Analysis of Factors Influencing Hardness of Al-TiB_2 Composites Using Response Surface Methodology

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ABSTRACT

Metal Matrix Composite (MMC) concentrate initially on improved strength, hardness and tensile properties. The AMMCs are generally used in large range of aerospace and automotive application as it has superior properties than other MMC. Material properties based upon its processing parameters, manufacturing techniques, reinforcements and selection of matrix. Aluminium matrix reinforced with titanium diboride (TiB2) yield superior properties than the aluminium alloy reinforced with other particulates such as Al2O3, Sic, and Tic. The main distinct is to yield the composite cost effective way to meet the above essentials. The major motive of this paper is to establish a mathematical model for hardness over Response Surface Methodology (RSM) and analyze the impact of the entire Stir casting parameters (composition of TiB2, stirrer speed, composition of magnesium (Mg) on the responses in stir casting with Aluminium Metal Matrix Composites (AMMC) assisted with titanium diboride. Experimental investigation is carry out by Box Behnken design matrix in RSM with three factors and levels.

KEY WORDS: Aluminium Metal Matrix Composites (AMMC), Stir Casting, Response surface methodology (RSM), Hardness, SEM, XRD.

1. INTRODUCTION

Conventional monolithic materials have some constraint to obtain good combination of hardness. Aluminium and its alloys has a significant role in the production of MMC. AMMC materials has more influence on specific fields owed to their yield strength, stiffness, dimensional stability and high specific strength. There are several ways to fabricate AMMC by considering its application. For metal matrix material Aluminium alloys AA6061 is quite famous. Y because of great formability in nature as well as optimal heat treatment strengthen composite. Al–TiB2P composite difficult in producing so it not available in market in higher order as well as too costly. Few tries were done to produce it by in-situ process.

The microstructure and mechanical properties of aluminium base metal matrix composites materials synthesized using stir casting path were investigated. They had used fly ash reinforced in aluminium matrix and reported the improvements in mechanical properties up to 20% of fly ash A similar issue of titanium carbide particle reinforced with aluminium 6061 matrix composites was observed by Samal (2013), had investigated specific strength and hardness of the material improved appreciably with more addition of Tic. They have reported information regarding Al2O3 particles up to 10% increase in tensile strength and hardness. Sozhamannan (2012), told that AL-TiB2 composite (2.5% TiB2P) shows good improvement in harness as compared to pure Aluminium. It is espied that excess amount to the tune of 120% KBF4, is essiential to get optimum level of TiB2. Any additional increases to 140%KBF4, increases hardness but increases wear rate due to weakening of matrix as a result of segregation of TiB2 particles. TiB2 particle are moderately distributed in Aluminium matrix and are clearly visible in SEM Micrograph. Sozhamannan.G.G suggested that AL-Si alloy/10 wt% Sic composites would be produced using stir-casting technique by varying the stirrer speed and melting temperature. Stir casting technique is the conventional and economical way of producing AMMC. But, conventional stir casting technique has difficulties in production of a particulate reinforced composite. In this present method, apt changes were drift out on conventional stir casting technique to take care of the reaction of molten aluminium with atmosphere, exlution of reinforcing particles and wettability. Restrained base pouring layout tends to regulate the molten metal flow. But, when compared to the current method, difficult in process infiltration. Rengasamy (2016), noticed that stirring speed, position of impeller in melt, holding temperature, size of impeller had important factors during production of cast metal matrix composites as these have an impact on mechanical properties. Shanmughasundaram (2012), investigated mathematical model successfully predicted the delamination during milling of GFRP composites. The advanced second-order response surface model was substantiate using confirmation test and the error was found to be within ±0.3 percent. This procedure is comfort to forecast the main effects and interaction effects of distinct prominent combinations of machining parameters. Pradeep (2013), suggested that the optimum value would be obtained in radial flow model impeller. The verification experiment was conducted for the optimum parameter. The best results have been obtained. The nearby results of prediction situated on calculated S/N ratios and experimental results show that the Taguchi experimental technique used successfully for both optimization and prediction. Present work aimed to predict the hardness of fabricated composites by developed mathematical model using RSM. Regression and graphical analysis of the data collected can be designed by “Expert 8.0” software. Response surface contour graph used to analyze the effect of stir casting parameters on the hardness. Validity of the model as well as finding the significant parameter can be analyzed using ANOVA. Hardness test experiments was conducting to predict the second order quadratic model along with to explore the interdependence of the process parameters using RSM design. Analysis of Variance (ANOVA) was used to test the results gathered using statistical tools similar to RSM. MMC Al 4032 is assisted with reinforcement particles TiB2 and ZrB2in distinct weight. (%) at room temperature over stir casting method was analyzed with the help of MRR and results were showed that the mechanical property of the Al 4032 composite alloy is substantially increased along with increase in weight. % of various reinforcement particles such as Tib2 and Zrb2 when compared with unreinforced Al 4032 alloy. The behavior of an EDM'ED TiB2 ceramic with copper electrode and positive polarity was studied to do so. In this they reported that to achieve a stable process selection of low values of both pulse time and duty cycle was required. In the case of MRR variable, current in density and duty cycle turned out to be the most influential parameters, to such an extent that an increase of both parameters, led to higher MRR values In situ mixed salt reaction method ZA27-TiB2 in situ composites were fabricated and analysis report showed that TiB2, particles much better wear response of the Zinc – based matrix alloy. By increasing the TiB2 content both friction coefficient and wear rate decreases dramatically. TiB2–x vol.% SiC (x = 15, 20, 25 and 30) ceramics at 1850 °C for 120 min was successfully fabricated by hot pressing method and the results reported that the incorporation of SiC to TiB2 ceramic is a promising way to enhance the sinter ability, densification behavior and indentation fracture toughness of this material aware hot pressing of Al – B2O3-TiO2 system and Al-B-TiO2 system derive in the evolution of ceramic Al2O3 and TiB2 particulates (~50nm) and coarse intermetallic Al3Ti content showed considerably higher tensile strength but poorer tensile ductility compared to composite 1.

