Journal of Manufacturing Engineering, March, 2017, Vol. 12, Issue. 1, pp 41-48

OPTIMIZING THE PARAMETERS OF AZ91/SICP REINFORCED COMPOSITE BY ACCUMULATED DIFFUSION BONDING PROCESS

* Sivaramakrishnan V[1], Rajamuthamilselvan M[2] , Ramanujam N[3], Ramanathan S[4] and Rajakumar S[5]

[2,4,5]Department of Manufacturing Engineering, Annamalai University, Chidambaram, Tamil Nadu, India
[1,3]Department Of Mechanical, E.G.S. Pillay Engineering College, Nagapattinam, Tamil Nadu, India.

ABSTRACT

AZ91 Magnesium matrix with 15% weight fractions of SiC_p(10µm) reinforced composite were produced through accumulative diffusion bonding (ADB) process. ADB process parameters such as bonding temperature, bonding pressure and holding time of the bond specimen play a major role to determine the bond strength. In this investigation an attempt was made to develop empirical relationships to predict the lap shear strength of diffusion bonding of AZ91/SiC_p incorporating above said parameters. Box–Behnken design was applied to optimize the diffusion bonding process parameters to attain the maximum shear strength of the bond. From this investigation, it is found that the bonds fabricated with the bonding temperature of 400◦C, bonding pressure of 10MPa and holding time of 60 min exhibited maximum shear strength of 85 MPa. Shear stress fracture and the interface between the magnesium matrix and SiC was characterized by scanning electron microscopy (SEM) and Energy dispersive spectrum (EDS) analysis. The results show a refined matrix structure of the composite compared to the matrix alloy that no reaction takes place during the synthesis of the magnesium matrix with SiC_p reinforced composites.

Keywords: *Composites, Accumulative diffusion bonding, shear strength, bonding strength*

1. Introduction

The attractive properties of magnesium and its alloys are their low density, high strength to weight ratio in cast form or wrought form [1]. Recently, as a result of general requirement for lighter weight automobiles to conserve energy, there has been a growing use of magnesium in the automobile field. The extraordinary growth in magnesium structural casting is explained by the need of car manufactures to lower fuel consumption while increasing comfort and safety of cars [2]. Reduction of car weight may be achieved by utilization of light weight structural magnesium castings.

Reinforcement of magnesium alloys with ceramic particulates has engineered a new family of materials that are marketed under the trade name metal-matrix composites offers one possibility to overcome these deficiencies [3].

The excellent mechanical properties of these materials, together with weight saving (using reinforcement with lower density than the metal matrix) and relative low cost in production makes them very attractive for a variety of engineering applications [4]. The reinforcement used can take the form of continuous fibers, whiskers, short fibers or particles. Low density ceramics, e.g. boron carbide, silicon carbide, and alumina are materials which have been produced in these various forms. The fiber reinforced composites offer the highest specific stiffness along the reinforcement direction, while particulate reinforced composites are more isotropic in the properties and are also easier to the process via powder metallurgical or casting route. Silicon carbide particulates remain the most commonly selected reinforcement because of its cost, compatibility with magnesium matrix and high modulus [3]. There are different techniques that can be used for the fabrication of metal matrix composites such as diffusion bonding, powder metallurgy in the solid state [5], perform infiltration compo casting or stir casting. The disadvantages of these processes, compared to solid state processes, are generally related to higher processing temperature used, which result in a greater propensity for matrix with reinforcement chemical reactions. If the matrix powder is large relative to the reinforcement, the reinforcing particles will agglomerate in the interstices of the coarse particle, and will be very in homogeneously distributed in the final product [3]. Also composites produced through powder metallurgical (PM) process did not show

*Corresponding Author - E- mail: siva_vms78@yahoo.co.in

high elongation. The reason may be due to fine oxide particles dispersed in the magnesium matrix. The main difficulty when joining magnesium (Mg) and other alloys by fusion welding lies in the formation of oxide films and brittle intermetallic in the bond region which affects the integrity of the joints. However diffusion bonding is a suitable process to join these two materials as no such characteristic defects are produced at the joints [6]. The diffusion bonding process parameters such as bonding temperature, bonding pressure, holding time, and surface roughness of the specimen play a major role in determining the shear strength.

