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Diffusion Bonding and Estimation of Microbiologically Influenced Corrosion of Aerospace Grade Aluminium Alloy

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Abstract: Diffusion bonding process is an emerging solid state joining process in which the material that is being welded does not melt and recast. Rolled plates of 5 mm thick AA5083 aluminum alloys were fabricated by diffusion bonding. Empirical relationships were made to predict the bonding strength of AA5083/AA5083 aluminium alloys integrating diffusion bonding parameters such as bonding temperature, bonding pressure and holding time. The microbiologically influenced corrosion (MIC) behaviour of aluminium alloy is estimated in natural aqueous environment. An optical microscopy is used to confirm the micro colony formation of rod and cocci bacterial species. Polarization study is also carried out to determine the shift in free corrosion potential of metallic structure due to bio film formation. Corrosion rate is determined by finding the intersection point of E_{corr} and I_{corr} in the polarization curve. It is observed that the bonding and shear strength increased with the increase in bonding temperature and bonding pressure mainly due to the diffusion of atoms in the interface. Furthermore, The viable cell counts after one month are averaged $1810x10^7CFU/ml$ and the species Pseudomonas found dominant.
Keywords: Diffusion bonding, Aluminium Alloys, Bonding Strength, Bio-corrosion and Pseudomonas.

I.   INTRODUCTION

Diffusion bonding provides a novel joining operation for similar (Al-Al alloys)and dissimilar materials (Al-X alloys) without gross microscopic distortion and with minimum dimensional tolerance, the bond strength increased with the increase in bonding temperature and this is essentially due to the increase in the width of the brittle intermetallic compounds[1,2].The bond specific strengths achieved were dependent on interface grain boundary migration and grain growth during the bonding process, and these were considered to be the main mechanisms by which the initial bond interface was removed[3,4].

Strengths are believed to have occurred because of variations in the amount of liquid gallium used[5]. Bonding temperature due to the formation of finer size intermetallic compounds and good bonding between mating surfaces, Increases in the joining temperature cause the volume fraction of intermetallics to increase. These intermetallics lower the strength of diffusion bonds when proposed at higher temperature[6]. Bonding time increases the hardness of the joint interface increases due to intermetallic compounds formation, with the increasing of bonding time, the shear strength of the joints increases due to diffusion of atoms in the interface[7].Hence, the researchers[8].recommend diffusion bonding technique to join these dissimilar materials. The selection of diffusion bonding process variables affecting the interface structure, compound formation and morphology is critical to attain good quality bonds. The predominant process parameters in diffusion bonding process are: (bonding) temperature (bonding) pressure and (holding) time[9].

However, the bonded aluminium alloys shows more susceptible to corrosion by attack from halide ions such as chlorides. This susceptibility to localized corrosion appears to make aluminium alloys vulnerable to MIC. Contaminants in the fuel such as surfactants, water and water-soluble salts may contribute to bacterial growth. The two mechanisms for MIC of aluminium alloys have been documented: the production of water-soluble acids by bacteria and fungi, and the formation of differential aeration cells[10].

Microbial colonization of metal surfaces causes severe change in the ions concentration, pH, conductivity, and redox potential, altering the passive or active behaviour of the metallic substratum and its corrosion products, as well as the electrochemical variables. Micro organisms influence corrosion by changing the electrochemical conditions at the metal–solution interface. These changes may have different effects, ranging from the induction of localized corrosion to corrosion inhibition through a change in the rate of general corrosion[11].. A proper identity of bacteria by which corrosion may be exploited on metal surface and role of microbial contaminants in the specific environment as a useful tool to prevent frequent MIC effects[12]..

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Very few investigations evaluated the diffusion bonding of aluminium materials. Moreover, those literatures are focusing on microstructure analysis, phase formation studies, hardness survey at the interface and their subsequent influence on bonding strength. Hence, the present investigation was carried out to develop an empirical relationship to predict the bonding strength of diffusion bonded joints of aluminium alloys incorporating process parameters like binding temperature, bonding pressure and holding time. In addition, it also assessed to elucidate microbial corrosion behaviour of aluminium alloy (AA5083) in natural aqueous environment. Biomass is evaluated as a spectrophotometric analysis of carbohydrate and protein content. The bacterial colonies are expressed as colony-forming units per ml (CFU/ml). The bacterial species are identified up to genus level by employing morphological and biochemical characterization.

