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Optimization of preheating temperature for TiB_2 reinforcement on the preparation of stir cast LM4 + TiB_2 composites and effect of artificial aging on hardness improvement using ANOVA

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Abstract. This work emphasizes the optimization of preheating temperature of TiB_2 reinforcement powder with LM4 composites, and statistical analysis for predicting hardness improvement during aging treatment using ANOVA, are illustrated in this article. A two-stage stir casting procedure was used to fabricate LM4 + TiB_2 (1, 2 and 3 wt.%) composites. The impact of preheating TiB_2 reinforcement powder at various temperatures such as 600, 500, 450, 350 and 250 °C, to attain uniform distribution of reinforcements in the matrix was studied. Optical microstructure analysis clearly shows that the optimum preheating temperature of TiB_2 powder for effective preparation of composites is 350 °C for 30 min without agglomeration of reinforcement particles. After successful preparation of composites, the as-cast samples were subjected to single-stage and multistage solutionizing treatments and then artificially aged at 100 and 200 °C to obtain peak hardness. Micro Vickers Hardness test was done to calculate the hardness of both age hardened LM4 alloy and its composites and results were analyzed. An increase in wt.% of TiB_2 (1–3%), the hardness of composites increased, and multistage solutionizing treatment followed by artificial aging at 100 °C was proven to achieve the highest peak hardness value for LM4 + 3 wt.% TiB_2 composites. Compared to as-cast LM4 alloy, 80–150% increase in hardness was observed when aged at 100 °C and 65–120% increase in hardness was observed at 200 °C during SSHT and MSHT, respectively. ANOVA was performed with wt.%, solutionizing type, aging temperatures as factors, and peak hardness as the outcome. From the results, it can confirm that all three factors contributed effectively for achieving the peak hardness. R^2 value validates that the factors account for 100% of the variance in the hardness results.

Keywords: Preheating / multistage solutionizing heat treatment (MSHT) / stir casting / analysis of variance (ANOVA) / single-stage solutionizing heat treatment (SSHT)

1 Introduction

Researchers have lately been inspired to study novel alternative materials as a result of worldwide environmental initiatives to recycle. Given the automotive engineering industry's desire to increase engine efficiency and fuel economy, novel components are being used to reduce engine size while improving the mechanical and physical properties [1]. Recent advancements in the automotive and aerospace sectors have resulted in the creation of new lightweight alloys and composites utilized in the manufacture of various components [2]. Researchers favor Al alloys as base matrix due to their widespread usage in energy-efficient transportation since they are easy to produce and beneficial in reducing energy due to their low weight and substantial strength [3]. Various publications also indicate that for the same reinforced ceramic particle, aluminium alloy exhibits superior improved characteristics as a base alloy than other matrices such as Mg, Cu, and so on. Titanium diboride (TiB_2) has emerged as an excellent reinforcement for soft metals such as aluminium and its alloys due to its remarkable properties like high melting point (2790 °C), strong thermal stability, and high hardness (960 HV). TiB_2 also has the benefit of being resistant to pull-out damage when compared to other ceramic materials [4]. Stir casting process is the best method to fabricate composites as it is cost-effective, provides high metal yield, and causes less damage to reinforcement particulates [5].

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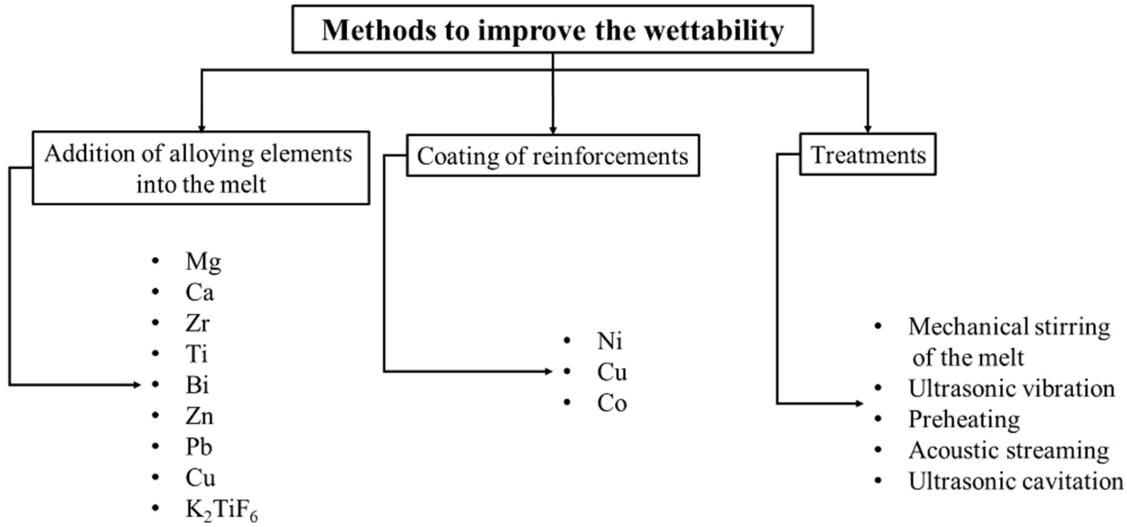

Fig. 1. Different methods to improve wettability.

1.1 Wettability and preheating

Researchers observed that the inhomogeneous reinforcement distribution over the molten matrix, excessive interfacial energy, surface tension, and poor wettability had degraded mechanical properties. Composite properties can be improved by preheating the reinforcement to remove moisture and absorbed gases, using surface coatings and adding alloying elements in an inert gas atmosphere, injecting particles to reduce particle agglomeration, and improving wettability and homogeneous distribution [5].

