oC)	Bonding Pressure (MPa)	Holding time (min)	SS	BS	DLT	IH
1	-1	-1	-1	475	8	30	26	42	2	50
2	+1	-1	-1	525	8	30	33	56	8	77
3	-1	+1	-1	475	16	30	22	51	4	60
4	+1	+1	-1	525	16	30	28	65	10	85
5	-1	-1	+1	475	8	60	27	66	5	65
6	+1	-1	+1	525	8	60	30	70	11	95
7	-1	+1	+1	475	16	60	29	51	6	70
8	+1	+1	+1	525	16	60	32	54	12	100
9	-1	0	0	450	12	45	27	50	2	50
10	+1	0	0	550	12	45	35	64	12	98
11	0	-1	0	500	4	45	27	77	5	62
12	0	+1	0	500	20	45	25	72	8	73
13	0	0	-1	500	12	15	32	40	5	66
14	0	0	+1	500	12	75	36	51	11	94
15	0	0	0	500	12	45	58	99	9	75
16	0	0	0	500	12	45	58	98	8	74
17	0	0	0	500	12	45	57	96	6	73
18	0	0	0	500	12	45	59	97	8	76
19	0	0	0	500	12	45	58	97	8	74
20	0	0	0	500	12	45	58	98	8	75

Table 4 : ANOVA test results for responses

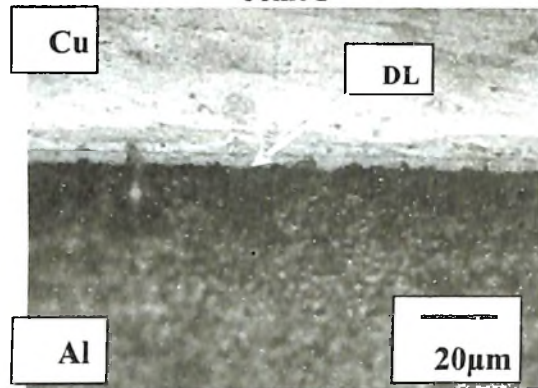
Terms	Diffusion Layer thickness (DLT)	Interface hardness (IH)	Shear strength (SS)	Bonding strength (BS)
<b>First order terms</b>				
Sum of squares (SS)	160.4866	3686.758	100.3582	436.1251
Degrees of freedom (df)	3	3	3	3
Mean square (MS)	272.2116	2165.682	419.51	436.1251
<b>Second order terms</b>				
Sum of squares (SS)	4.500697	157.5331	4273.001	9081.927
Degrees of freedom (df)	6	6	6	6
Mean square (MS)	7.633921	92.5384	17861.68	15095.55
<b>Error terms</b>				
Sum of squares (SS)	4.833333	5.5	2	5.5
Degrees of freedom (df)	5	5	5	5
Mean square (MS)	0.966667	1.1	0.4	1.1
<b>Lack of fit</b>				
Sum of squares (SS)	1.062323	11.52354	0.392273	0.516293
Degrees of freedom (df)	5	5	5	5
Mean square (MS)	0.212465	2.304708	0.078455	0.103259
<b>Fratio</b>				
Prob > F	< 0.0001	< 0.0001	< 0.0001	< 0.0001
R2	0.9654	0.9956	0.9993	0.9992
<b>Model</b>	Significant	Significant	Significant	Significant



