

Development of Mathematical Model to Predict Micro-Hardness of Al7075/Al₂O₃ Composites Produced by Stir-Casting

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Received 12 July 2011; Revised 12 August 2011; Accepted 15 July 2012

Abstract

The hardness model was developed for aluminium metal matrix composites having Al7075 matrix reinforced with particles of Al₂O₃ and fabricated by stir-casting. Four factors, five levels, central composite, rotatable design matrix was used to optimize the number of experiments. Adequacy of the model was tested by employing analysis of variance (ANOVA). The experimental results showed that size of reinforcement was the major parameter influencing the hardness of the composites among the other control factors, followed by weight fraction of reinforcement. However, the holding temperature and time had lower effects. The model suggests that one must take into account the interaction of parameters for predicting hardness of composites so that the optimal combination of the testing parameters could be determined and predicted.

Keywords: A-Metal Matrix Composites, Hardness, Modeling.

1. Introduction

Metal matrix composites (MMCs) are advanced materials formed by combining a ductile metal/metallic alloy with one or two hard phases, called reinforcements, to exploit the advantages of both [1-5]. Alumina (Al₂O₃), boron (B), carborandum (SiC), zirconium (Zr), etc are the most commonly used non-metallic reinforcements, combined with aluminium alloys to obtain aluminium matrix composites (AMCs); and Al₂O₃/SiC, in the form of short fibers or particulates, are found to possess excellent compatibility [6,7]. Unlike the monolithic materials, the composites provide unique combination of properties such as high strength-to-weight ratio, stiffness, hardness, wear resistance, fatigue resistance, thermal/electrical conductivity, etc. Consequently, they find applications in automobile, defence and aircraft industries [8, 9]. AMCs are generally produced either by (1) solid state processing techniques like powder metallurgy, diffusion bonding, physical vapor deposition or (2) liquid state processing methods such as stir casting, infiltration, spray deposition and reactive processing [1, 2]. Several researchers have attempted to study mechanical and tribological properties of the aluminium matrix composites [10-20]. Among many other parameters, size of abrasives and %weight of Al₂O₃ are reported to be the most effective parameters influencing wear, hardness and other mechanical engineering properties of AMCs [15-17]. Knowledge of hardness is very useful in understanding the ductility behavior of composites and in turn, is paramount in

predicting the resistance to crack initiation/propagation. A number of modeling techniques are available to predict the behavior of AMCs [21]. Huda et al [12] have developed the hardness equation for Al/Al₂O₃ composites, using response surface methodology and indicated that effect of volume fraction of reinforcement is very dominant. However, to the best of our knowledge, there is no systematic procedure developed to evaluate the hardness of these composites. This paper presents an account of modeling the hardness of Al7075/Al₂O₃ composites produced by stir casting, in terms of reinforcement size, % weight of reinforcement, holding temperature and holding time. Rotatable four factors, five levels, factorial design was used [22, 23]. Accordingly, the 'Design Matrix' with 16 factorial points, 8 star points and 7 central points, was employed for producing the composites. Second order equation was fitted by regression to predict the hardness. Adequacy of the model was checked using Analysis of Variance.

2. Experimental procedure

In order to find the range of input variables, a number of trial casts were produced by stir-casting process by changing one-factor at a time keeping the other three at a constant setting [24]. Table 1 shows the coded values of the different parameters and Table 2 shows the design matrix as per which 31 metal casts were produced as per the details explained earlier. Randomization was applied to avoid entry of any systematic error in the model. The samples were fabricated by stir-casting process and specimens extracted from defect-free regions of the castings were subjected to indentation on a Vickers hardness tester.

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Table 1. Coded values of input variables at different levels

Coded values	Input parameters	Notation	Units	Levels						
				Lower level	Middle level	Upper level	-2	-1	0	+1
X1	Size of reinforcement	<i>D</i>	μm	36	45	54	63	72		
X2	% Wt of Al ₂ O ₃	<i>W</i>	Gm	5	6.75	8.5	10.25	12		
X3	Holding temperature	<i>T</i>	°C	150	250	350	450	550		
X4	Holding time	<i>t</i>	Hrs	1	2	3	4	5		

Table 2. Central Composite Design Matrix for Preparation of Samples along with Responses

