Optimization of preheating temperature for TiB<sub>2</sub> reinforcement on the preparation of stir cast LM4 + TiB<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub> (1, 2 and 3 wt.%) composites. The impact of preheating TiB<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub> (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<sub>2</sub> 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<sup>2</sup> 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<sub>2</sub>) 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<sub>2</sub> 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].
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 + TiB2 (0, 3, 6 and 9 wt.%) composites using stir casting method where TiB2 particles are preheated at 450°C for 30 min. From results it was concluded that dislocation density improved between matrix and reinforcement as TiB2 addition refined aluminum alloy grains and improved the microhardness of Al2024-TiB2 composites. Ramesh et al. [5] investigated the effect of preheated TiB2 reinforcement powder on mechanical properties of AA7075 + TiB2 (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 TiB2 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 TiB2. Pazhouhanfar et al. [7] prepared Al6061 composites with TiB2 (3, 6 and 9 wt.%) as reinforcements. K2TiF6 (potassium titanium fluoride) was added to the melt to improve the wettability of TiB2 in the matrix, also TiB2 particles were preheated at 250°C for 2 h. They concluded from the results that the addition of K2TiF6 and preheating of TiB2 resulted in uniform reinforcement distribution of TiB2 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 + TiB2 (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.% TiB2 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 Al2O3 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 Al2O3 with Cu, Ni, and Co separately and prepared A356 composites. From results, they concluded that coating improved the wettability of Al2O3 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

![Methods to improve the wettability](image)

**Fig. 1.** Different methods to improve wettability.
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.

### 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 | TiB₂ (0, 4, 8, 12 wt.%) | 500                                      | –                                    | 135 VHN @ 12 wt.% addition         | [5]        |
| 2     | Al2024 | TiB₂ (0, 3, 6, 9 wt.%) | 450                                      | 30                                   | 129 VHN @ 9 wt.% addition          | [6]        |
| 3     | Al7075 | TiB₂ (0, 3, 6, 9 wt.%) | 450                                      | –                                    | –                                  | [10]       |
| 4     | A356   | Nano SiCₚ    | 850 (calcination), 250 (preheating)      | 120, NA                              | –                                  | [13]       |
| 5     | A356   | SiC and Al₂O₃ coated with Ni and Cu | 350                                      | 120                                   | 118 HV @ Ni coated Al₂O₃ + Cu coated SiC | [11]       |
| 6     | A356   | Al₂O₃ and SiC coated with Cu | 350                                      | 120                                   | 190 HV @ 1.5 wt.% Al₂O₃ and 1.5 wt.% SiC addition | [16]       |
| 7     | Al6061 | TiB₂ (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 + Al₂O₃ | 800                                      | 120                                   | –                                  | [18]       |
| 10    | Al6061 | TiB₂ (0, 2, 4, 6, 8 wt.%) | 670                                      | –                                    | 73.93 HV @ 10 wt.% TiB₂ addition | [19]       |
| 11    | LM25   | TiC (10 wt.%) | 350                                      | 25                                   | 129 HV @10 wt.% of TiC addition    | [20]       |

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 + B₄C 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 TiB₂ reinforced composites. So, in this article, the effect of preheating temperature of TiB₂ reinforcement powder on the composite preparation was studied and optimum preheating temperature to attain uniform distribution of reinforcements in the matrix was accomplished. LM4 + TiB₂ 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 + B4C             | 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 + TiB2                 | Friction behaviour | ANOVA              | Load, wt.%, speed                                                               | 3               | wt.% (74.72%)                             | [26]       |
| 6     | LM4 + TiB2                 | 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 + SiCp               | Wear behaviour | ANOVA                 | Process, load, sliding distance, temperature, counter surface temperature, wt.% | 3               | Speed with (60.16%, 40.74%) for volume loss and wear index | [29]       |
| 9     | Al6061 + TiB2              | Mechanical and wear behaviour | ANOVA              | Load, wt.%, sliding distance                                                    | 3               | -                                         | [19]       |
| 10    | Al6063 + SiC + ash         | Tribological  | ANOVA                 | Load, wt.%, speed                                                               | 4               | Feed (43.6%)                             | [29]       |
| 11    | A356 + SiCp                | Operating parameters | ANOVA              | Axial depth of cut, cutting speed, feed                                         | 3               | Feed (88.92%)                            | [31]       |
| 12    | Al6061 + rock dust         | Turning operation | ANOVA              | Particle size, wt.%, speed, feed, depth of cut                                   | 3               | Feed (43.6%)                             | [30]       |
| 13    | CuNiSi + Si3N4             | Wear behaviour  | ANOVA                 | Load, sliding velocity, sliding distance                                         | 3               | Feed (50.15%)                            | [32]       |
| 14    | Al7075 + SiC + Al2O3       | 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]       |
2 Materials and experimental methodology

In this work, LM4 cast alloy was selected as matrix material, and as a reinforcement material TiB₂ (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. TiB₂ reinforcement powders were preheated at different temperatures (600, 500, 450, 350, and 250°C) before adding to the molten metal. The treated TiB₂ 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 gms 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 TiB₂ were taken as factors. Here the null hypothesis ($H_0$) and alternative hypothesis ($H_a$) are defined as follows, $H_0$: 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.