II. EXPERIMENTAL WORK

A. Abricating the Joints and Preparing the Specimens

Rolled plates of 5 mm thick AA5083 aluminium alloys are used in this investigation. The chemical composition and mechanical properties of the AA5083 are presented in Tables 1& 2. The plate was cut to the required size (50 x 50 mm) by power hacksaw followed by milling. The bonding surfaces of samples were ground flat by 200, 400 and 600 grit SiC papers and cleaned in acetone prior to diffusion bonding. The polished and chemically treated specimens were covered at the bottom and top with a mica sheet (RUBY) in a die made up of H-13Tool Steel, and were inserted into a vacuum chamber (vacuum pressure of 29Hg was maintained).The specimenis heated up to the bonding temperature by using the induction furnace with a heating range of 25ºC/min. simultaneously; the required bonding pressure was applied with the holding time. After the completion of bonding, the samples are cooled to room temperature before taking away from the chamber. The parameters used for the diffusion bonding are bonding temperature, bonding pressure and holding time.

Table 1 Chemical Composition (wt. %) of AA5083 aluminium alloy

Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Others	Al
0.26	0.35	0.05	0.60	4.74	0.11	0.05	0.06	0.01	Balance

Table 2 Physical and Mechanical Properties of AA5083 aluminium alloy

Density (g/cm^3)	Melting Point (^{o}C)	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	Poisson's Ratio	Crystal Structure
2.7	665	290	145	22	0.3	FCC

B. Developing the Experimental Design Matrix

Due to an extensive range of factors, the use of three factors and a central composite rotatable design matrix were chosen to minimize the number of experiments. The design matrix consists 20 sets of coded setting (comprising a full replication three factorial of 8 points, six corner points and six center points) was chosen in this investigation. Table 3 represents the range of factors considered, and Table 4 shows the 20 sets of coded and actual values used to conduct the experiments. For the ease of observing and processing experimental data, the upper and lower levels of the factors were coded here as +1.682 and -1.682. The coded value of each intermediate value could be calculated using the following relationship.

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

where, X_i is 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 ofthe variable, X_{max} is the upper level of the variable.

Table 3 Important factors and their levels

S. No	Factor	Unit	Notation	Levels				
				-1.682	-1	0	1	1.682
1	Bonding Temperature	°C	T	490	496	505	514	525
2	Bonding Pressure	MPa	P	5	7	10	13	15
3	Holding Time	min.	t	15	21	30	39	45

C. Recording the Responses

After the bonding process, the samples were prepared from the Al/Al diffusion bonded joints using a wire-cut EDM, the tensile test was carried out in 50 KN capacity servo controlled universal testing machine. Determine the mechanical properties of the bonding and shear-tensile test were carried out on the diffusion bonded samples, to measure shear strength of the joints, lap joint samples were prepared in accordance with ASTM Standard D1002-99. The diffusion bonded side was etched with a Keller's solution (3ml HCl, 2 ml HF and 90 ml distilled water). The test specimens were polished in disc polishing machine for scratch fewer surfaces and the surface.Biofilms are consented to grow on metal surfaces using 1% of glucose as nutrient to natural lake water under laboratory conditions. The contamination of water is avoided by adding 25 ml of fresh natural lake water after the same quantity of water is pipetted out from the system for every five days. The chemical constituents of water sample, such as, water temperature, pH, conductivity and dissolved oxygen concentration are measured throughout the experiment. All the experiments are carried out at constant atmospheric conditions. An UV-VIS spectrophotometer is used for carbohydrate and protein content analysis (M/s Shimadzu, Japan). Micro colony confirmation is done by an optical microscopy with CCD camera (M/s Shimadzu, Japan). Enumeration of viable cell can be counted by plate count technique using digital colony counter (M/s ELICO, India). The polarization studies are carried out using Potentiostat/Galvanostat (M/s Princeton, Model No. 362). All the chemicals used in this experiment, are purchased from M/s Fluka.