The purpose of this study is to produce silicon carbide (SiC) particles dispersed AZ91 magnesium alloy composite having improved ductility. Casting process may cause heterogeneous dispersion of SiC particles because of the difference in density between SiC of 3.214 g/cm^3 and magnesium of 1.7 g/cm^3

In the present study, we propose an accumulative diffusion bonding, ADB, process for production of SiC particle dispersed magnesium alloy composite using neither magnesium melt nor powder. The application of powder interlayer makes it possible to vary their composition to ensure the correspondence of the chemical composition of the bonding materials [7]. DB can be carried out through powder layers of both the chemical compounds, pure metals and also chemically active materials. Advantage of the ADB process except for mechanical properties is mass productivity. The ADB process is applicable on large-sized structural materials because of its simple hot pressing process. Therefore, the ADB processed AZ91 alloy composite plate has a potential to practical structural applications. The ADB of composites could be realized with satisfactory casting quality. Nevertheless, up to now, it is still quite necessary to carry on further fundamental investigation on the casting characteristics of discontinuously reinforced magnesium alloy matrix composites by vacuum diffusion bonding process [8]. Accordingly, the present work was undertaken to magnesium based metal matrix composite (AZ91) reinforced with 15% various weight fractions of SiC particulates using diffusion bonding technique. In this study AZ91/SiC_p composite shear strength, fracture analysis, and the particle/matrix interfacial reaction between SiC_p and AZ91-matrix were investigated by using SEM and EDS-analysis.

2. EXPERIMENTAL PROCEDURE

2.1 ACCUMULATIVE DIFFUSION BONDING

Square-shaped specimens (50x50 mm) were machined from rolled plates of 4 mm thick magnesium (AZ91) alloy from ingot. The chemical composition of the base metal (AZ91) used in this investigation is shown in Table 1. The SiC particulates with average sizes of 10 μm were selected as the reinforcement phase. Three plates were surface treated by a stainless steel wire brush with cleaned using acetone and stacked with SiC particles. The stacked plates were uniaxially hot pressed to 10 mm height (Fig.1). The test materials used in the present investigation were SiCp reinforced magnesium alloy matrix composites manufactured by means of an accumulative diffusion bonding (ADB). The bonding surfaces of samples were ground flat by different grit SiC papers and cleaned in acetone before diffusion bonding [9]. Then the polished and chemically treated specimens were stacked in a die made up of 316L stainless steel and experimental set-up for diffusion bonding, shown in Fig.1, was inserted into a vacuum chamber. The specimens were heated up to the bonding temperature using induction furnace. Simultaneously the required pressure was applied. After completion of bonding, the samples were cooled to room temperature before removal from the chamber.

Table 1. Chemical Composition (wt%) of base metal

Al	Zn	Mn	Cu	Si	Fe	Ni	Mg
9.08	0.72	0.26	0.07	0.15	0.02	0.001	Bal.

2.2. PLAN OF EXPERIMENTS

The design of experiments technique is a very powerful tool, which permits us to carry out the modeling and analysis of the influence of process variables on the response variables. The response variable is an unknown function of the process variables, which are known as design factors [10]. Response surface methodology (RSM) was applied to optimize the diffusion bonding process parameters to attain the maximum shear strength of the joint [11]. An important stage in response surface model generation by RSM is the planning of experiments.

Table 2. Diffusion bonding parameters

Factor	Notation	Units	Low Coded	High Coded	Low Actual	High Actual
A	T	^{o}C	-1	1	300	500
B	P	MPa	-1	1	9	11
C	H	min	-1	1	40	80