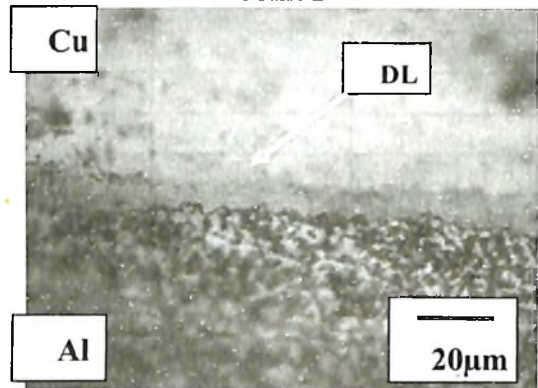
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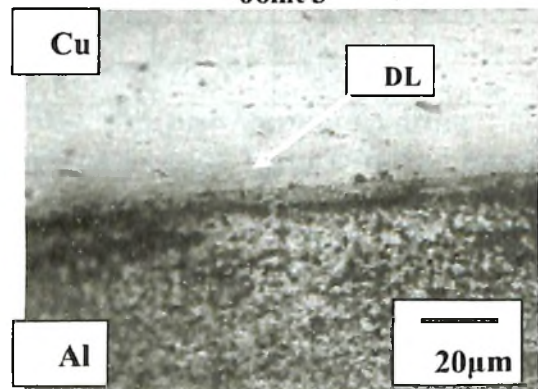
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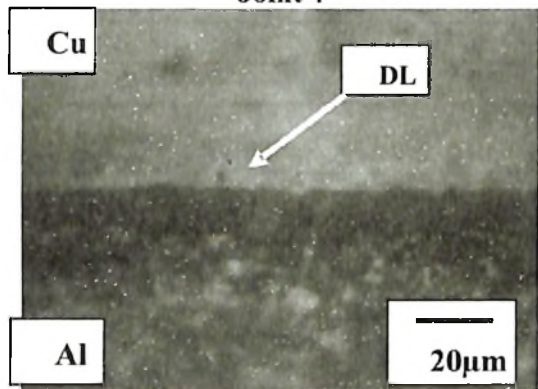
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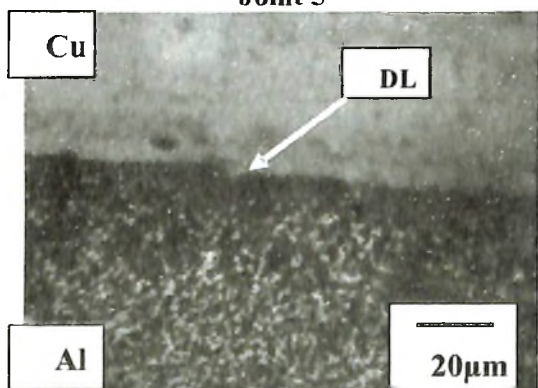
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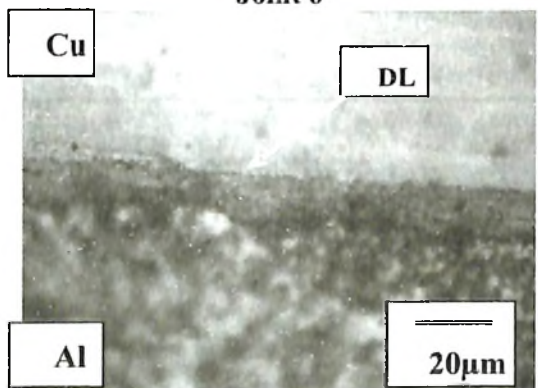
Joint 5



Joint 6

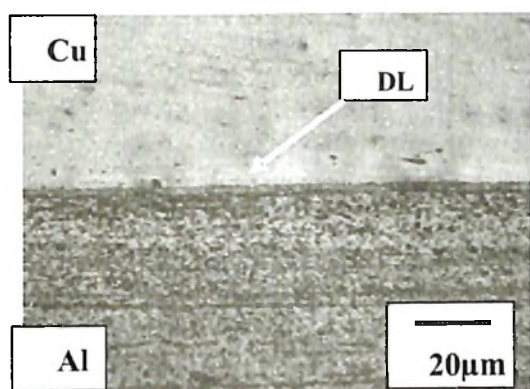


Joint 7

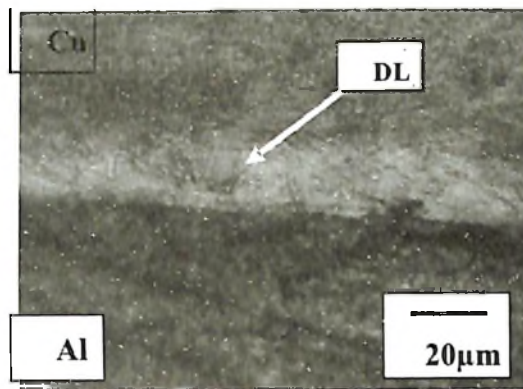


Joint 8

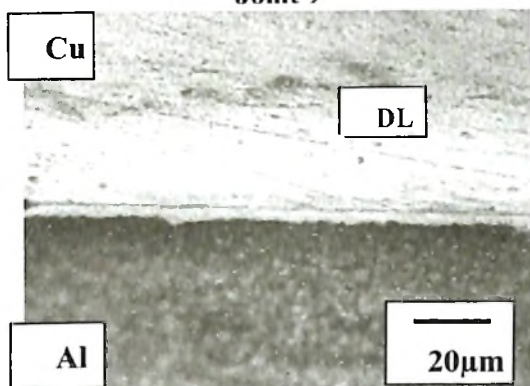
Fig. 5 Optical micrographs of interface region of Al-Cu bonds