Dipankar et al. [6] prepared Al2024 + TiB_2 (0, 3, 6 and 9 wt.%) composites using stir casting method where TiB_2 particles are preheated at 450 °C for 30 min. Form results it was concluded that dislocation density improved between matrix and reinforcement as TiB_2 addition refined aluminium alloy grains and improved the microhardness of Al2024-TiB_2 composites. Ramesh et al. [5] investigated the effect of preheated TiB_2 reinforcement powder on mechanical properties of AA7075 + TiB_2 (4, 8, 12 wt.%) composites. From results, they concluded that preheating of reinforcement powder at 500 °C caused better bonding with matrix and reduced the porosity level. Composite with 12 wt.% of TiB_2 exhibited a 42.4% increment in hardness when compared to AA7075 alloy. Also, the tensile and wear properties of composites increased with an increase in wt.% of TiB_2. Pazhouhanfar et al. [7] prepared Al6061 composites with TiB_2 (3, 6 and 9 wt.%) as reinforcements. K_2TiF_6 (potassium titanium fluoride) was added to the melt to improve the wettability of TiB_2 in the matrix, also TiB_2 particles were preheated at 250 °C for 2 h. They concluded from the results that the addition of K_2TiF_6 and preheating of TiB_2 resulted in uniform reinforcement distribution of TiB_2 and enhancement of mechanical properties.

The following three methods can be used to improve the wettability: (i) adding alloying elements, (ii) coating of reinforcements and (iii) treatments of reinforcements [8] as shown in Figure 1.

When using Mg as a wettability agent, use caution, as too much of it might affect the composition of the composite [9]. Abhijit et al. [10] fabricated Al7075 + TiB_2 (0, 3, 6 and 9 wt.%) composites using the stir casting method, in which reinforcement was preheated at 450 °C before adding to molten metal. To improve the wettability of molten metal 2% Mg was added before reinforcement addition. From wear studies, they concluded that Al7075 + 9 wt.% TiB_2 composite can be used in mating part applications as it exhibited less wear when compared to other samples. As per Hanizam et al. [9] addition of Mg (0.5 wt.%) during the stir casting process helped to improve the wettability and reduced the surface tension between matrix and reinforcement, however, the impact of Mg addition on increasing mechanical characteristics was found to be very low. Afkham et al. [11] coated SiC and Al_2O_3 separately with Ni and Cu to improve the wettability when fabricating composites using A356 as the base alloy. Ni coated to fine SiC showed non-uniform distribution in the matrix, however other combinations showed better reinforcement distribution. Pourhosseini et al. [12] coated Al_2O_3 with Cu, Ni, and Co separately and prepared A356 composites. From results, they concluded that coating improved the wettability of Al_2O_3 in the matrix and among them, Ni coating was more successful than other coatings which improved the mechanical properties of composites. According to Wu et al. [13] when compared to typical mechanical stirring alone, high-intensity ultrasonic vibration combined with stirring ensures a more uniform reinforcement distribution, especially when the reinforcement powders are of nano size. As the nano and micron particles have a large surface to volume ratio they tend to form clusters and agglomerate which are the major hurdles to attaining uniform distribution [14]. Long stirring time will provide uniform distribution of reinforcements at the cost of adding more gas and oxidation to the molten melt [15]. Among all the methods discussed above, preheating of reinforcements is the affordable and efficient

Table 1. Different reinforcement preheating temperatures used by various authors in the preparation of composites.

S. No	Matrix	Reinforcement	Reinforcement preheating temperature in °C	Reinforcement preheating time in min	Highest hardness value of composite	References
1	AA7075	TiB_2- (0, 4, 8, 12 wt.%)	500	–	135 VHN @ 12 wt.% addition	[5]
2	Al2024	TiB_2- (0, 3, 6, 9 wt.%)	450	30	129 VHN @ 9 wt.% addition	[6]
3	Al7075	TiB_2 (0, 3, 6, 9 wt.%)	450	–	–	[10]
4	A356	Nano SiC_p	850 (calcination), 250 (preheating)	120, NA	–	[13]
5	A356	SiC and Al_2O_3 coated with Ni and Cu	350	120	118 HV @ Ni coated Al_2O_3 + Cu coated SiC	[11]
6	A356	Al_2O_3 and SiC coated with Cu	350	120	190 HV @ 1.5 wt.% Al_2O_3 and 1.5 wt.% SiC addition	[16]
7	Al6061	TiB_2 (3, 6, 9 wt.%)	250	120	150 HV @ 9 wt.% addition	[7]
8	A356	WC (0–4 wt.%)	595	–	120 HV @ 4 wt.% addition	[17]
9	Al7075	SiC + Al_2O_3	800	120	–	[18]
10	Al6061	TiB_2 (0, 2, 4, 6, 8 wt.%)	670	–	73.93 HV @ 10 wt.% TiB_2 addition	[19]
11	LM25	TiC (10 wt.%)	350	25	129 HV @10 wt.% of TiC addition	[20]

method to improve wettability and to attain uniform reinforcement distribution in the matrix material. Different preheating temperatures used by various researchers while fabricating composites are shown in Table 1.

1.2 ANOVA (analysis of variance)

Design of experiment (DOE) is a systematic and organized strategy for determining the relationship between many factors affecting a process and its output [21]. The primary goal of DOE is to evaluate the influence of different factors and their interactions on the response by conducting a small number of experiments and determining the best combination of parameters that offers the best response [22].

Radhika et al. [22] conducted DOE on abrasive wear behaviour of Al-Si + B_4C composites with three levels of speed, sliding distance, and load. Results revealed that sliding distance (42.2%) has a major effect on mass loss and that ANOVA aids in the construction of the regression equation based on the influence of factors. Sachit et al. [23] investigated wear behaviour of LM4 + WC (0.5, 1, 1.5 and 2 wt.%) composites. ANOVA was used to find major impacting factor among load, speed, and wt.% of reinforcements on wear behaviour of composites. Results revealed that load is having a major impact on wear rate with 72.56% followed by sliding speed and wt.% of reinforcements. ANOVA used by some of the researchers to check the contribution of various factors in improving certain mechanical properties are listed in Table 2.