In the current work, RSM used to predict the Hardness of Al-Tib2 Composites by statistical model. Regression and graphical analysis of the data collected can be designed by "Expert 8.0" software. The research outcome of input parameters on the hardness of Al-Tib2 Composites was done by analysing the response surface contour plots. Analysis of variance (ANOVA) is used to review the legality of the model and for asset the necessary parameters. The hardness of the composite used to predict by X-ray Diffraction (XRD) & Scanning Electron Microscope (SEM).

2. MATERIALS AND METHODS

Material, Process Parameter and Measurement: Metal mart ltd, Coimbatore took Composition test(Table-1) from Omega Inspection and Analytical lab, Guindy, Chennai. TiB2P(powder form) supplied for Alfa Aesar (India) was used as reinforcement.

Table.1. Composition of Aluminium Alloy 6061

Element	Si	Fe	Cu	Mn	Mg	Z	Cr	Ti	Remaining Aluminium
(Wt %)	0.71	0.15	0.27	0.39	0.77	0.010	0.005	0.02	97.55

Stir Cast Process: Stir Cast Process Production in Aluminium-TiB2P composite over stir casting method.1kg of aluminium was melted in a graphite crucible. For the current work melt temperature was raised to 900k. Then the TiB2P weight, varying stirrer speed and composition of Mg (based on Box Behnken design) was combained to the aluminium melt for making of 17 different composites. Moisture were removed by preheated TiB2P particles to 573k. Monetery pure aluminium was melted by elevate the temperature to 950k . After that mild steel stirrer is used to stir it. Fig.1 shows the stir cast machine setup. In Table.2. and 3 Size and material composition of TiB2P, stir casting machine specification is tabulated. TiB2P particle and magnesium 1% (added in all composites because due to wettability) were added to the melt at the time of evolution in the melt due to stirring. Process parameters for stir casting is mentioned in table 4. The melt temperature 950k be maintained during the addition of the particles. After that graphite crucible were used to cast the melt. The particle size analysis for TiB2P and Chemical composition analysis was done for Aluminium 6061. The micro hardness measurement machine with 0.5kg load and diamond ball intender used to take hardness for all composites. Indenter can be maintained 10 seconds on surface of composites to find micro hardness. The SEM & XRD was carried out for all the samples. SEM carries out to find homogeneous distribution and porosity of particle.