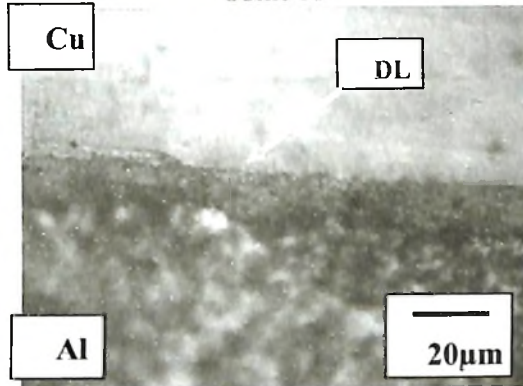
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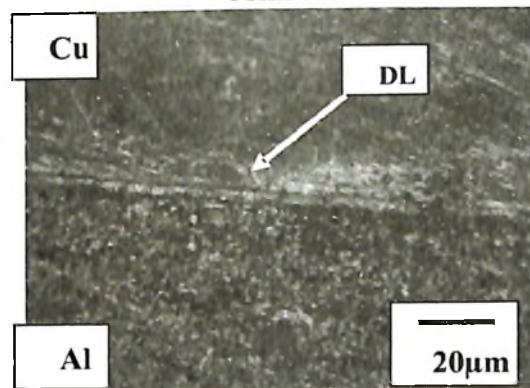
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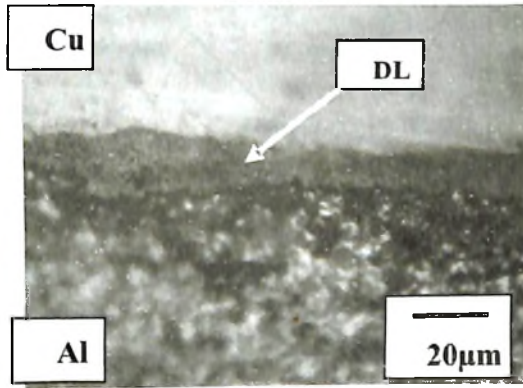
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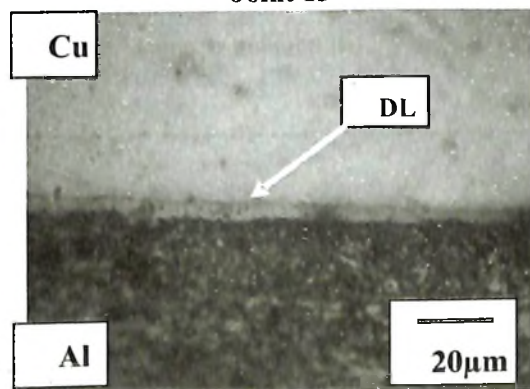
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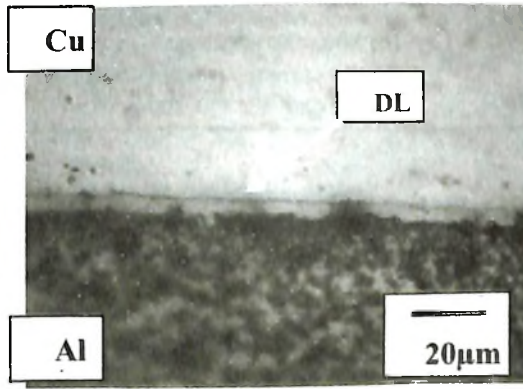
Joint 13