Based on the thorough literature review, it was observed that limited research work was carried out on MSHT to improve the mechanical properties of TiB_2 reinforced composites. So, in this article, the effect of preheating temperature of TiB_2 reinforcement powder on the composite preparation was studied and optimum preheating temperature to attain uniform distribution of reinforcements in the matrix was accomplished. LM4 + TiB_2 composites were fabricated using two-stage stir casting method and subjected to both single-stage solution heat treatment (SSHT) and multistage solution heat treatment (MSHT) followed by artificial aging treatment. The effects of SSHT and MSHT on composite hardness were investigated, as well as their comparison. The prospect of improving composite hardness by altering different aging factors at different time intervals to identify the peak aging condition was examined. Statistical analysis was performed using Analysis of Variance in MINITAB 15 with wt.%, solutionizing type and aging temperature as factors and their contribution in hardness improvement was investigated.

Table 2. Statistical test ANOVA used by various authors to identify the best contributing factor for property improvement.

S. No	Material used for the study	Type of study	Statistical test used	Factors tested	Number of levels	Best contributing factor	References
1	A319	Wear studies	ANOVA	Rotating speed, transverse feed, tool pin diameter	3	Tool pin diameter	[24]
2	LM4 + WC	Wear studies	ANOVA	Load, sliding speed, wt.%	4	Load (72.56%)	[23]
3	AlSi5Cu3 + B_4C	Wear studies	ANOVA	Load, speed, distance from outer periphery	3	Load (25.5%)	[22]
4	Al-5.7Si	Wear studies	ANOVA	Process, load, sliding distance, temperature	3	Process (92.24%)	[25]
5	LM4 + TiB_2	Friction behaviour	ANOVA	Load, wt.%, speed	3	wt.% (74.72%)	[26]
6	LM4 + TiB_2	Wear behaviour	ANOVA	Load, wt.%, speed	3	Load (56.31%)	[4]
7	LM4 + Ta/NbC	Wear behaviour	ANOVA	Load, sliding speed, wt.%	4	Load (65.74%)	[27]
8	Al-Si + SiC_p	Wear behaviour	ANOVA	Load, sliding distance, reciprocating velocity, counter surface temperature, wt.%	2	Sliding distance (43.65%) for weight loss; load (43.82%) for COF(μ)	[28]
9	Al 6061 + TiB_2	Mechanical and wear behaviour	ANOVA	Load, wt.%, sliding distance	3	–	[19]
10	Al6063 + SiC + ash	Tribological	ANOVA	Load, wt.%, speed	4	Speed with (60.16%, 40.74%) for volume loss and wear index	[29]
11	A356 + SiC_p	Operating parameters	ANOVA	Axial depth of cut, cutting speed, feed	3	Feed (43.6%)	[30]
12	Al6061 + rock dust	Turning operation	ANOVA	Particle size, wt.%, speed, feed, depth of cut	3	Feed (88.92%)	[31]
13	CuNiSi + Si_3N_4	Wear behaviour	ANOVA	Load, sliding velocity, sliding distance	3	Load (50.15%)	[32]
14	Al7075 + SiC + Al_2O_3	Wear behaviour	ANOVA	Load, sliding speed, hybrid composite combination	3	Load (53.86%)	[33]
15	LM25 + TiC	Mechanical and adhesive wear	ANOVA	Load, sliding velocity, sliding distance	3	Load (54.2%)	[20]
16	Medium carbon steel	Mechanical properties	ANOVA	Quenchant, temperature, soaking time	3	Soaking time (31.95% for hardness, 62.46% for YS, 66.76% for UTS)	[34]

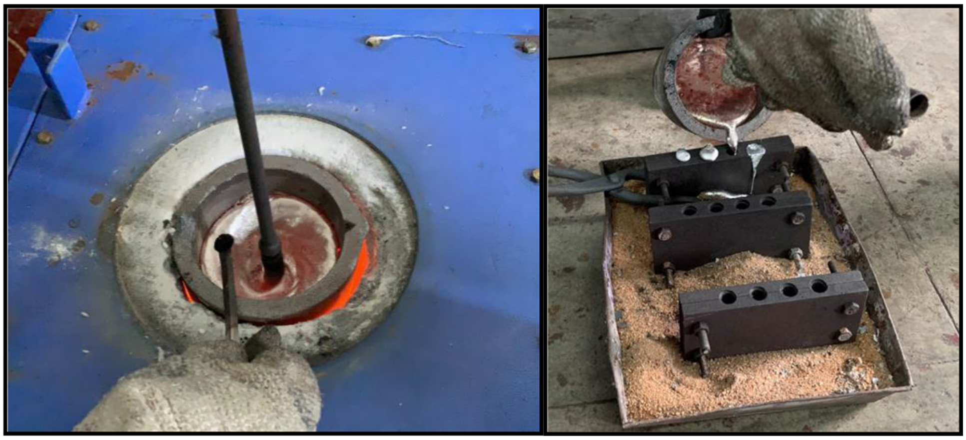

Fig. 2. Stir casting process used for present study.

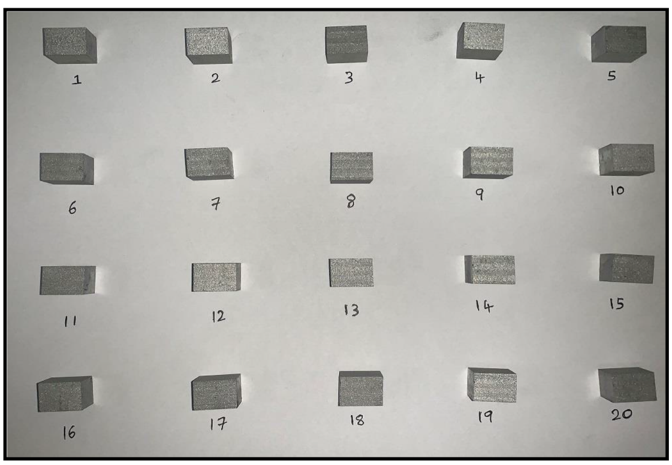

Fig. 3. Wire-ED machined samples used for hardness measurement after aging treatment.