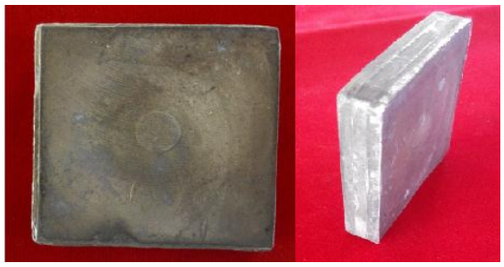

Fig.1 The AZ91/SiC$_p$ accumulated diffusion bonding specimen

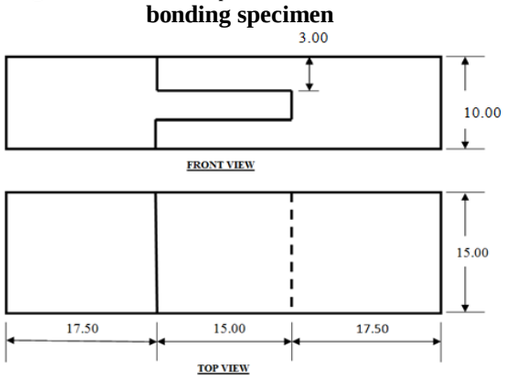

Fig.2.1 Schematic drawing of Shear test specimen

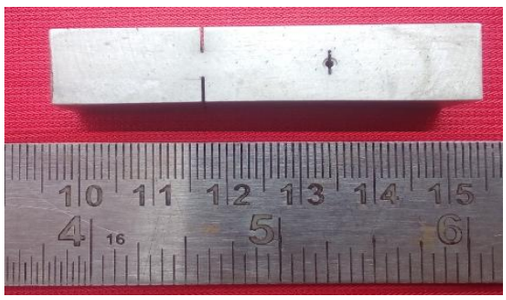

Fig. 2.2 A photograph of the shear test specimen.

Fig. 2.3 A photograph of the fractured specimen

The factors which have a significant influence on bond strength of diffusion bonding process were identified they are bonding temperature, bonding pressure, holding time. Large numbers of trial runs were carried out to determine maximum and minimum values of ADB process parameters are given in Table 2. The shear test specimen schematic drawing as shown in fig.2.1. The fig. 2.2 and fig 2.3 for shear test specimen, it is cut by wire cut Electrical Discharge Machine. The experiments were conducted to determine the working range of the above factors. Feasible limits of the parameters were chosen in such a way that the diffusion bonds should be free from any visual defects. The important factors lap shear strength and their working range for AZ91 magnesium alloy and SiC are presented in Table 3. Notations for the following parameters T-Bonding Temperature, P-Bonding Pressure, H- Holding Time are described in Table.3.

2.3. RESPONSE SURFACE MODEL FOR BONDING STRENGTH

Shear strength of the diffusion bonded are represented by BS respectively. These responses are function of bonding temperature (T), bonding pressure (P), holding time (H) and they can be expressed as

$$BS = f(T,P,H) \quad (1)$$

The second order polynomial (regression) equation used to represent the response surface Y is given by [12]

$$Y = \beta 0 + \Sigma \beta j\, xi + \Sigma \beta jj\, x\, i\, 2 + \Sigma \beta ij\, xixj + er \quad (2)$$

Where y is response, i.e., bonding strength; xj represents bonding temperature, bonding pressure, holding time, β0, βj, βjj, and βij represent the constant, linear, quadratic, and interaction terms, respectively. The three factors, the selected polynomial could be expressed as

$$BS = b0 + b1(T) + b2(P) + b3(H) + b11(T2) + b22(P2) + b33(H2) + b12(TP) + b13(TH) + b23(PH) \quad (3)$$

The bonding strength obtained from experimental results for different combination of parameters is given as input to the design expert software, and a second order mathematical model for predicting weld strength is developed. The developed mathematical model for diffusion bonding is given below.

Final Equation in Terms of Actual Factors:

Lap Shear Strength =-227.063+0.7 (T) +68.5(P) +2.6(H) - 0.0009(T2)-11.5(P^2)-0.02563(H^2) +0.015(TP) +0.000125(TH)-0.0625(PH) (4)

A total of 17 experiments were conducted at different levels of parameters to obtain diffusion bonding. The values of bonding strength obtained from experiments and those predicted from response surface model along with design matrix and design summary were tabulated in Table 3 and 4 respectively.

Table 3. Design matrix and experimental results

Ex.No	Bonding Temperature ($^{\circ}$C)	Bonding Pressure (MPa)	Holding Time (min)	Lap Shear Strength (MPa)
1	300	9	60	64
2	500	9	60	68
3	300	11	60	64
4	500	11	60	74
5	300	10	40	68
6	500	10	40	73
7	300	10	80	64
8	500	10	80	70
9	400	9	40	64
10	400	11	40	73
11	400	9	80	62
12	400	11	80	66
13	400	10	60	89
14	400	10	60	88
15	400	10	60	86
16	400	10	60	90
17	400	10	60	87

Table 4. Design Summary

Study Type- Response; Surface Runs -17; Initial Design Box-Behnken; Blocks- No Blocks; Design Model- Quadratic