Joint 14



Joint 15



Joint 16

Fig. 5 Optical micrographs of interface region of Al-Cu bonds

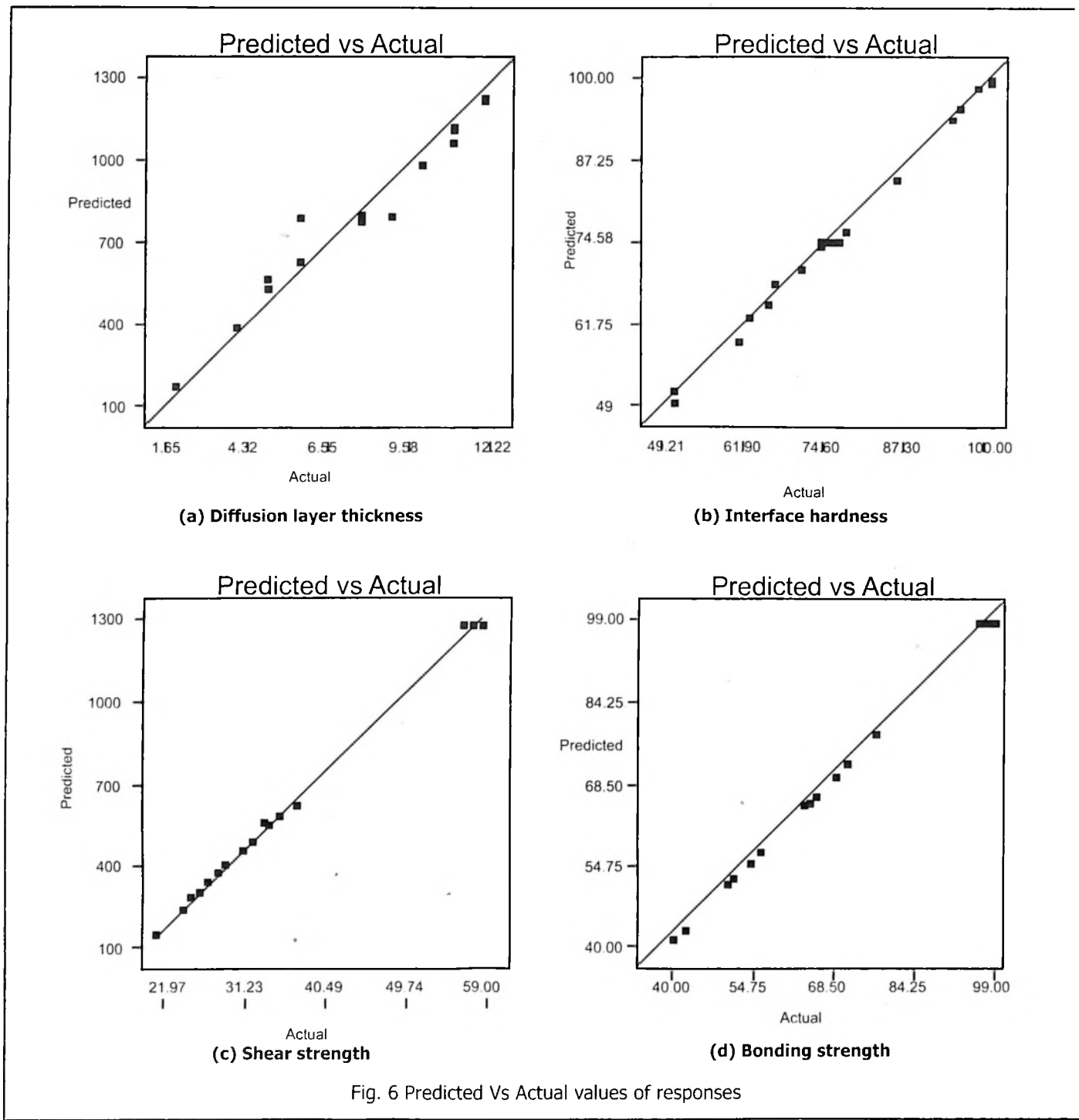


Fig. 6 Predicted Vs Actual values of responses

**CONCLUSIONS**

From this investigation, following important conclusions are derived.

- Empirical relationships were developed to predict (at 95% confidence level) the diffusion layer thickness, interface hardness,

bonding strength and shear strength, of the Al-Cu diffusion bonded joints by incorporating diffusion bonding process parameters using statistical tools such as design of experiments, analysis of variance and regression analysis.

- From the ANOVA test results, it is understood that the bonding temperature has greater influence on the bonding characteristics than bonding pressure and bonding time.
- From the experimental results, it is found that the joints fabricated at a

bonding temperature of 500 oC, a bonding pressure of 12 MPa and a holding time of 50 minutes exhibited superior shear and bonding strength of 59 and 99 MPa respectively. This may be due to the formation of optimum diffusion layer thickness and hardness.

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