2 Materials and experimental methodology

In this work, LM4 cast alloy was selected as matrix material, and as a reinforcement material TiB_2 (1, 2 and 3 wt.%) powder with an average particle size of 6.765 µm was selected. Stir casting method was chosen to fabricate the composites, for this LM4 alloy was melted (at 780 °C) using a furnace in a 2 kg crucible. TiB_2 reinforcement powders were preheated at different temperatures (600, 500, 450, 350, and 250 °C) before adding to the molten metal. The treated TiB_2 particles were added to the vortex (formed during stirring action) of the melt and stirred continuously for 10 min. Mechanical stirring helps with better reinforcement distribution in the matrix material. Composite melt is poured (at 730 °C) into the molds (preheated at 500 °C for 1 h) and allowed to solidify. Stir casting process is shown in Figure 2.

Hardness samples are machined from the as-cast composite samples using wire EDM as shown in Figure 3. Now, the samples are divided into two sets and subjected to SSHT (520 °C for 2 h) and MSHT (495 °C for 2 h and 520 °C for 4 h) followed by hot water quenching at 60 °C. Solutionized samples were subjected to artificial aging treatments at 100 and 200 °C separately and hardness measurements were noted at different aging times till peak age hardness is obtained. To measure the hardness of alloy and composites Micro Vickers Hardness Tester, Model – MMT X 7A was used with 200 gmf load and 15 seconds dwell time. Before performing the hardness test all the samples were polished using a disc polishing system with a velvet cloth impregnated with diamond paste so that the oxide layer and other impurities produced during heat treatment can be removed. ANOVA is performed using MINITAB 15 and type of solution heat treatment, aging temperature, wt.% of TiB_2 were taken as factors. Here the null hypothesis (H_n) and alternative hypothesis (H_a) are defined as follows, H_n: Defined factors are not responsible for achieving peak hardness, H_a: Defined factors are responsible for achieving peak hardness. Factors and levels used for ANOVA are shown in Table 3.

Table 3. Factors and their levels.

Factor	Levels	Values
wt.%	3	1, 2, 3
Solutionizing type	2	MSHT, SSHT
Aging temperature in °C	2	100, 200

3 Results and discussion

3.1 Preheating of TiB_2 reinforcement powder

As received TiB_2 powder particles may have absorbed moisture as well as other volatile and nonvolatile impurities. Figures 4a and 4b shows the Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Analysis (EDAX) report of as received TiB_2 reinforcement powder. All major peaks of EDAX correspond to Ti and B. From SEM we can observe that the powder has finer TiB_2 particles as per the requirement.

To achieve uniform distribution of TiB_2 in aluminium melt different researchers have tried different preheating temperatures. Poria et al. [35] preheated TiB_2 powder at

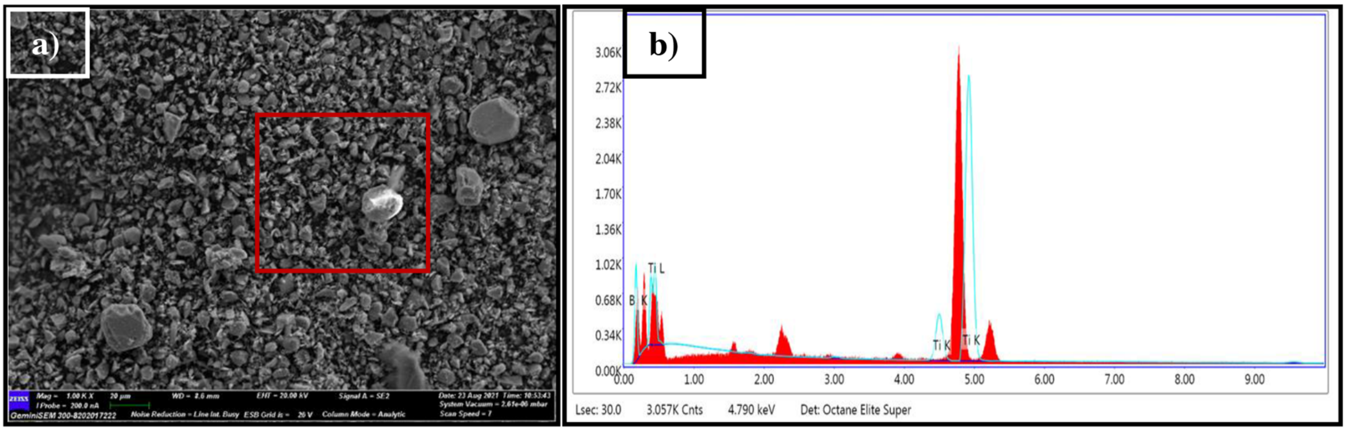

Fig. 4. (a and b) SEM and EDAX report of TiB_2 reinforcement powder.

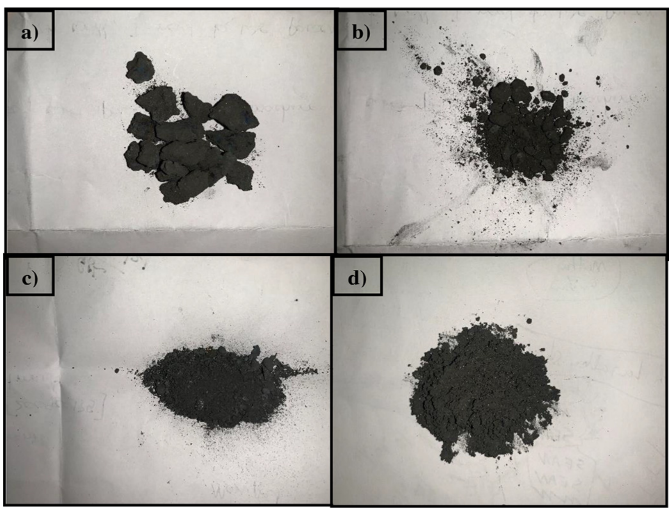

Fig. 5. TiB_2 powder preheated at, (a) 600 °C for 5 min, (b) 500 °C for 5 min, (c) 450 °C for 5 min, (d) 350 °C for 30 min.