3 Results and discussion

3.1 Preheating of TiB₂ reinforcement powder

As received TiB₂ 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 TiB₂ reinforcement powder. All major peaks of EDAX correspond to Ti and B. From SEM we can observe that the powder has finer TiB₂ particles as per the requirement.

To achieve uniform distribution of TiB₂ in aluminium melt different researchers have tried different preheating temperatures. Poria et al. [35] preheated TiB₂ powder at
600°C, Dipankar et al. [6] preheated TiB₂ at 450°C for 30 min, Ramesh et al. [5] preheated TiB₂ at 500°C and Pazhouhanfar et al. [7] preheated TiB₂ 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 TiB₂ 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.

Now when TiB₂ 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 TiB₂ is 350°C for 30 min. LM4 + TiB₂ (1, 2 and 3 wt.%) samples were prepared with 350°C as preheating temperature of TiB₂. Optical microscope images

Fig. 4. (a and b) SEM and EDAX report of TiB₂ reinforcement powder.

Fig. 5. TiB₂ 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.

Fig. 6. Effect of preheating TiB₂ at 250°C for 30 min, (a, b) TiB₂ popped out of molten melt, (c) Bar mold sample after pouring and solidification.
of LM4 alloy and LM4 + TiB₂ (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 + TiB₂ (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.% TiB₂ samples they were not considered for further analysis and testing. So, hardness testing and analysis were performed only for LM4 alloy and LM4 + TiB₂ (1, 2 and 3 wt.%) composites which are discussed in the next sections.

### 3.2 Hardness measurement for as-cast and LM4 + TiB₂ age hardened samples

Vickers Hardness test was performed on both as-cast and LM4 + TiB₂ (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 + TiB₂ (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].

**Fig. 7.** Optical microstructure images of (a) LM4 alloy, (b) LM4 + 1 wt.% TiB₂, (c) LM4 + 2 wt.% TiB₂, (d) LM4 + 3 wt.% TiB₂ composites.

**Fig. 8.** Optical microstructures image of LM4 + 5 wt.% TiB₂ composite.


200°C for different aging times for both LM4 alloy and LM4 + TiB₂ 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) \( \theta^\prime \)-Al₂Cu and \( \theta^- \)-Al₃Cu 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 + TiB₂ (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 TiB₂ (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

![Fig. 9](image-url)
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.

\[ R^2 = 100\% \text{ and } R^2(\text{adj}) = 99.97\% . \]

Main effect plots 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.

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.% TiB₂ | SSHT               | 100                      | 108.3               | 40.6926   | 108.30|
| 2     | LM4 + 2 wt.% TiB₂ | SSHT               | 100                      | 117                 | 41.3637   | 117.00|
| 3     | LM4 + 3 wt.% TiB₂ | SSHT               | 100                      | 125.75              | 41.9902   | 125.75|
| 4     | LM4 + 1 wt.% TiB₂ | MSHT               | 100                      | 145.3               | 43.2453   | 145.30|
| 5     | LM4 + 2 wt.% TiB₂ | MSHT               | 100                      | 160.5               | 44.1095   | 160.50|
| 6     | LM4 + 3 wt.% TiB₂ | MSHT               | 100                      | 175.8               | 44.9004   | 175.80|
| 7     | LM4 + 1 wt.% TiB₂ | SSHT               | 200                      | 108                 | 40.6685   | 108.00|
| 8     | LM4 + 2 wt.% TiB₂ | SSHT               | 200                      | 115.2               | 41.2290   | 115.20|
| 9     | LM4 + 3 wt.% TiB₂ | SSHT               | 200                      | 126.3               | 42.0281   | 126.30|
| 10    | LM4 + 1 wt.% TiB₂ | MSHT               | 200                      | 140.8               | 42.9721   | 140.80|
| 11    | LM4 + 2 wt.% TiB₂ | 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   |           |         |         |         |

\( S = 0.377503 \) \( R^2 = 100.00\% \) \( R^2(\text{adj}) = 99.97\% . \)

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.
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 TiB₂ 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 TiB₂ 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 TiB₂ 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.% TiB₂ 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.

**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                 |

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

**Fig. 12.** Interaction plot for 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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