Trial No	Input Parameters				Output Parameters Vickers hardness VHN
	X1 Size of Al ₂ O ₃ , <i>D</i> (μm)	X2 Wt.% of Al ₂ O ₃ , <i>W</i> (gm)	X3 Sintering Temperature, <i>T</i> (°C)	X4 Holding Time, <i>t</i> (Hrs)	
1	-1	-1	-1	-1	115
2	+1	-1	-1	-1	116
3	-1	+1	-1	-1	132
4	+1	+1	-1	-1	104
5	-1	-1	+1	-1	180
6	+1	-1	+1	-1	122
7	-1	+1	+1	-1	148
8	+1	+1	+1	-1	123
9	-1	-1	-1	+1	149
10	+1	-1	-1	+1	137
11	-1	+1	-1	+1	146
12	+1	+1	-1	+1	152
13	-1	-1	+1	+1	96
14	+1	-1	+1	+1	127
15	-1	+1	+1	+1	131
16	+1	+1	+1	+1	130
17	-2	0	0	0	131
18	+2	0	0	0	126
19	0	-2	0	0	124
20	0	+2	0	0	145
21	0	0	-2	0	133
22	0	0	+2	0	158
23	0	0	0	-2	97
24	0	0	0	+2	137
25	0	0	0	0	128
26	0	0	0	0	132
27	0	0	0	0	132
28	0	0	0	0	106
29	0	0	0	0	128
30	0	0	0	0	128
31	0	0	0	0	128

2.1 The stir-casting process

The stir-casting process comprised melting Al-7075 in an electric furnace while it was being continuously stirred by a ceramic-coated stainless steel motorized impeller. Particulates of Al₂O₃, cleaned through fluxing and degassing for 3-4 minutes and preheated to 600°C were added to the melt at 730°C. Castings were prepared in the form of rods measuring 2.5mm diameter × 370 mm long. Figure 1 shows the schematic diagram of stir-casting process with various components and Figure 2 shows the close-up view of the actual set-up [1, 2].

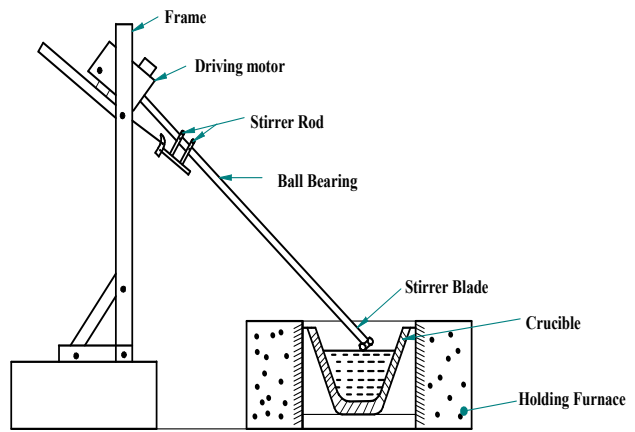


Fig. 1. Schematic diagram showing various components of stir-casting process. The stirrer is made of a ceramic coated stainless steel rod and is operated by a servomotor.



Fig. 2. Close-up view of the actual set-up. Al₂O₃ particulates are added to the melt at 730°C.

3. Planning of the experiment

AMCs comprising Al-7075 matrix and particulates of Al₂O₃ were fabricated by stir-casting. Table 3 & 4 present the details of the chemical composition and other important properties of the matrix material used. Factorial design of experiments was used for mathematical modeling of micro-hardness (VHN), in terms of reinforcement size (*D*), varying weight percent (*W*), holding temperature (*T*) and holding time (*t*). Al₂O₃ particulates were in the range of 36 to 72 μm, weight per cent of Al₂O₃ selected was from 5 to 15%, holding temperature was in the range of 150-550°C and holding time was varied from 1 to 5 Hrs. Analysis of Variance (ANOVA) was performed to determine the influence of the 4 input parameters and their interactions. Adequacy of the model was checked by Fisher's F-test.

Table 3. Chemical Composition of matrix material (Al-7075)

Cr	Cu	Mg	Zn	Al	Density g/cc at 20°C
0.22	1.60	2.80	5.50	Balance	2.89

Table 4. A few important properties of matrix material (Al-7075)

Tensile Strength MPa	Yield Strength MPa	%Elongation	Hardness VHN	Thermal Conductivity Cal/Cm ² /Cm/°C at 25°C	Elect. Resistivity μΩ-Cm at 20°C
227.53	103.42	17	78.50	0.29	5.74

4. Mathematical modeling

Equation 1 gives the general form of the response as per factorial design of experiments [22].

$$Y_u = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 \quad (1)$$

where, Y_u is the response, X_1, X_2, X_3 and X_4 are the coded values of the variables and b_0, b_1, \dots are the regression coefficients.