D. Biomass Measurement

Biofilm samples for mass analysis are obtained from the outer surface of each metal at the end of experiments. Biomass data represents the average of ten measurements from the same sample. The biofilm samples from the respective metal are subjected for the analysis of carbohydrates and protein. The analysis of carbohydrates involves the treatment of samples with sulphuric acid to cause the hydrolysis of glycosidic linkages and to dehydrate the monosaccharide in order to allow the reaction with anthrone to yield blue-green coloured complex which is measured calorimetrically at 660 nm[13].Bradford protein assay is based on the observation that the absorbance maximum for an acidic solution of Coomassie Brilliant Blue G-250 shifts from 465 nm to 595 nm when binding to protein occurs[14].The metal biofilm samples are also subjected to determine the presence of sulphate reducing bacteria. The detection of sulphate reducing bacteria in biofilm samples can be performed using the method developed by the American Petroleum Institute.

E. Identification of Bacteria

The biofilm samples are carefully scraped with a non-metallic spatula and are serially diluted. Then the samples are plated on nutrient agar medium to enumerate the heterotrophic bacteria[3].. The bacterial colonies are expressed as colony-forming units per ml (CFU/ml). The most numerous similar colonies from all plates are identified through their morphological and biochemical characteristics[16].

F. Electrochemical Study

The biofilm growth on metal surface can influence a change in free corrosion potential and also corrosion current. Corrosion potential and corrosion current of biofilm adhered aluminium alloy is measured against a saturated calomel reference electrode (SCE). The working electrodes constitute test material of 1 cm^2 of test area. All the experiments are carried out at constant temperature with natural lake water as an electrolyte. The slope of the polarization curve can be used to characterize the degree to which any stage of anodic or cathodic processes is inhibited. The coordinates of this point define the free corrosion potential of the system, as well as total corrosion current ($I_{corr}=I_{anodic}=I_{cathodic}$). The intersecting point of E_{corr} and I_{corr} in the polarization curve can be used to determine the corrosion rates through the Faradays law equation (1) [16].

$$\text{Corrosion Rate (CR)} = (I_{corr} * M)/zAF\rho \qquad (1)$$

where CR is the corrosion rate (mm/yr), I_{corr} is the corrosion current (A), M is the molar mass of metal (g/mole), A is area of electrode (mm^2), z is number of electrons transferred per metal atom, F is the Faraday's constant and ρ is the density of metal (g/mm^3).

III. DEVELOPING AN EMPIRICAL RELATIONSHIP

In the present investigation, to correlate the diffusion bonding parameters and the bonding (BS) strength of the joints, a second order quadratic model was developed. The response (bonding strength) is a function of bonding temperature (T), bonding pressure (P) and holding time (t) and it could be expressed as,

$$\text{Bonding Strength (BS)} = f(T, P, t) \qquad (2)$$

The empirical relationship must include the major and interactive effects of all factors and the selected polynomial becomes:

$$Y = bo + \sum bi\, xi + \sum bii\, xi2 + \sum bij\, xi\, xj \qquad (3)$$

For three factors, the selected polynomial can be expressed as

BS = 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) (4)

where b0 is the average of responses (BS) and b$_1$, b$_2$, b$_3$… b$_{11}$, b$_{12}$, b$_{13}$… b$_{22}$, b$_{23}$, b$_{33}$, are the coefficient that depend on the respective the major and interactive factors, which are calculated using the mathematical expression as,

$$Bi = \sum (Xi, Yi) / n$$

where, 'i' ranges from 1 to n, in which Xi is the equivalent coded value of a factor and Yi is the equivalent response output value (BS) obtained from the experiment and 'n' is the total number of combination measured. All the coefficients were obtained from central composite rotatable design matrix using DX10, design expert statistical software package. Once the significant coefficients were determined (at 95% confidence level), the final relationship was developed using only these coefficients. The final empirical relationships obtained by the above procedure to estimate the bonding and shear strength of diffusion bondedAA6082 aluminium alloy is given below,

Bonding Strength (BS) = 25.18 + 3.35(T) + 1.39(P) + 0.92(t) - 0.84(TP) + 0.24(Tt) + 1.44 (Pt) + 1.82(T^2) + 0.44(P^2) + 0.83(t^2)
(5)