Figure.1. Stir casting machine set up

Table.2. Size and material composition of TiB_2P

Material	Oxygen	Carbon	-325 mesh	Particle size, D50
Composition	0.266%	0.476%	99.80%	14.7μm

Table.3. Stir casting machine specification

Sl.No	Machine	SPECIFICATION
1	Minimum & Maximum Stirrer speed(rpm)	100 to 1000
2	Motor power (hp)	0.5
3	Temperature of furnace(°C)	1000
4	Preheating furnace (°C)	1000
5	Crucible	Graphite
6	Stirrer material	Mild steel

Table.4. process parameter for stir casting

Process Parameter	Units	Variable	Level		
			-1	0	1
Composition of TiB_2	%	A	3	7.5	12
Stirrer speed	Rpm	B	200	400	600
Composition of Mg	%	C	1	2	3

Impeller model: Impeller model Three type 4 bladed impeller models as radial flow radial flow were designed and fabricated with 0.7 IOD/CID ratios (Impeller outerdia to Crucible inner dia) . Generally impeller has four blades that can be fixed together at 180° to each other along a vertical axis and blades are fixed to the hub. The impeller models viewed like a birds-eye are shown in Fig.2.Fluid flow pattern (radial flow impeller).The radial flow impeller cage a radial flow pattern transfer aside from the impeller, against the sides of crucible. The flow brunt the side also moves up and down direction to fill the top and base of the impeller.

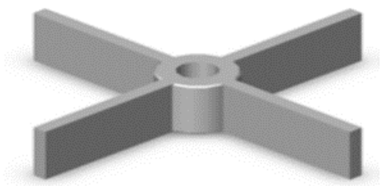

Figure.2. Impeller model for radial flow

3. RESULTS

Microstructures analysis: Microstructures analysis The micro structure of two composites has been taken (400rpm with 3% TiB2P, 600rpm with 12% TiB2P). Specimens were prepared for metallographic testing that can be cut, polished using grinding and emery papers for cast MMCs. After that specimens were etched with the etchant(2.5 ml Nitric acid, 1.5 ml HCL , 1.0 ml HF,95.0 ml Water). After etching microstructures of the samples were examined by Scanning Electron Microscope with secondary electron images. Microstructure of specimen as cast MMCs are shown in Fig.3&4 at different percentageTiB2 particles of the casting. The distributions of TiB2 particles of cast MMC viewed in micrograph image are not uniform all over the casting and segregation of particles are more in the eutectic region. The size of TiB2 particles were uneven in the range of 10-20µm.Porosity commonly established in stir casting of Aluminium composites developed with increase in "TiB2P" particles contents in Aluminium matrix principally containing high percentage of alloy addition. The Titanium diboride is not uniformly distributed. But Rama durai.K discussed SEM images of TiB2. Fig.3. Shows microstructure of this composites (400rpm with 3% TiB2P) Titanium diboride distributed in homogenous manner.