4.1 Evaluation of the coefficients of models

The values of the regression coefficients were evaluated with the help of the following equations (2) to (5).

$$b_0 = 0.142857(0y) - 0.035714\sum(iiy) \quad (2)$$

$$b_i = 0.014667(iy) \quad (3)$$

$$b_{ii} = 0.031250(iiy) + 0.003720\sum(iiy) - 0.035714(0y) \quad (4)$$

$$b_{ij} = 0.0625(ijy) \quad (5)$$

Substituting the values of the coefficients the hardness model is written as,

$$VHN = 126 - 2.303D - 3.056W + 2.028T + 4.806t - 0.611D*D - 2.111W*W - 4.861T*T - 2.264t*t - 3.170D*W - 1.295D*T - 5.830D*t - 0.167W*T - 4.292W*t - 0.417T*t \quad (6)$$

Using equation (6), ANOVA was performed and Table 5 presents the results of the same. It is noticed that $F_{Model} > F_{Table}$. Hence the model is adequate.

Table 5. Analysis of variance (ANOVA)

Source	DF	SS	MS	F	Remarks
Regression	14	6576.74	469.76	5.78	$F_{Model} > F_{Table}$
Lack of Fit	10	2094.36	209.44		Hence, the Model is Adequate
Residual Error	6	488.00	81.33		
Total	30	9159.10	---		

Value of F-ratio as per Table (14, 6, 0.05) = 3.96; R-Sq = 0.947; R-Sq (adj) = 0.939

4.2 Confirmation of the experiments [27]

Experiments were conducted to confirm the correctness of the hardness model developed in this work. For this purpose, five experimental runs were used with different values of reinforcement size (D), %weight of reinforcement (W), sintering temperature (T) and sintering time (t). The coded

values of the parameters were determined using equation (7). The difference in the experimental values of hardness corresponding to a set of input parameters and the predicted values were taken as error of prediction and are calculated as per equation (8) reported as % error in Table 6 along with other results. It is observed from this table that the results are within the acceptable range and the maximum error is 6.59%.

$$X_i = 2[2X - (X_{max} + X_{min})] / (X_{max} - X_{min}) \quad (7)$$

where, X_i is the code to be used in the model for the input variable, X, X_{max} and X_{min} are the upper and lower levels of that parameter, respectively.

$$\%Error \text{ of prediction} = 100 * (\text{Experimental Value} - \text{Predicted Value}) / \text{Predicted Value} \quad (8)$$

Table 6. Results of Confirmation Experiments

D	Input variables			Experimental Values of Hardness	Predicted Values of Hardness	% Error
	%W	T	t			
37	10.00	400	3.00	134	130	3.07
44	8.00	315	2.50	121	119	1.68
53	9.60	420	3.75	116	123	-5.69
54	8.50	350	3.00	124	126	-1.59
63	8.50	400	4.00	110	106	3.77

5. Results and Discussions

Table 5 presents the results of ANOVA. Figures 3 to 6 present the details of the indentation study performed on the composites. A minimum of 10-15 indentations were taken for each specimen and the average value of the hardness for a few typical cases are indicated, along with the corresponding levels of the factors. It is observed that the hardness of the composites is affected by the 4 main factors and their interactions. Graphical relations (Figures 7-16), depicting the variation of hardness are presented along with the discussions.

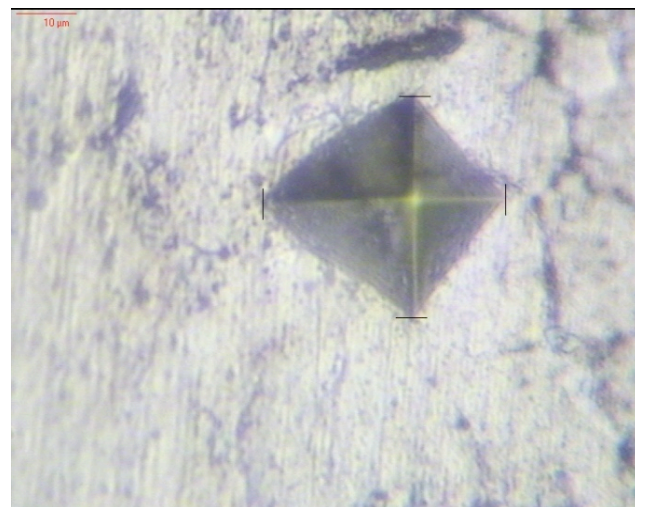


Fig. 3. Indentation details for the combination D = 72μm, W = 8.50% Al₂O₃, T = 350°C & t = 3hrs. Average Hardness = VHN126.