The Analysis of Variance (ANOVA) technique was used to find the important main and interactive factors. The second order response surface model is fitted using Analysis of Variance (ANOVA) are given in Table 5. The coefficient (r^2) indicated the goodness of fit for the model. The Model F-value of bonding and shear strength are 27.86 implies the model is significant respectively. There is only a 0.01% chance that a Model F-Value could occur due to noise. The ''Prob> F'' obtained should be less than 0.05 indicate that the model factors are significant. In this case bonding strength includes T, P, t, TP, Pt, T^2 and t^2 are significant model term. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The ''Lack of Fit F-value'' of bonding and shear strength are 2.05 implies the Lack of Fit is not significant relative to the pure error respectively. There is a 22.46% chance that a ''Lack of Fit F-value'' of bonding strength this large could occur due to noise. Non-significant Lack of Fit is good. The Predicted R-Squared of bonding strength are 0.7819 in reasonable agreement with the Adjusted R-Squared of 0.9271; i.e. the differences are less than 0.2. Adeq Precision measures the signal to noise ratio. P ratio greater than 4 is desirable. The ratio for bonding strength are 19.178 indicates an adequate signal.

IV. RESULTS AND DISCUSSION

A. Effect of Bonding Temperature

Table 4 shows the effect of temperature on bonding strength at constant bonding pressure and holding time of 10 MPa and 30 minrespectively. It is observed that, with the increase in temperature, the bonding increases. This is attributed to the grain growth. It is also observed that, an increase in temperature from 490 to 505°C was accompany by only a small increase in strength, while a rapid increase in strength occurred when the bonding temperature was raised from 505 to 520°C, which is close to the best possible superplastic temperature range.

Additional increase in temperature decreases the variability in specific strength with less effect on the average strength. This was recognized to the association of diffusion coefficient of atoms with temperature and therefore the acceleration and facilitation of the diffusion process at elevated temperatures[17]. But rising the temperature has a drawback too. The interface of solid-state welded of AA5083 aluminium alloys is at risk to the nucleation and growth of intermetallic compounds and the formation of oxide layers at temperatures greater than 520°C.

This is a thermally activated process and by increasing the temperature the nucleation and growth of intermetallic compounds and formation of oxide layers are accelerated. These compounds have a nonmetallic covalence bond and therefore are brittle and can weaken the bonding strength[17,18].

As it can be seen that, at temperatures higher than 500 °C at a holding time of 39 min or more, the extension of these intermetallic compounds and also the oxide formations due to the vacuum-free environment, leads to the joint strength degradation. It is apparent in above diagrams that a bonding temperature of 520 °C in conjunction with a holding time of 30min, leads to the highest shear strength value.

B. Effect Of Bonding Pressure

The effect of bonding pressure on bonding with bonding temperature 505°C and holding time 30 min are shown in the Table 4. It is observed that the bonding strength increases, as the pressure increases. However, the further increase in bonding pressure decrease the ability of the holding time required to get good bonds, without considering the bonding temperature. Thus bonding pressure is one of the desirable parameter for diffusion bonding to oppose the specimen roughened surface. The diffusion paths are created mainly due to movement of atoms.

The influence of pressure with respect to temperature was found as the re-crystallization behavior of a material; it decreases the re-crystallization temperature and this increase in bonding temperature will enhance the grain boundary re-crystallization and growth of the grain[19]. The effect of bonding pressure on strength is less when being compared with temperature and time. The clarification is that the bonding surfaces are not at all absolutely smooth; they are always rough to some extent. When such surfaces are brought together, they contact only at the protrusions on the bonded surface, so the contact rates and the strength of the bonded joint are lower.

Table 4 Design matrix and Experimental results

Experiment No.	Bonding Temperature T (°C)	Bonding Pressure P (MPa)	Holding Time T (min.)	Bonding Strength BS (MPa)
1	496	7	21	19.14
2	514	7	21	27.94
3	496	13	21	21.14
4	514	13	21	25.24
5	496	7	39	18.14
6	514	7	39	26.54
7	496	13	39	24.54
8	514	13	39	29.71
9	490	10	30	19.64
10	520	10	30	30.94
11	505	5	30	18.44
12	505	15	30	23.74
13	505	10	15	20.44
14	505	10	45	23.94
15	505	10	30	22.14
16	505	10	30	21.14
17	505	10	30	20.14
18	505	10	30	20.14
19	505	10	30	20.14
20	505	10	30	20.14