Factor	Name	Units	Type	Low Actual	High Actual
A	Bonding Temperature	^{o}C	Numeric	300	500
B	Bonding Pressure	MPa	Numeric	9	11
C	Holding Time	Min	Numeric	40	80
Factor	**Name**	**Low Coded**	**High Coded**	**Mean**	**Std. Dev.**
A	Bonding Temperature	-1	1	400	68.59
B	Bonding Pressure	-1	1	10	0.68
C	Holding Time	-1	1	60	13.71

Response Name- Lap Shear Strength in MPa; Obs-17; Analysis- Polynomial; Minimum-62; Maximum-90;Mean-73.52941; Std. Dev.- 10.25986; Ratio-1.451613; Model- Quadratic

3. RESULTS AND DISCUSSION

3.1 OPTIMIZING THE DIFFUSION BONDING PARAMETERS

A.1 Analysis of variance

Analysis of variance is the separation of variance ascribable to one group of causes from the variance ascribable to other group. It is nothing but an arithmetical procedure used to express the total variation of data as the sum of its non- negative components. Analysis of variance (ANOVA) is similar to regression in that it is used to investigate and model the relationship between a response variable and one or more independent variables.

The adequacy of the developed relationship is tested using the analysis of variance technique. As per this technique, if the calculated value of the F-ratio of the developed model is less than the standard F-ratio value at a desired level of confidence, then the model is said to be adequate within the confidence limit. ANOVA test results are presented in Table 6.1 for the model. The determination coefficient (R2) indicates the goodness of fit for the model. In this case, the value of the determination coefficient (R2 = 0.9894) indicates that 98.94% of the total variability is explained by the model after considering the significant factors.

Table 5. ANOVA test result for shear strength

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	1666.485	9	185.165	73.02283	< 0.0001
A-Bonding Temperature	78.125	1	78.125	30.80986	0.0009
B-Bonding Pressure	45.125	1	45.125	17.79577	0.0039
C-Holding Time	32	1	32	12.61972	0.0093
AB	9	1	9	3.549296	0.1016
AC	0.25	1	0.25	0.098592	0.7627
BC	6.25	1	6.25	2.464789	0.1604
A^2	341.0526	1	341.0526	134.4996	< 0.0001
B^2	556.8421	1	556.8421	219.5997	< 0.0001
C^2	442.3684	1	442.3684	174.4552	< 0.0001
Residual	17.75	7	2.535714		
Lack of Fit	7.75	3	2.583333	1.033333	0.4677
Pure Error	10	4	2.5		
Cor Total	1684.235	16			

df, degrees of freedom; F, Fisher_s ratio; P, probability
SD = 1.592393, mean = 73.52941, CV% = 2.165654, PRESS = 139.625, R^2 = 0.989461, adj. R^2 = 0.979511, pred. R2 = 0.917099, adeq. precision = 20.36748

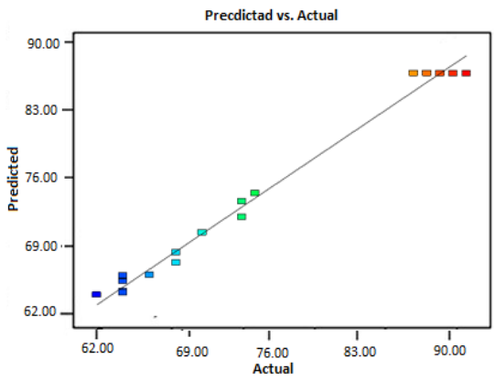

Fig. 3 Normal probability plot of experimental versus predicted shear strength

The models are not over fitted as indicated by the comparison of R2 and R2 – adjusted values. Only less than 1% of the total variations are not explained by the model. The value of adjusted determination coefficient (adjusted R2 = 0.97) is also high, which indicates a high significant of the model. Predicted R2 = 0.9795 is in good agreement with the adjusted R2 and shows that the model would be expected to explain 97.95% of the variability in new data. A 'p' value less than 0.05 indicated the significant model terms. Value of probability greater than F in Table 5 for the model is less than 0.05, which indicates that the model is significant. Lack of fit is insignificant and therefore indicates that the model fits well with the experimental data. The high p value for the lack of fit test also indicates that the model does adequately fit with the response surface for shear strength.