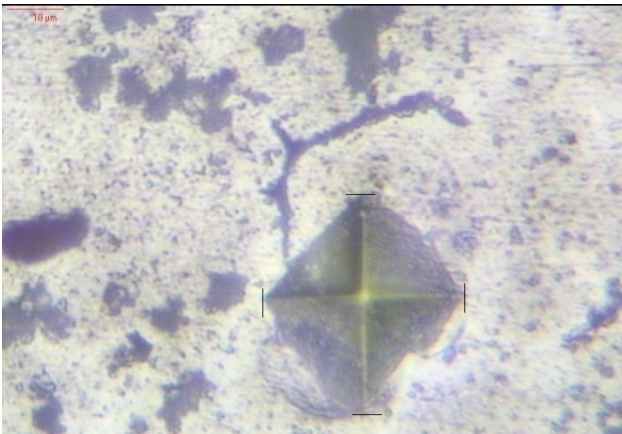


Fig. 4. Indentation details for the combination D = 54 μm, W = 8.50% Al₂O₃, T = 150°C & t = 3 hrs. Average Hardness = VHN133.

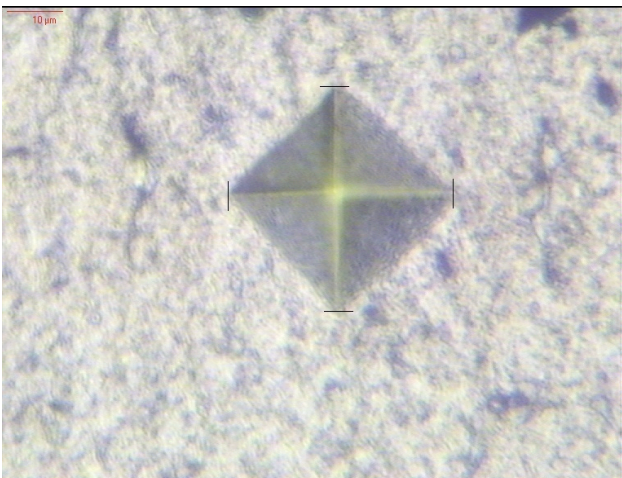


Fig. 5. Indentation details for the combination D = 36 μm, W = 8.50% Al₂O₃, T = 350°C & t = 3 hrs. Average Hardness = VHN131.

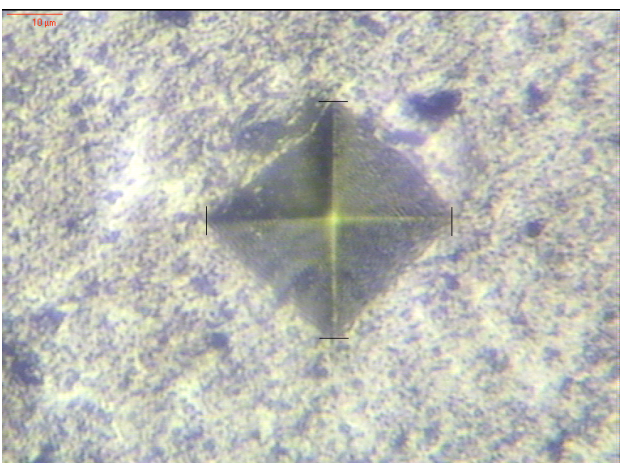


Fig. 6. Indentation details for the combination D = 54 μm, W = 8.50% Al₂O₃, T = 550°C & t = 3 hrs. Average Hardness = VHN158.

5.1 Effect of Main Factors

Figures 7 to 10 show the effect of 4 main factors, namely, the reinforcement size (D), % weight of reinforcement (%W), the sintering temperature (T) and the sintering time (t).