C. Effect of Holding Time

Table 4 shows the effect of holding time on bonding of AA5083 aluminum alloy with bonding temperature 505°C and pressure 10 MPa respectively. It is seen that, with the increment in time, the strength increases. These results show that when holding time increases from 30 to 45 min., bonding strength make bigger with press deformation. In general, holding time has little effect on the contact ratio. Increase in bonding time increases the bond strength, when the bonding temperature and press deformation are stable. This allows the atoms to move farther on the average. Hence, diffusion bonding is better. The contact ratio however, is not considerably increased. A longer holding time has advantage on shear strength because atom movement will eventually reach equilibrium with a given change in microstructure and shorter holding time corresponds to a higher production rate with the immunity of from other benefit mentioned earlier[20].

D. Bacteria Identification

The biofilm structures of all the three metal samples are confirmed on examination under a computer enhanced optical microscopy with CCD camera. The CCD image of dominant bacterial species *Pseudomonas* is shown in Figure 1.
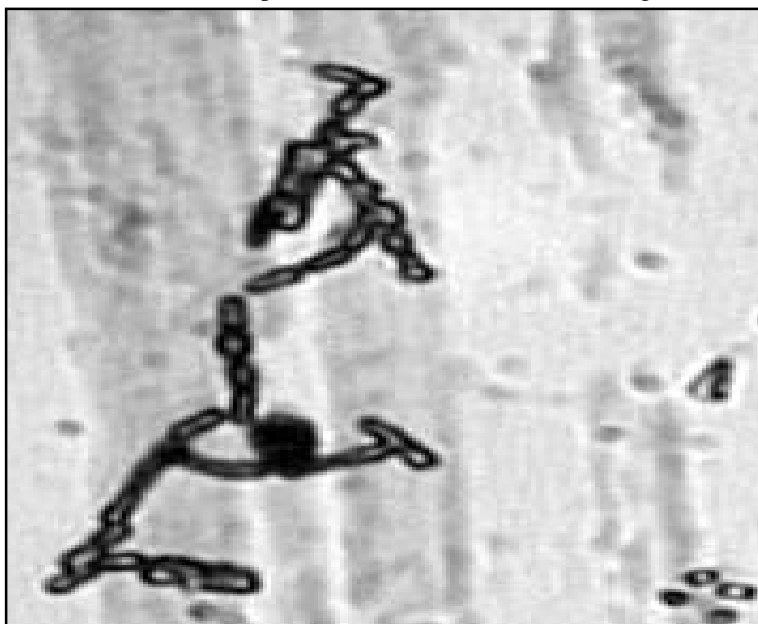

Fig. 1 CCD image of dominant bacterial species *Pseudomonas*

A mixture of micro colony formation, including rod shaped bacteria and cocci are identified through microscopic analysis. The viable cell counts after one month are averaged 1810×10^7 CFU/ml. The biofilm sample is subjected to find out the presence of sulphate reducing bacteria. Based on the results of sulphate reducing bacteria determination, it is confirmed that the biofilm sample is not constituted with sulphate reducing bacteria. The changes in chemical constituents of water, such as pH, conductivity and dissolved oxygen after one month are averaged given in the Table 5.

Table 5Analysis of Chemical Constituents of Water

Chemical Constituent					
pH		TDS (µS)		Dissolved Oxygen (mg/L)	
Initial	Final (Average)	Initial	Final (Average)	Initial	Final (Average)
7	7.8	664	772	1198.52	675.44

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E. Corrosion Assessment by Polarization Experiment