All the above considerations indicate on excellent adequacy of the regression model. Each observed value is compared with the predicted value calculated from the model in Fig.3. This figure shows the normal probability plots of the residual for the composites with respect to the shear strength. The residuals in each plot generate near the straight line, implying that the errors are distributed normally.

3.2 OPTIMIZING THE ACCUMULATED DIFFUSION BONDING PARAMETERS

The response surface methodology (RSM) was used to optimize the diffusion bonding parameters in this study. RSM is a collection of mathematical and statistical techniques that are useful for designing a set of experiments, developing a mathematical model, analyzing for the optimum combination of input parameters, and expressing the values graphically [13].

Response surfaces were developed for the empirical relationship, taking two parameters in the 'X' and 'Y' axis and response in 'Z' axis. The response surfaces clearly indicate the optimal response point. The maximum shear strength is exhibited by the apex of the response surface. The surface plots showing the effect of input parameters taken two at a time on shear strength. To obtain the influencing nature and optimized condition of the process on shear strength, the surface plots and contour plots which are the indications of possible independence of factors have been developed for the proposed empirical relation by considering two parameters in the middle level and two parameters in the X- and Y-axis.

These response contours can help in the prediction of the response for any zone of the experimental domain [14]. The apex of the response plot shows the maximum achievable shear strength. In Fig.6 and Fig.7, the shear strength increases with increasing the bonding temperature, bonding pressure and holding time and then decreases.

A contour plot is produced to display the region of the optimal factor settings visually. For second order responses, such a plot can be more complex compared to the simple series of parallel lines that can occur with first-order models. To classify this, it is most straightforward to examine it through a contour plot. Contour plots play a very important role in the study of a response surface. It is clear from Fig.6 and Fig.7 that the shear strength increase with the increase of bonding temperature, bonding pressure and holding time to a certain value and then decrease.

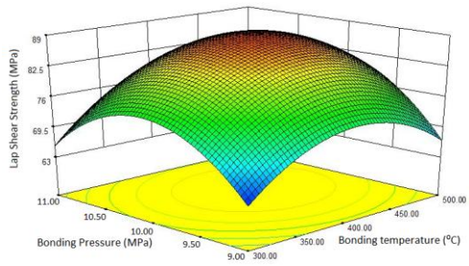

Fig.4(a)

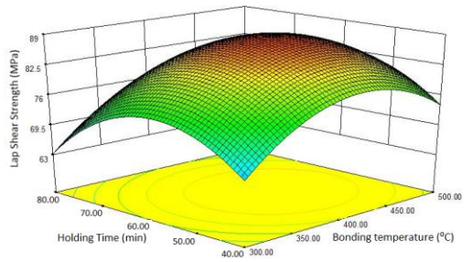

Fig.4(b)

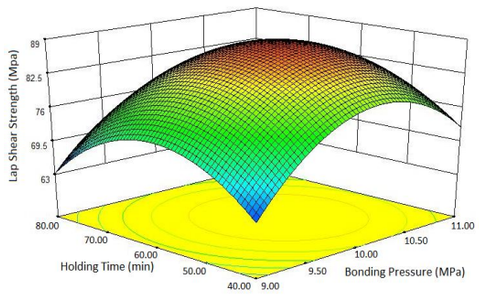

Fig.4(c)

Fig.4 (a), (b) and (c) Response graphs for shear strength

RSM is used to find the optimal set of process parameters that produce a maximum or minimum value of the response [15]. By analyzing the response surfaces and contour plots (Figs. 4 and 5), the maximum achievable shear strength value is found to be 85 MPa. The corresponding process parameters that yielded this maximum value are bonding temperature of 400 ◦C, bonding pressure of 10MPa, holding time of 60 min. Using these optimized diffusion bonding process parameters bonding were fabricated. From these joints, lap shear were fabricated and then tested. The average lap shear strength values are found to be 80 MPa. From these values, it is inferred that the predicted and experimental optimized strength values are in good agreement and the variations is found to be less than ±10%.