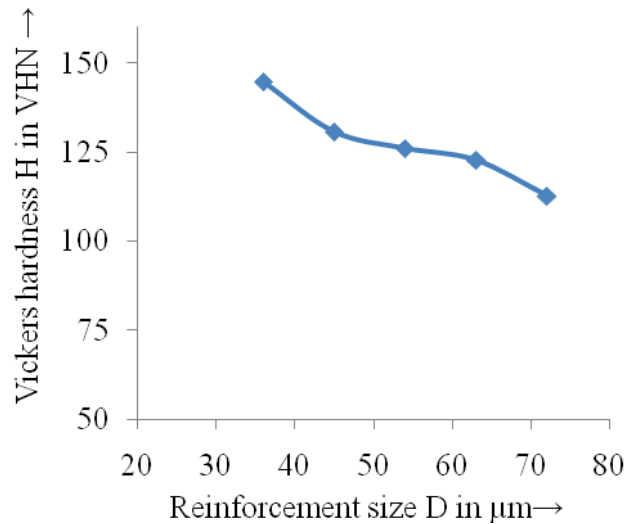


Fig. 7. Plot showing the Vickers hardness H in VHN v/s Reinforcement size D in μm

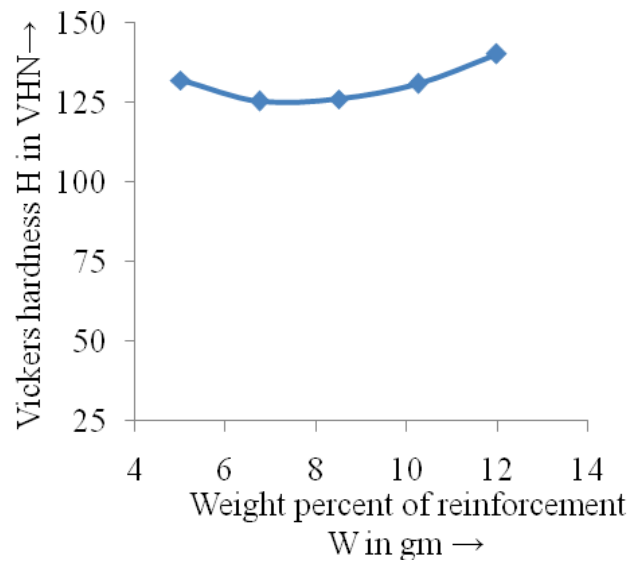


Fig. 8. Plot showing the Vickers hardness H in VHN v/s Weight percent W in gm

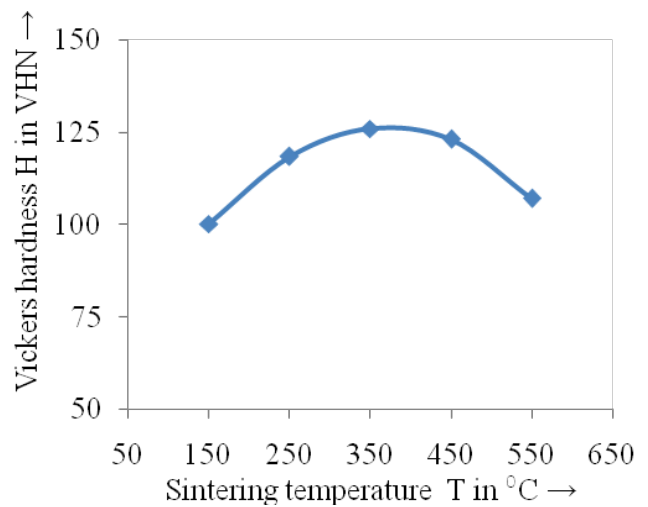


Fig. 9. Plot showing the Vickers hardness Vickers hardness H in VHN v/s Sintering temperature T in °C