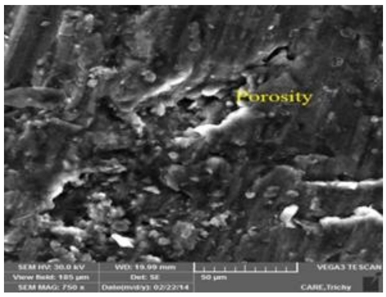

Figure.3&4. SEM image for 3% & 12% of TiB_2 with 3% of Mg and stirred in 400rpm

X-RAY Diffraction pattern analysis: X-RAY Diffraction pattern analysis. X-ray Diffraction (XRD) shows the crystalline size of the elements present in the composite. Fig.5, 6 and 7, shows the XRD results of the prepared composites with their intensity peaks at 3%, 7.5% and 12% wt. % of TiB2. It is noticed the intensity of TiB2 was higher in the plane. And so, the intensity of Al3Ti and aluminium was noticed with different peaks and confirmed over JCPDS software and increased with the weight percentage of TiB2 in the composite. That increase in the intensity of titanium diboride peaks with in increasing of the composites is evident. A continues negligible variation of the Al peaks to greater angles with an increase in the weight % of the titanium diboride content is also clear . The X- Ray diffraction peaks indicates the presence of crystalline TiB2 peaks in all conditions. Minor variation in the peaks were observed by increasing the %TiB2 content and the intensity level increases drastically. It was clearly identified by XRD patterns.

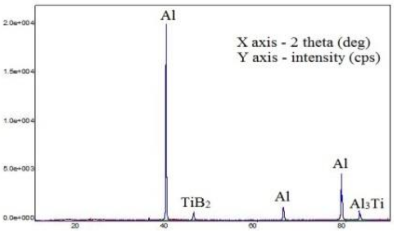

Figure.5. X-Ray diffraction image for condition of 3% of TiB_2 and 3% of Mg are stirred by 400 rpm

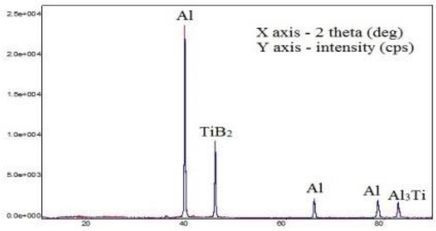

Figure.6. X-Ray diffraction image for condition of 7.5% of TiB_2 and 2% of Mg are stirred by 400 rpm

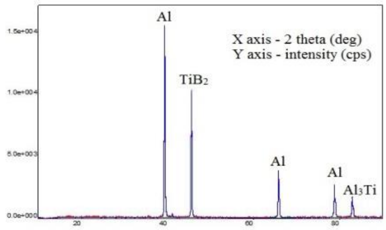

Figure.7. X-Ray diffraction image for condition of 12% of TiB_2 and 3% of Mg are stirred by 400 rpm

Micro Hardness: Hardness is most important factors for the selection in stir casting one among parameters. The study of stir casting characteristics of AMMCs composites depends on many factor and is more influenced by the stir casting parameter like composition stirrer speed, stirrer model, magnesium percentage, pre heating temperature etc… for a given stir casting parameter set up. The prepared cursory favored that the quadratic model is probably important for analysis of hardness. Specifications of Vickers test is mentioned in table 5 The result of the quadratic model for hardness in the mode of ANOVA is plot in table .7,8. The value of R^2 and adjusted R^2 for hardness are 99.86 and 99.68 percentage respectively. This means that regression model provides an excellent explanation of the relationship among the separate factor and the responses. The associated p value for the model is lower than the model is considered to be statically significant. Further factor A and A^2 only have significant parameter for the hardness. Variation of hardness mentioned in Fig.8. The results exposed that the composition of TiB2 is more significant parameter for the micro hardness, when related with the stirrer speed and composition of Mg because of very high F-value. And other model premise are said to be pointless. The shortage of fit was found to less than F0.20 in present research work study and, hence the developed model may be accepted The experimental values are analyzed using response surface analysis and the following relation has been established for hardness of AMMC.