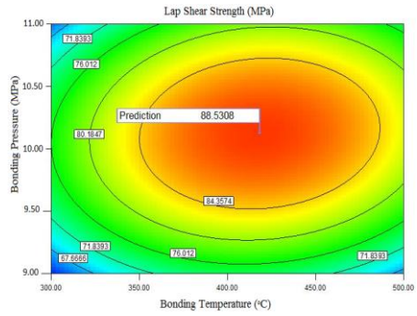

Fig.5(a)

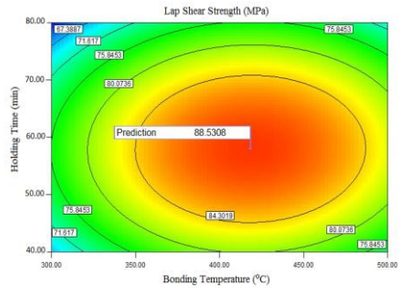

Fig.5(b)

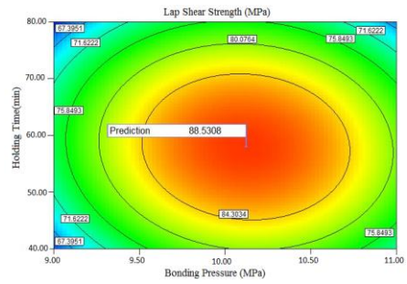

Fig.5(c)

Fig.5 contour graphs for shear strength

Contributions made by the process parameters on strength of the joint can be ranked [16] from their respective ‘F’ ratio value which was presented in Table.6 provided the degrees of freedom are same for all the input parameters. 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 bonding temperature is contributing more on shear strength, and it is

followed by bonding pressure and holding time for the range considered in this investigation.

Table 6. Estimated regression coefficients for shear strength

Factor	Shear strength
Intercept	88
A-Bonding Temperature	3.125
B-Bonding Pressure	2.375
C-Holding Time	-2
AB	1.5
AC	0.25
BC	-1.25
A2	-9
B2	-11.5
C2	-10.25

3.3 Fracture morphology observation

SEM observations of the fractured surfaces of the bonded samples show the presence of plastic deformation as seen in Fig.6. No oxides and or other contaminants are observed on the fracture surfaces. It shows the brittle fracture of the composites with flat fracture surface and reveal that there are some plastic pits originated from the small SiC particles being pulled out from the AZ91 matrix after plastic deformation.

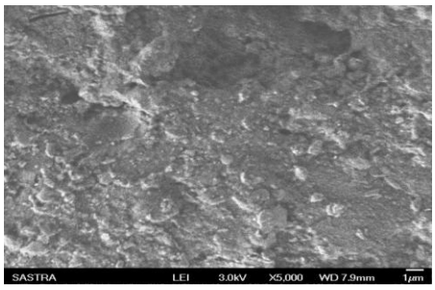

Fig.6 Fracture surface of the SiCp/AZ91 composites

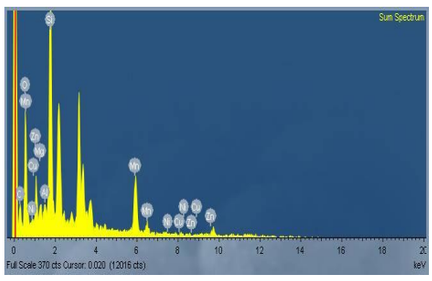

Fig.7 EDX analysis of AZ91/SiC_p composites

However, small dimples are also found in the AZ91 matrix, as shown in Fig.6 shows that the smaller dimples also exist at the SiC_p–AZ91 matrix interface, which reveals the good interface bonding between matrix and particulates [17]. The observations suggest that the process may have beneficial effects on the interfacial bonding. The Fig.7 Shows the major peaks and minor peaks of intermetallic phases such as Mg, Al, Ni ,Cu, Zn, Fe, Mn, Si and C was observed.

4. CONCLUSION

- Accumulative Vacuum diffusion bonding of AZ91/SiC_p composites was carried out successively. Empirical relationships were developed to predict the shear strength of the diffusion bonded AZ91/SiC_p composites and incorporating process parameters. The developed relationship can be effectively used to predict the shear strength of diffusion bonds at 95% confidence level.
- A maximum shear strength of 85 MPa could be attained under the bonding conditions of 400 ◦C of bonding temperature, 10MPa of bonding pressure, 60 min of holding time. The experimentally determined shear strength was 88.53 MPa shows the consistency of the model. Bonding temperature was found to have greater influence on shear strength followed by bonding pressure, holding time.
- The fracture observation revealed brittle fracture of the composites. The size of dimples in the matrix of was smaller, which indicated that the plastic of the SiC_p/AZ91 composite was improved.