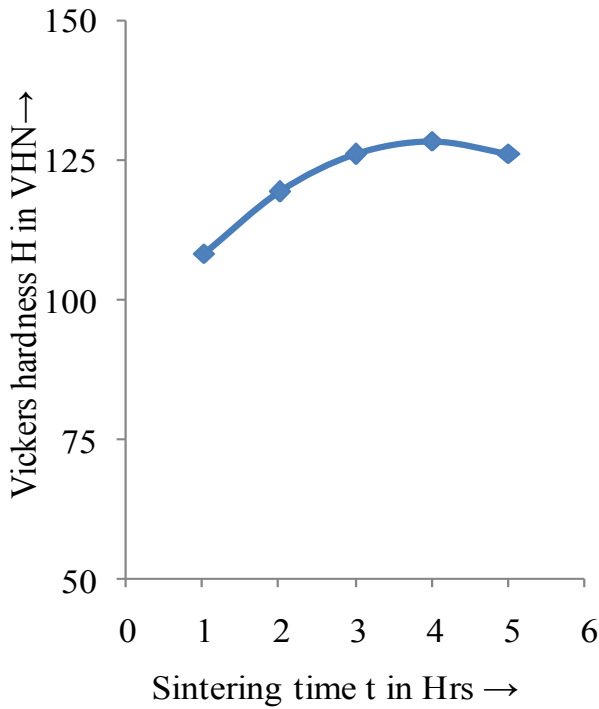


Fig. 10. Plot showing the Vickers hardness Vickers hardness H in VHN v/s Sintering time t in Hrs.

From Figure 7 it is observed that hardness of the composites consistently decreases from 150 VHN-110 VHN, as the size of reinforcement increases. This is due to the fact that higher grain size results in less dense distribution of Al_2O_3 particulates in the aluminium matrix and as such the hardness obtained is the average of the measured hardness values over the entire area of the specimens. However, an average hardness of about 130 VHN is obtained for $D = 63 \mu m$.

Figure 8 shows the variation of hardness with % weight of Al_2O_3 particulates. It is noticed that the hardness is minimum (128 VHN) for 7.6% W. It is also observed that beyond this point the hardness increases consistently to a value of 140 VHN at 15%. This can be explained by the fact that as the % of reinforcement (higher hardness component) increases, the ratio of reinforcement-to-matrix becomes richer in Al_2O_3 content which imparts increased hardness to the composite.

Figure 9 gives the effect of sintering temperature (T) on the hardness. It is observed that there is an optimum temperature ($\sim 370^\circ C$) at which we can produce the composites with a hardness of around 135 VHN. The effect of sintering will be to remove moisture and harden the matrix. However, at temperatures other than this value hardness has a tendency to decrease. This may be due to the fact that, at lower temperatures, the sintering is still not effective and at higher ones the particulates of Al_2O_3 seem to soften out.

Figure 10 shows the effect of sintering time on the hardness of the composite. Again, it is observed that there is an optimum sintering time (~ 4 Hrs) which will result in a hardness of around 135 VHN.

5.2 Effect of Interaction of Factors

Figures 11 to 16 present the effects of parameter-interactions on the micro-hardness of the composite under study.

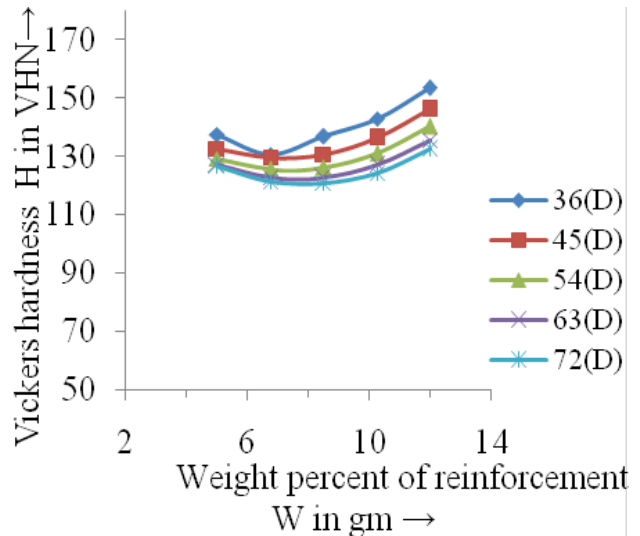


Fig. 11. Plot showing the interaction effect of % weight, W in gm and size of reinforcement, D in μm on Vickers hardness, H in VHN. Note: $T = 350^\circ C$ & $t = 3$ Hrs

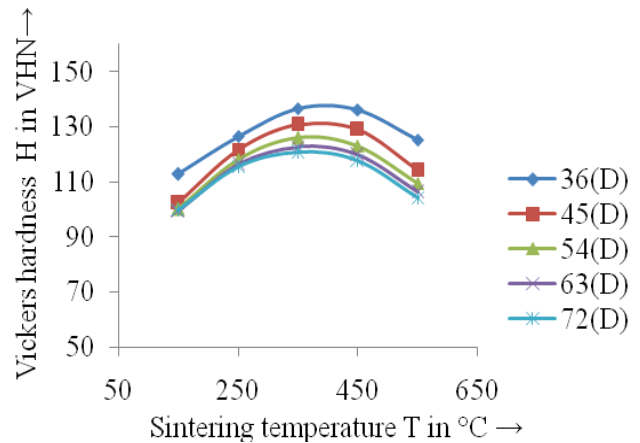


Fig. 12. Plot showing the interaction effect of sintering temp, T in $^\circ C$ and size of reinforcement, D in μm on Vickers hardness, H in VHN. Note: $W = 8.50 gm$ & $t = 3$ Hrs.