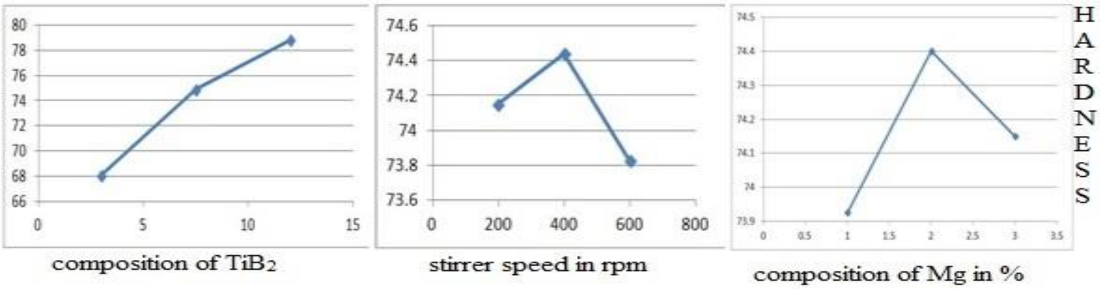

Figure.8. Variation graph for three parameter

The trend exposed that the value of hardness increases by adding the TiB2 percentage. If adding the Mg the trend shows that the higher the composition will decrease the hardness values at the value of 2-2.5% of Mg gives the higher hardness values. Higher TiB2additon and 2-2.5 Mg addition with 400 rpm stirrer speed gives the maximum hardness values.

Table.5. Specification of Vickers micro hardness test machine

Micro hardness	Vickers
Intender	Diamond
Maximum load	2 Kg
Time	10 sec
Load applied	0.5 Kg

Table.7. ANOVA for Hardness

Serial number	**Response 1 Source**	**Hardness Sum of Squares**	**Df**	**Mean Square**	**F Value**	**Prob> F**	
1	Model	241.58	9	26.84	556.72	< 0.0001	Significant
2	*A-Composition of TiB_2*	*231.13*	*1*	*231.13*	*4793.70*	*< 0.0001*	Significant
3	*B-Stirrer speed*	*0.21*	*1*	*0.21*	*4.38*	*0.00746*	Insignificant

4	*C-Composition of Mg*	*0.10*	*1*	*0.10*	*2.10*	*0.1906*	Insignificant
5	*AB*	*0.000*	*1*	*0.000*	*0.000*	*1.0000*	Insignificant
6	*AC*	*1.000E-002*	*1*	*1.000E-002*	*0.21*	*0.6626*	Insignificant
7	*BC*	*0.063*	*1*	*0.063*	*1.30*	*0.2924*	Insignificant
8	*A2*	*8.70*	*1*	*8.70*	*180.46*	*< 0.0001*	Significant
9	*B2*	*0.55*	*1*	*0.55*	*11.48*	*0.0116*	Insignificant
10	*C2*	*0.29*	*1*	*0.29*	6.02	*0.0439*	Insignificant
11	Residual	0.34	7	0.048			
12	*Lack of Fit*	*0.20*	*3*	*0.066*	1.88	0.2738	
13	*Pure Error*	*0.14*	*4*	*0.035*			
14	Cor Total	241.92	16				

Table.8. Rank for three parameters.

Level	Composition of TiB_2	Stirrer speed	Composition of Mg
1	68.075	74.15	73.925
2	74.92	74.44	74.4
3	78.825	73.825	74.15
Delta	10.75	0.615	0.475
Rank	1	2	3

Final Equation in Terms of Coded Factors:

Hardness =+75.20+5.38* A-0.16* B+0.11*C+0.000* A *B+0.050*A*C+0.12* B * C-1.44* A2-0.36*B2-0.26*C2

Figure.9. shows the correlation among the forecast and the experimental value for hardness of AMMCs composites. The influence of different casting process parameter on AMMC composites are studied by using response graph and response table. The influence of casting parameters on hardness is shown in graph.1. And the main effects are exposed in graph.1. it is detected that the hardness value increases with increases composition of TiB2p, compared with 200rpm and 600rpm of stirrer speed in 400rpm gives better hardness and 2grams composition of Mg gives better hardness from the responses table.6, it is asserted that composition of TiB2 is main parameter which affects the hardness followed by stirrer speed and composition of Mg.

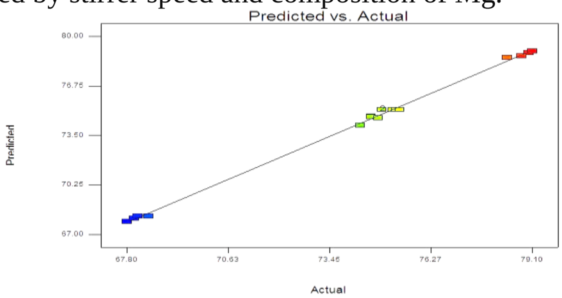

Figure.9. correlation graph for hardness

Table.6. Design of experiment for three factors, three levels each.