ACKNOWLEDGMENTS

The authors are grateful to acknowledge the provision of equipments for the fabrication and testing of composites from Centre for Materials Joining & Research and Department of Manufacturing Engineering, Annamalai University, Chidambaram, Tamil Nadu, India.

REFERENCES

1. *Pai B C Pillai U T S and Srinivasan A (2011), "Creep Resistant Cast Magnesium Alloy Developments for Automotive Applications", Indian Foundry Journal, Vol. 57 (4), 35-43.*

2. *Eigenfeld K Tilch W and Erchow S (2004), "Integrated Magnesium Technology", Advanced Eng. Material, Vol. 6(7), 520–525.*

3. *Kandil A (2012), "Microstructure and mechanical properties of SICp/AZ91 magnesium matrix composites processed by stir casting" , J. Engineering Sciences, Assiut University, Vol. 40(1), 255-270,*

4. *Manoj Kumar B V Basu B Murthy V S R and Manoj Gupta (2005), "The role of tribochemistry on fretting wear of Mg-SiC particulate composites", Part A , Vol.36, 13-23*

5. *Kandil A Amer A Abdul Fattah H and Salem H G (2011), "Effect of TiC particulates reinforcement on mechanical properties and aging behavior of micro and nanostructured matrices of AA2124", Metallurgy 65 Jahrgang 9, 393.*

6. *Joseph Fernandus M Senthilkumar T Balasubramanian V and Rajakumar S (2012), "Optimizing Diffusion Bonding Parameters in AA6061-T6 Aluminum and AZ80 Magnesium Alloy Dissimilar Joints", Journal of Materials Engineering and Performance, Vol. 21(11), 2303-2315.*

7. *Zeer G M Zelenkova E G Koroleva Yu P Mikheev A A and Prokopy'ev S V (2013), "Diffusion bonding through interlayers", Welding International, Vol. 27,(8), 638–643,*

8. *Koichi Kitazono Susumu Komatsu and Yuki Kataoka (2011), "Mechanical Properties of Titanium Particles Dispersed Magnesium Matrix Composite Produced through Accumulative Diffusion Bonding Process", Materials Transactions, Vol. 52 (2), 155-158.*

9. *Zhao L M and Zhang Z D (2008), "Effect of Zn Alloy Interlayer on Interface Microstructure and Strength of Diffusion-Bonded Mg-Al Joints", Scripta Mater., Vol.58, 283–286.*

10. *Arunkumar L and Raghunath B K (2013), "Electro Discharge Machining Characteristics of Mg/SiCP Metal matrix composites by Powder Metallurgy (P/M) Techniques", International Journal of Engineering and Technology, Vol. 5(5), 4332-4338.*

11. *Joseph Fernandus M Senthilkumar T Balasubramanian V and Rajakumar S (2012), "Optimising diffusion bonding parameters to maximize the strength of AA6061 aluminium and AZ31B magnesium alloy joints", Materials and Design Vol.33, 31–41.*

12. *Vigneshwaran P Prabakaran M P Selvaraj T and Kannan G R (2014), "Weld Strength Optimization by using Box-Behnken Design", Int. J. Engineering Research & Technology, Vol. 3 (4), 1879- 1882*

13. *Palani P K and Murugan N (2007), "Optimization of Weld Bead Geometry for Stainless Steel Claddings Deposited by FCAW", J. Mater. Process. Technol., Vol.190, 291–299.*

14. *Zhao L M and Zhang Z D (2008), "Effect of Zn Alloy Interlayer on Interface Microstructure and Strength of Diffusion-Bonded Mg-Al Joints", Scripta Mater., Vol.58, 283–286.*

15. *Tien C L and Lin S W (2006), "Optimization of Process Parameters of Titanium Dioxide Films by Response Surface Methodology", Opt. Commun., Vol.266, 574–581.*

16. *Hou T H Su C H and Liu W L (2007), "Parameters Optimization of a Nano-Particle Wet Milling Process Using the Taguchi Method, Response Surface Method and Genetic Algorithm", Powder Technol., Vol.173, 153–162.*

17. *Cheng N P Zeng S M and Liu Z Y (2008), "Preparation microstructures and deformation behavior of SiCp/6066Al composites produced by PM route", J. Mater Process Technol., Vol.202, 27–40.*