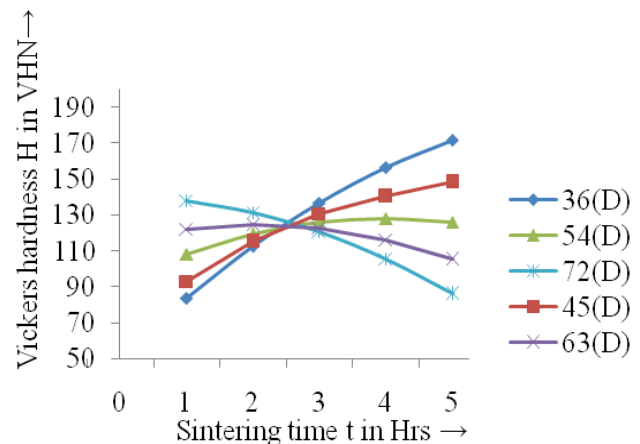


Fig. 13. Plot showing the interaction effect of sintering time, t in Hrs. and size of reinforcement, D in μm on Vickers hardness, H in VHN. Note: $W = 8.50 gm$ & $T = 350^\circ C$

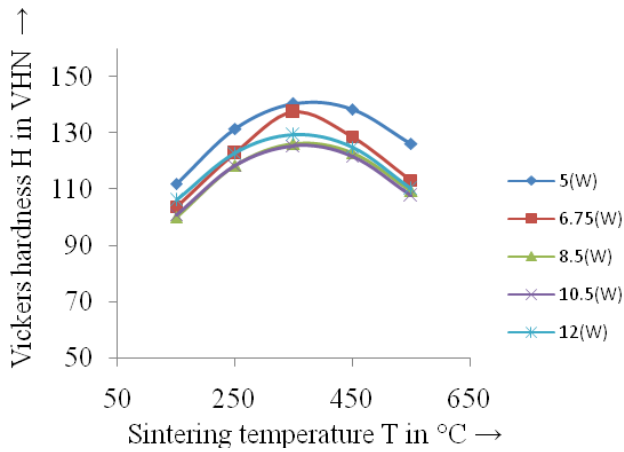


Fig. 14. Plot showing the interaction effect of sintering temp, T in °C and % weight of reinforcement, W in gm on Vickers hardness H in VHN. Note: D = 54 μm & t = 3 Hrs.

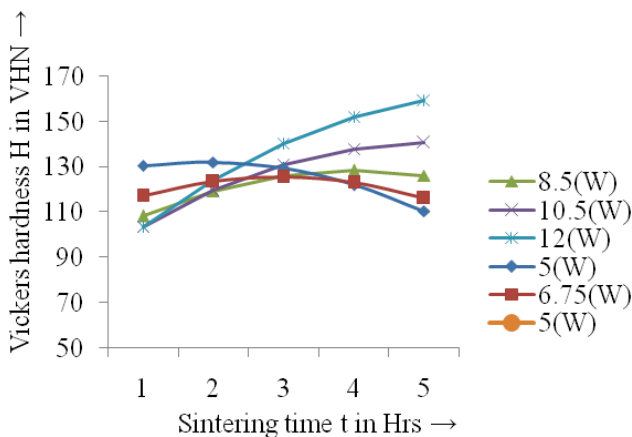


Figure 15 Plot showing the interaction effect of sintering time, t in Hrs. and % weight of reinforcement, W in gm on Vickers hardness, H in VHN. Note: D = 54 μm & T = 350°C