		Factor 1	Factor 2	Factor 3	
Std	**Run**	**A:Composition of TiB2**	**B:Stirrer speed**	**C:Composition of Mg**	**Hardness**
		%	rpm	%	HV
5	1	3.00	400.00	1.00	68
14	2	7.50	400.00	2.00	75.2
8	3	12.00	400.00	3.00	79.1
17	4	7.50	400.00	2.00	75.4
10	5	7.50	600.00	1.00	74.3
1	6	3.00	200.00	2.00	68.4
3	7	3.00	600.00	2.00	67.8
6	8	12.00	400.00	1.00	78.8
4	9	12.00	600.00	2.00	78.4
7	10	3.00	400.00	3.00	68.1
15	11	7.50	400.00	2.00	75.2
16	12	7.50	400.00	2.00	75.3
2	13	12.00	200.00	2.00	79
11	14	7.50	200.00	3.00	74.6
13	15	7.50	400.00	2.00	74.9

12	16	7.50	600.00	3.00	74.8
9	17	7.50	200.00	1.00	74.6

Evaluation of Composition TiB2 and Stirrer Speed: Evaluation of Composition TiB2 and Stirrer Speed Figure 10 and 11, shows the effect of composition of TiB2 and stirrer speed on the hardness. According to this figure 10 and 11, the effect of composition of TiB2 is not enhanced as the effect of stirrer speed. This is consistent with the result from the study. Also the optimum hardness appears above 77.2HV while composition of TiB2 is 9.75% and the stirrer speed is in the range 400rpm.

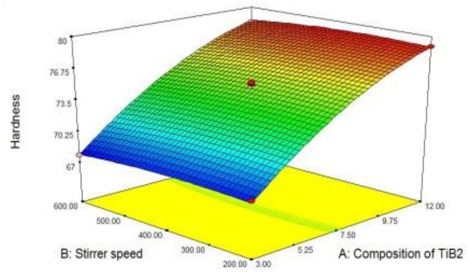

Figure.10. Response surface graph shows the effect the composition of TiB_2 and stirrer speed on hardness

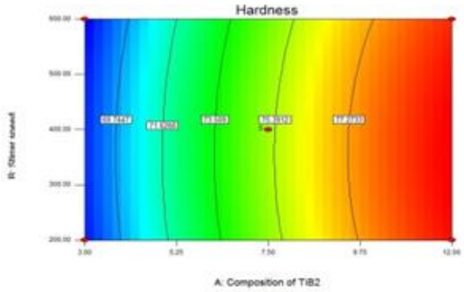

Figure.11. Contour graph shows the effect of composition of TiB_2 and stirrer speed on hardness

Evaluation of Composition TiB2 and Mg: Figure 12 and 13, shows the effects of the composition of Mg and the composition of TiB2 on the hardness of the composites. Withal , the effect of the composition of Mg is not as pronounced as the effect of the

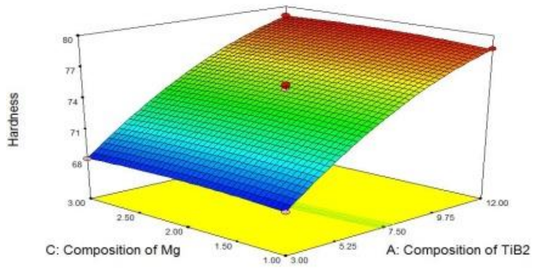

Figure.12. Response surface graph shows the effect of composition of TiB_2 and Mg on hardness.