600 °C, Dipankar et al. [6] preheated TiB_2 at 450 °C for 30 min, Ramesh et al. [5] preheated TiB_2 at 500 °C and Pazhouhanfar et al. [7] preheated TiB_2 at 250 °C for 2 h to get uniform distribution. In all the above-mentioned cases authors have stated that better results were observed with their respective preheating temperatures. Similarly, to optimize the preheating temperature in the present work the reinforcement powder are preheated at 600, 500, 450, 350, and 250 °C for different durations of time. The macro photograph of TiB_2 powders preheated at 600, 500, 450, and 350 °C is shown in Figure 5. Preheating at 600 °C for 5 min induced sintering, resulting in lumpy particles as shown in Figure 5a, this cannot be added to the liquid melt since it produces agglomeration. When preheated at 500 and 450 °C for 5 min (Figs. 5b, 5c) reduced sintering nature was observed but finer agglomerates were present which hinders the free flow of particles. Preheating at 350 °C for 30 min (Fig. 5d) has improved the free flow of particles and reinforcement is used for dispersion in molten melt.

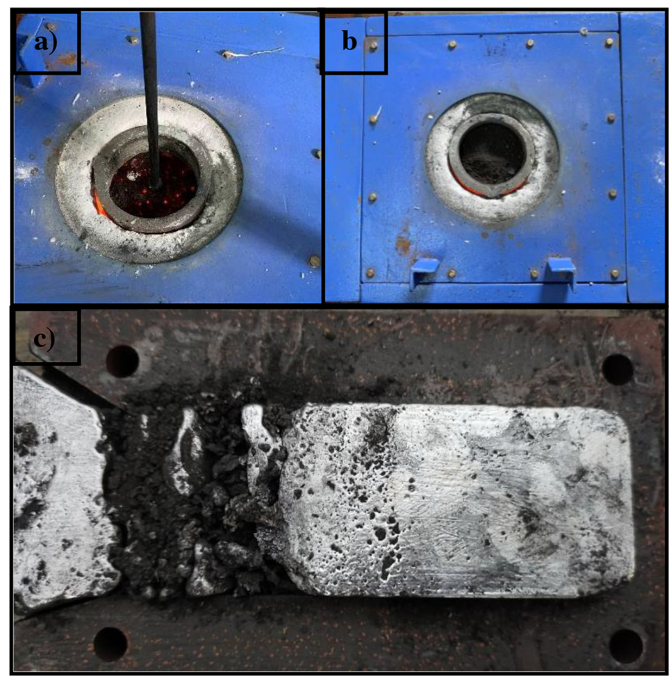

Fig. 6. Effect of preheating TiB_2 at 250 °C for 30 min, (a, b) TiB_2 popped out of molten melt, (c) Bar mold sample after pouring and solidification.

Now when TiB_2 powder is preheated at 250 °C for 30 min, lumps formation was not observed, and free flow of particles was observed similar to preheating at 350 °C. While casting, particles preheated at 250 °C for 30 min started to pop out of the molten melt as shown in Figures 6a, 6b due to lack of wettability, the bar mold specimen after pouring and solidification is shown in Figure 6c. These results clearly show that at higher preheating temperatures (600, 500, and 450 °C) densification and sintering of particles were observed, preheating at 250 °C for 30 min was not enough to get good wettability. The idle parameter for preheating TiB_2 is 350 °C for 30 min. LM4 + TiB_2 (1, 2 and 3 wt.%) samples were prepared with 350 °C as preheating temperature of TiB_2. Optical microscope images

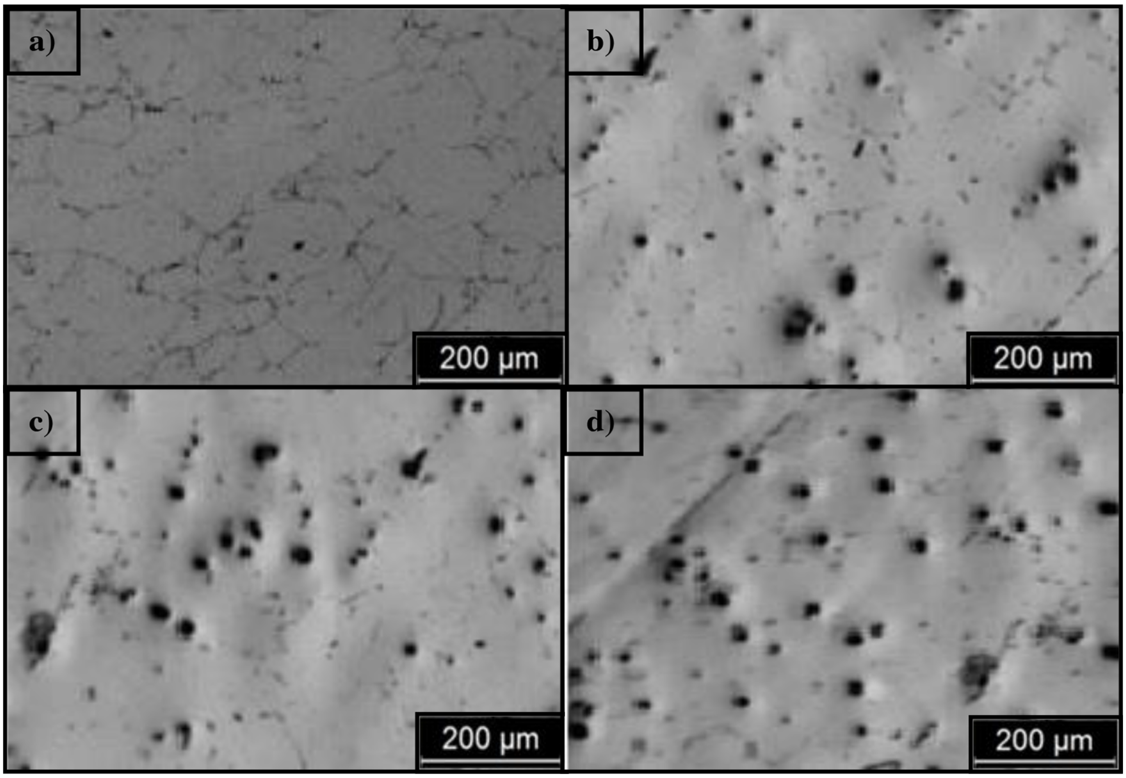

Fig. 7. Optical microstructure images of (a) LM4 alloy, (b) LM4 + 1 wt.% TiB_2, (c) LM4 + 2 wt.% TiB_2, (d) LM4 + 3 wt.% TiB_2 composites.