The test coupon exposed to natural lake water is subjected to polarization experiments. The potentio dynamic polarization curve of aluminium alloy immersed in natural lake water is shown in .fig.2
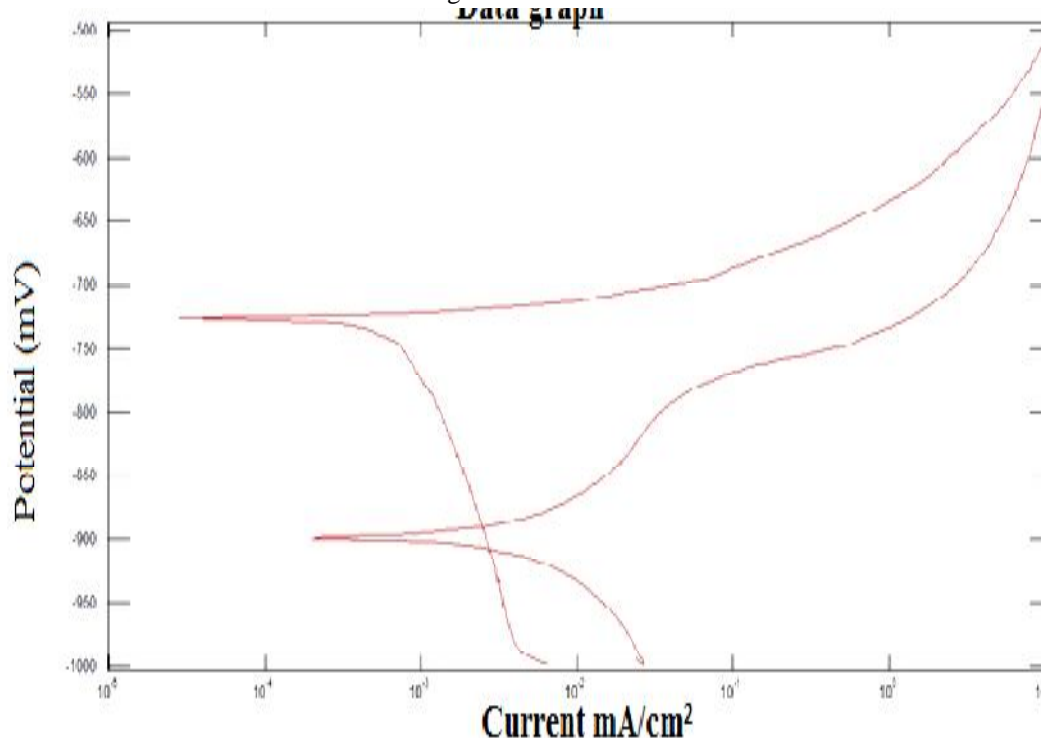

Figure2:CorrosionGraph ofAA5083

The E_{corr} value achieved is typical for this experimental procedure with the aluminium alloy having a potential of -890 mV relative to SCE, as the base metal shows -720 mV. The corrosion current density is the measure of corrosion activity. From the polarization curves, the corrosion current density (I_{corr}) is determined by Tafel extrapolation method.

Passive behaviour is not prevalent for test sample after one month period, but exhibited Tafel behaviour, indicating the surface is under polarization control. The obtained anodic current density is probably a result of the decreasing surface aluminium oxide film on the sample surface. The intersecting point of open circuit potential and corrosion current can be used to determine the microbial corrosion rate of aluminium alloy (AA5083).

Table 6Analysis of Chemical Constituents of Water

Biomass (mg/ml)		Cell Count (CFU/ml)	Dominant Species	E_{corr} (mV) vs. Ag/AgCl)	I_{corr} (mA/cm^2) x 10^{-3}	Corrosion Rate (mm/year)
Carbohydrate	Protein					
0.3935	0.1914	1810×10^7	*Pseudomonas*	-890	4.8	0.021

The corrosion rate of aluminium alloy after one month immersed in natural lake water is estimated 0.021 mm/year. Table 6 summarizes the obtained results for this experimental procedure. It should be noted that the experiment is carried out with constant laboratory conditions in 1% glucose nutrient to natural lake water.

V. CONCLUSIONS

A. Empirical relationships were developed to predict the bonding and shear strength of the diffusion bonded joints of AA5083 aluminium alloys incorporating important parameters. The developed relationship can be effectively used to predict the bonding and shear strength of diffusion bonds at 95% confidence level.

B. The optimized/maximum bonding strength of 30.94 MPa obtained under the condition of bonding temperature 520°C, bonding

pressure 10 MPa and holding time 30 min.

C. It was observed that the bonding strength increases with the increase of temperature, pressure and holding time. Bonding temperature was found to have greater influence on bonding strength of the joints followed by bonding pressure and holding time.

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