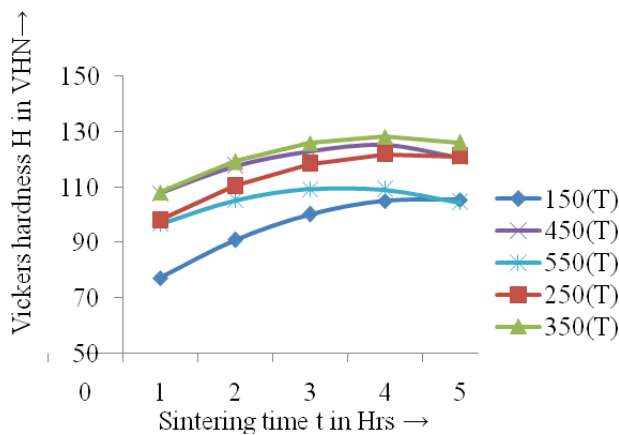


Fig. 16. Plot showing the interaction effect of sintering time, t in Hrs. and sintering temp, T in °C on Vickers hardness, H in VHN. Note: D = 54 gm & %W = 8.50 gm

Figure 11 shows that maximum hardness (~140 VHN) is obtainable with minimum size (D=36μm) and maximum %weight of Al₂O₃ (W=15%). This is due to the fact that higher proportion of the reinforcement will naturally result in increased harder phase and enhances the hardness of an, otherwise softer matrix of aluminium, by almost two times.

Figure 12 shows that at a sintering temperature (T) of around 350°C, particulates of lowest size (D=36μm) will result in a composite of hardness value 140 VHN. This can be explained by the fact that evenly distributed particulates of Al₂O₃ will set at that temperature and possess higher hardness. Thus sintering appears to be an important stage in the manufacture of these composites.

Figure 13 indicates the effect of interaction between particulate size (D) and the sintering time (t). It is observed that smallest size particulates held at around 380°C need about 4 Hrs for complete setting and attaining a hardness of about 140 VHN. However, there appears to be a point of inflexion at around 3Hrs and below this time, there is a drastic change in the dependence of hardness on the interaction effect of D and t.

Figure 14 shows the interaction effect of %weight (W) and sintering temperature (T). The influence of higher percent of Al₂O₃ is quite pronounced on hardness at around 400°C. However, the combination of lowest %W and lowest sintering temperature results in lower order of hardness (97 VHN). Therefore, it can be inferred that there is an optimum temperature at which the sintering has to be carried out to obtain composites of the desired hardness.

Figure 15 presents the interaction effect of %weight (W) of reinforcement and sintering time (t). Combination of higher t and higher W appears to give the highest hardness (~160 VHN), and the trend is reversed for lower %W.

Figure 16 shows the interaction effect of sintering temperature (T) and sintering time (t). It is observed that the increase in time beyond an optimum value has almost a negative effect on the hardness. This is, probably, due to softening of the set particulates of Al₂O₃ at higher temperatures and time. Consequently, the maximum hardness of around 130 VHN is obtainable at 380°C and a sintering time of 4Hrs.

Overall, it is evident from the foregoing discussion that an optimum combination of the process parameters is to be employed to obtain the composite of desired hardness. It is further observed that a composite of 135VHN can be produced by this method by mixing Al₂O₃ particulates of 36μm size, in 15% Al-7075 matrix at a sintering temperature of 380°C and sintering time of 4Hrs.

6. Conclusions

The stir-casting process can be used successfully to produce Al-7075/Al₂O₃ composites of desired hardness within the frame work of experimentation. The large volume fraction of alumina was observed to profoundly affect the behavior of the metal matrix composites of aluminium during fabrication as well as sintering. Within the range of input parameters studied, it was observed that the Vickers micro-hardness decreased consistently with the increase in particle size and %weight of Al₂O₃ [25-27]. However, the effects of sintering temperature and time were not quite consistent.

1. The analysis showed that central composite rotatable design can be used to systematically and thoroughly predict the micro-hardness of aluminium metal matrix composites (AMC) containing Al-7075 matrix and Al₂O₃ reinforcement.
2. The size and % weight of reinforcement were the two most significant parameters affecting hardness of the composites produced by stir-casting process. Thus Al-7075 matrix containing 15% of 36 μ Al₂O₃ particulates exhibited the highest micro-hardness.

3. The average micro-hardness of 135 VHN, as against the base metal hardness of 78.50 VHN (~72% increase), was obtainable for the combination of, $D = 36\mu\text{m}$, $W = 6.75\%$, $T = 360^\circ\text{C}$ and $t = 4\text{Hrs}$. Sintering at 360°C for 4 Hrs. was essential in imparting the desired hardness to the composite.
4. From the confirmation experiments it is observed that the maximum error of prediction is 5.69%.

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