of LM4 alloy and LM4 + TiB_2 (1, 2 and 3 wt.%) composites are shown in Figure 7. From Figures 7b, 7c and 7d) it is evident that the uniform distribution of reinforcement in matrix material took place, and agglomeration of particles was not observed, dark spots are considered to be reinforcement particles. LM4 + TiB_2 (5 wt.%) composite was also prepared, from its optical microstructure as shown in Figure 8, it is apparent that agglomeration occurred as a result of the small particle size and greater quantity of reinforcing powder. As agglomeration was observed in 5 wt.% TiB_2 samples they were not considered for further analysis and testing. So, hardness testing and analysis were performed only for LM4 alloy and LM4 + TiB_2 (1, 2 and 3 wt.%) composites which are discussed in the next sections.

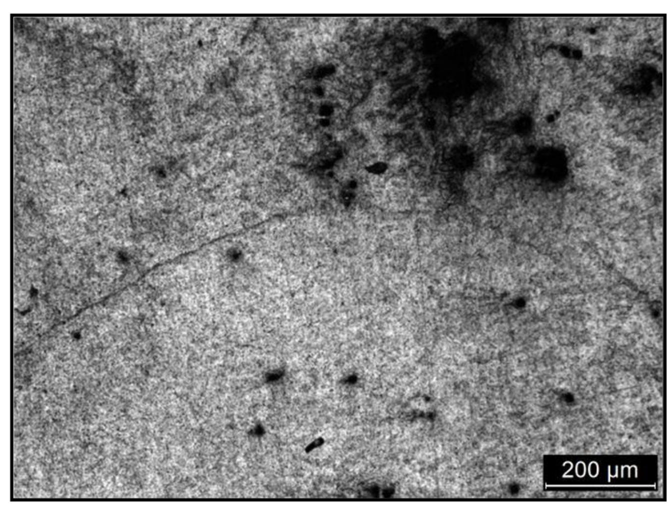

Fig. 8. Optical microstructures image of LM4 + 5 wt.% TiB_2 composite.

3.2 Hardness measurement for as-cast and LM4 + TiB_2 age hardened samples

Vickers Hardness test was performed on both as-cast and LM4 + TiB_2 (1, 2 and 3 wt.%) age hardened samples. Both the LM4 alloy and its composites show a progressive improvement in hardness with aging time. The hardness values steadily decrease after reaching their peak hardness due to over aging. At 100 and 200 °C aging temperatures, the time to attain peak hardness is found to be decreasing as the wt.% of reinforcement increases. As-cast LM4 alloy had a VHN of 70, but as-cast LM4 + TiB_2 (1, 2 and 3 wt.%) composites had 89, 95, and 103 VHN, respectively. The presence of hard dispersoids, which positively contribute to the hardness of composites, is primarily responsible for the increase in hardness of as-cast composites. The matrix deforms plastically to meet the reinforcement particles' smaller volume expansion, resulting in greater dislocation density. Increased dislocation density increases plastic deformation resistance and contributes to an increase in composite hardness. The current experimental findings are congruent with the published study findings [36–38]. Figures 9a–9d shows the hardness values of both SSHT and MSHT for different aging temperatures at 100 and