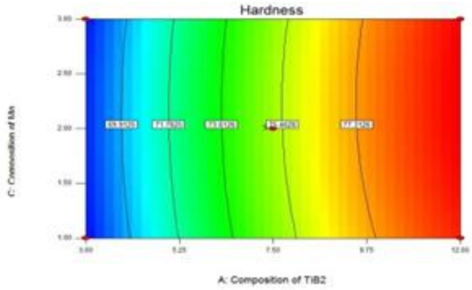

Figure.13. Contour graph shows the effect of composition of TiB_2 and Mg on hardness.

composition of TiB2.According to Figure 12 and 13, as the composition of TiB2 increase with 2% of Mg, resulting in optimum hardness.

Evaluation of composition of Mg and Stirrer speed: Following figure.14 and 15, shows the relationship among the composition of Mg and stirrer speed on hardness. According to this figure.13 and 14, the effect of stirrer speed and composition of Mg is not much significant when related with the effect of composition of TiB2 in the composites. From the response diagram of ANOVA Table 5 and 6, as would be expected, it was found that the 4 bladed radial flow impeller as the excellent model in obtaining the maximum mechanical properties of composite.

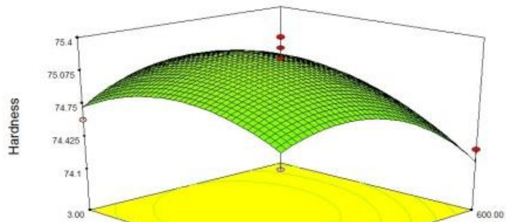

Figure.14. Response graph shows the effect of stirrer speed and composition of Mg on hardness

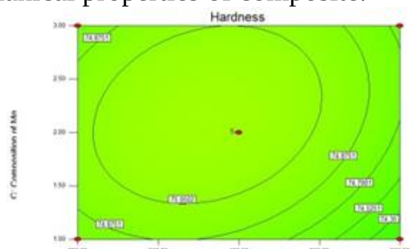

Figure.15. Contour graph shows the effect of stirrer speed and composition of Mg on hardness

Influence of Mg: The fabrication of AL-TiB2 composites by stir casting route is the more difficulty of wettability between the aluminium 6061 and TiB2. The addition of Mg plays the formation of liquids reaction elements and increases dynamic viscosity of composites slurry which reduces the floating of TiB2 particles. When the content of Mg is heat above 700°C TiB2 powder tent to react with aluminium forms intermediate state compound like Al3Ti. Which implies the hardness of composites are increases and also the TiB2 agglomerates in the aluminium composites again.

Confirmation Test: The error values for hardness are calculated and conferred in table 9. The above hardness model was validated using a confirmation test and error was found to be within ±0.8 percent. The model is demonstrated a feasible and effective way evaluation hardness factor for stir casting fabricated composites.

Table.9. Error percentage of experimental value and predicted value

Run	Experimental Value	Predicted Value	Error %
1	65.158	68	-4.17891
2	71.467	75.2	4.964535
3	74.475	79.1	5.847049
4	71.467	75.4	5.216618
5	70.233	74.3	5.473301
6	65.492	68.4	4.251939
7	63.092	67.8	6.944434
8	75.808	78.8	3.796548
9	73.842	78.4	5.814219
10	63.625	68.1	6.571209
11	71.467	75.2	4.964535
12	71.467	75.3	5.090744
13	76.242	79	3.49158
14	71.200	74.6	4.557637
15	71.467	74.9	4.583885
16	69.050	74.8	7.687162
17	72.883	74.6	2.301156

4. CONCLUSION

Based on this study, the following conclusions have been summarized.

- Study on particle reinforcement declared that highest amount of particles are entrapped and distributed uniformly at stirring speed 400rpm and 3% composition of Mg.
- The hardness tends to increase steadily with an increase in the composition TiB2.
- The value of hardness increases much as the composition of TiB2 increases and the optimum hardness is obtain in stirrer speed 400rpm and 2% composition of Mg. whether increase stirrer speed is increases much as the porosity also increases.
- Addition of Mg up to 2% of weight into the composites slurry increases the wettability and thus increases hardness through strong interfacial bonding with TiB2 particles.
- The developed mathematical model successfully predicted the hardness using stir casting parameter.
- The refined second order response surface model was validated using confirmation test and the error was found to be within +-0.8 percent.

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