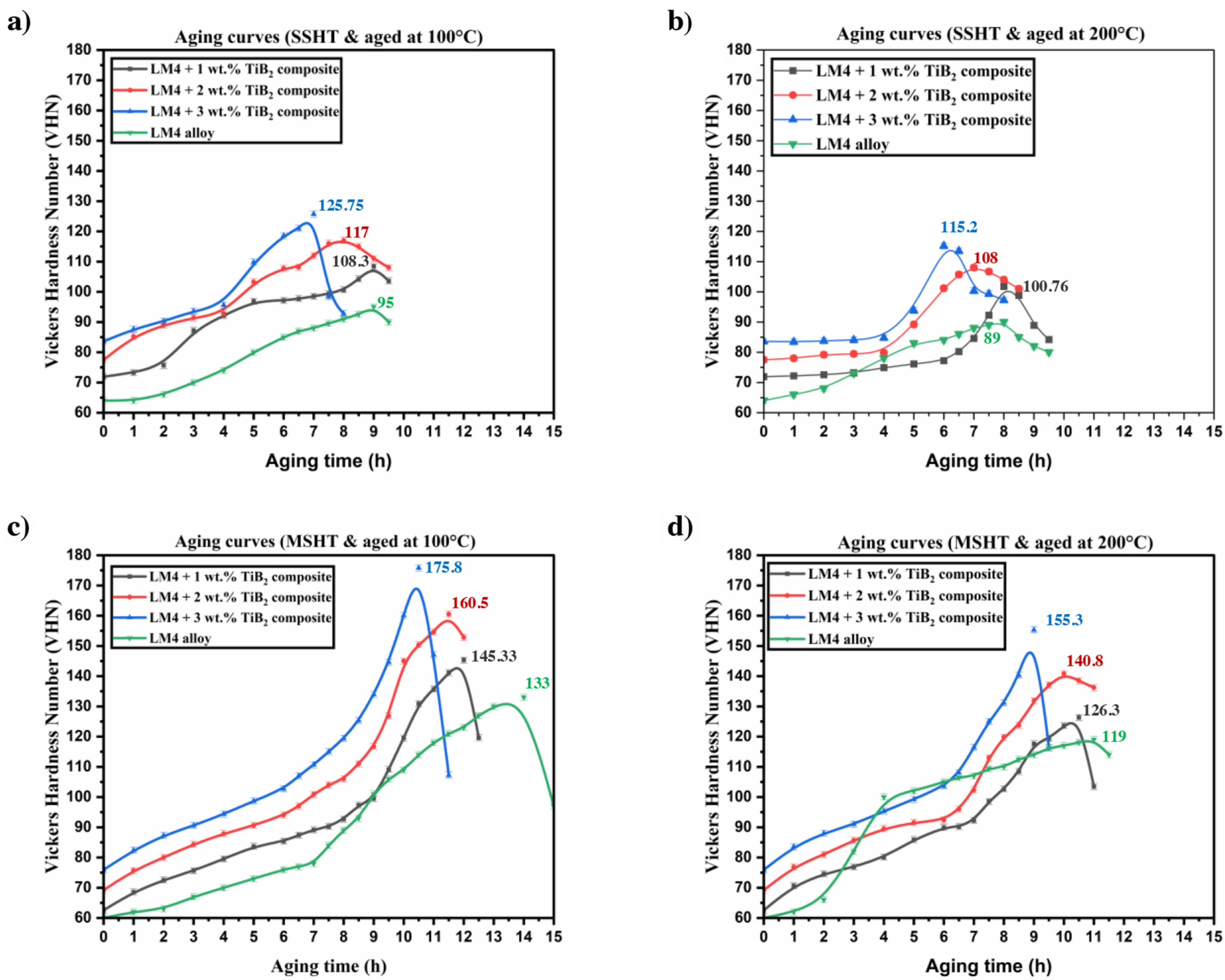

Fig. 9. (a–d) Aging curves of LM4 alloy and its composites for different aging treatment cycle.

200 °C for different aging times for both LM4 alloy and LM4 + TiB_2 composites. Compared to as-cast LM4 alloy, 80–150% improvement in hardness was observed when aged at 100 °C and 65–120% improvement in hardness was observed at 200 °C during SSHT and MSHT, respectively. Aging contributes to hardness improvement by precipitating solute-rich phases from supersaturated solid solution [36]. Lower aging temperatures (100 °C) have higher hardness than higher aging temperatures (200 °C); nevertheless, the time required to achieve peak hardness at 100 °C is longer, which may be explained by aging kinetics [39]. MSHT specimens are harder than SSHT specimens. Improvement in hardness values is due to the complete homogeneity of secondary solute-rich phases at room temperature throughout the multistage solutionizing process. The increase in hardness of peak aged samples compared to as-cast and other samples are due to the presence of metastable phases (strengthening phases) θ'-Al_2Cu and θ''-Al_3Cu which are responsible for peak aging. During the aging process, these phases will precipitate as fine precipitates inside the matrix, resulting in a considerable improvement in hardness, also more number of precipitates were observed in MSHT samples aged at 100 °C than that of SSHT aged at 100 °C [40,41], which is the reason why LM4 + TiB_2 (3 wt.%) MSHT samples aged at 100 °C displayed the highest hardness.

3.3 ANOVA

Table 4 shows the experimental results of peak hardness for TiB_2 (1, 2 and 3 wt.%) subjected to SSHT and MSHT followed by artificial aging at 100 and 200 °C along with S/N ratio and mean values obtained after performing ANOVA. The ANOVA test is used to determine the statistical significance of the goodness of fit. Standard Error (SE), adjusted R-Sq (Adj. R-Sq), and R-Sq are computed and reported in Table 5. At 95% confidence level, the estimated values of 'F-ratio' are more than tabulated values, indicating that the experiments are effective. The p-value indicates the probability of rejecting the null

Table 4. Experimental results for peak hardness along with mean and S/N ratio values.

S. No	Material	Solutionizing type	Aging temperature in °C	Peak hardness (VHN)	S/N ratio	Mean
1	LM4 + 1 wt.% TiB_2	SSHT	100	108.3	40.6926	108.30
2	LM4 + 2 wt.% TiB_2	SSHT	100	117	41.3637	117.00
3	LM4 + 3 wt.% TiB_2	SSHT	100	125.75	41.9902	125.75
4	LM4 + 1 wt.% TiB_2	MSHT	100	145.3	43.2453	145.30
5	LM4 + 2 wt.% TiB_2	MSHT	100	160.5	44.1095	160.50
6	LM4 + 3 wt.% TiB_2	MSHT	100	175.8	44.9004	175.80
7	LM4 + 1 wt.% TiB_2	SSHT	200	100.76	40.0658	100.76
8	LM4 + 2 wt.% TiB_2	SSHT	200	108	40.6685	108.00
9	LM4 + 3 wt.% TiB_2	SSHT	200	115.2	41.2290	115.20
10	LM4 + 1 wt.% TiB_2	MSHT	200	126.3	42.0281	126.30
11	LM4 + 2 wt.% TiB_2	MSHT	200	140.8	42.9721	140.80
12	LM4 + 3 wt.% TiB_2	MSHT	200	155.3	43.8234	155.30

Table 5. ANOVA for peak hardness.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
wt.%	2	1044.02	1044.02	522.01	3663.00	<0.001
Solutionizing type	1	4369.70	4369.70	4369.70	30662.78	<0.001
Aging temperature	1	620.50	620.50	620.50	4354.11	<0.001
wt.%*Solutionizing type	2	95.29	95.29	47.64	334.33	0.003
Solutionizing type*Aging temperature	1	85.92	85.92	85.92	602.92	0.002
wt.%*Aging temperature	2	2.54	2.54	1.27	8.93	0.101
Error	2	0.29	0.29	0.14		
Total	11	6218.26				

$S = 0.377503$ R-Sq = 100.00% R-Sq(adj) = 99.97%.

hypothesis when it is actually true. If the value of p is less than or equal to a predetermined significance level (0.05), the null hypothesis is rejected, and the alternative hypothesis is considered true. Null hypothesis cannot be rejected if the p-value is greater than 0.05. The p-value (<0.001) of three factors in the ANOVA for peak hardness is sufficient evidence to infer that the experiments and methods followed are adequate. R^2 value is 100% which means that the factors explain 100% of the variation in the response which is hardness. One major limitation is that if the number of independent variables are increased then the R^2 value will also increase, which is misleading. To counter that R^2(adj) is used which will add a penalty. Whereas in the present study, R^2(adj) value is 99.97% which indicates that the model is a good fit. Main effect plot for peak hardness, S/N ratio, and interaction plot for peak hardness are shown in Figures 10–12.

Main effect plots for peak hardness and S/N ratios for different wt.% of reinforcements, solutionizing type, and aging temperature are shown in Figure 11, it is evident that 3 wt.% with MSHT and 100 °C aging displayed the maximum hardness value when compared to other combinations.

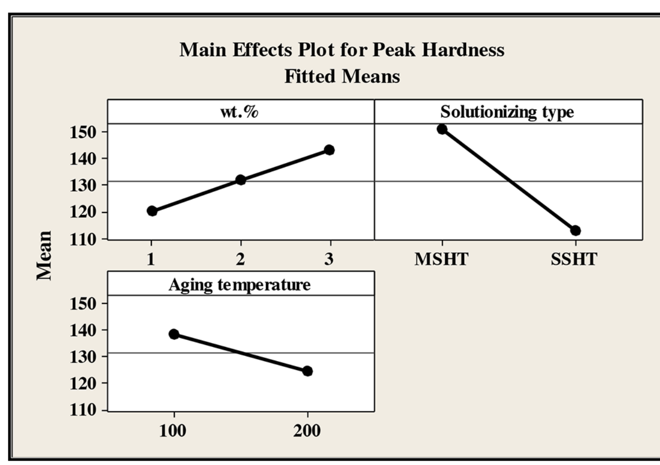

Fig. 10. Main effects plot for peak hardness.

Table 6 displays the response table for S/N ratio of peak hardness. The factor solutionizing type has the greatest effect on peak hardness, followed by weight percent and aging temperature.

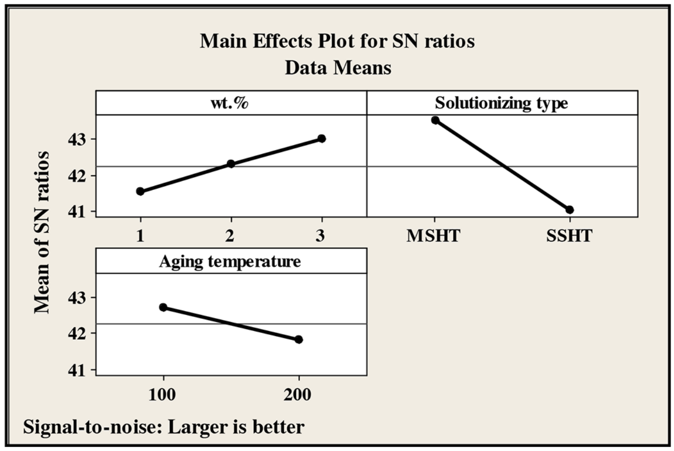

Fig. 11. Main effect plot for S/N ratios for peak hardness.

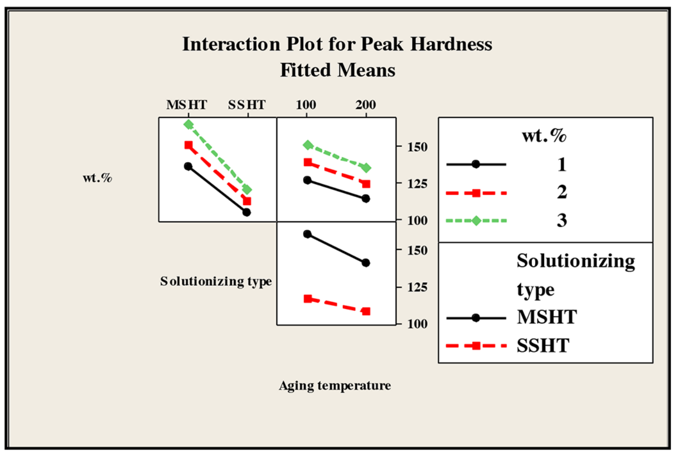

Fig. 12. Interaction plot for peak hardness.

Table 6. Response table for signal to noise ratios.

Level	wt.%	Solutionizing type	Aging temperature
1	41.51	43.51	42.72
2	42.28	41.00	41.80
3	42.99	–	–
Delta	1.48	2.51	0.92
Rank	**2**	**1**	**3**

4 Conclusions

The following conclusions were drawn from the present study:

- It was observed that when preheated at 600, 500, 450 °C for 5 min TiB_2 powder formed lumps and was not available in a state to perform casting.
- When preheated at 250 °C for 30 min lump formation was not observed but because of poor wettability casting was not successful.
- The optimum preheating temperature of TiB_2 powder for effective preparation and uniform distribution of reinforcements in the composites is at 350 °C for 30 min, which is confirmed by the optical micrographs.
- Micro Vickers Hardness results revealed that with an increase in wt.% of TiB_2 hardness of composites increased, also multistage solutionizing followed by artificial aging at 100 °C was proven to achieve the highest peak hardness value for LM4 + 3 wt.% TiB_2 composites. When SSHT and MSHT were performed on as-cast LM4 alloy, hardness increased by 80–150% at 100 °C aging and 65–120% at 200 °C aging temperatures, respectively.
- ANOVA was performed with wt.%, solutionizing type and aging temperatures as factors, and peak hardness as the outcome. R^2 value (100%) confirms that all three factors contributed effectively for achieving the peak hardness.

– From the conclusions drawn, this work can be extended to concentrate on the multistage aging concept. Also, the effect of deformation behaviour synchronized with aging treatment, known as thermomechanical treatment, can be performed and investigated.

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