Optimization of Stirring Parameters for Stir-Cast Magnesium Matrix Composites Using Response Surface Methodology

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Abstract: The response surface methodology is used to study the effect of stirring parameters on the mechanical properties of magnesium matrix composites (MMCs). The composites are manufactured using different stirring speeds (500, 600, and 700 r/min), stirring time (10, 20, and 30 min), and weight fractions (0, 2.5%, 5%, and 10%) of silicon carbide particles. The experimental results show that 700 r/min and 20 min are the best conditions for obtaining the best mechanical properties. Based on the desirability function methodology, the optimum parameter values for the best mechanical characteristics of produced composites are reached at 696.102 r/min, 19.889 min, and 9.961%(in weight).

Key words: composites; stir casting; magnesium; response surface methodology; optimization

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0 Introduction

In recent years, most sectors including aerospace, transportation, biomedicine, and telecommunications have been heavily concentrated on the use of lightweight components for various reasons, the most significant of which is to reduce project costs and maintain compliance with rules and standards[1-2]. Among the existing alloys, magnesium (Mg) alloy has become the most popular material to meet the increasing demand for lightweight due to its excellent strength-to-density ratio and other technical advantages[3-5]. However, additional constraints, including poor corrosion resistance, low room temperature formability, and low wear resistance, limit their use in many applications. By incorporating solid particles into Mg matrix alloys, magnesium matrix composites have been utilized to overcome this issue[6-8].

In particular, magnesium composites were lightweight and widely used in aviation and automotive fields compared with aluminium matrix composites[9-10]. Also, the magnesium matrix composite is a type of material made of hard ceramic reinforcement and magnesium matrix integrating metallic features with ceramic characteristics, resulting in increased strength and wear performance[11-12]. This leads to composites with good strength for magnesium matrix with higher wear resistance, superior thermal and electric conductivity, and good damping efficiency[13-14].

Production methods for magnesium composites are often classified into three groups based on the state of the process: (1) Liquid-state methods, (2) solid-

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state methods, and (3) gaseous-state methods[15]. To get the optimum mechanical properties of magnesium composites, solid-state processes are often used. The technology is utilized to produce fine grain management of composite microstructure and reinforcing application. Several magnesium composite diffusion bonding and powder metallurgy processes are now in use. Like steam deposition, gaseous-state processing is a fundamental process in which the matrix is deposited in single elements of the product from the steam phase[16-17]. The matrix is absorbed by plasma, solid vapor deposition, or chemical vapor deposition deposits. Liquid-state processing is widely used among them due to its low cost and natural manufacturing technique[18-19].

Throughout the last few years, magnesium composites produced by stir casting have piqued the interest of researchers[20-22]. For example, Mohammadi et al.[23] produced and assessed the mechanical characteristics of AZ91 alloys supplemented with varying weight percentages of B_4C particles via stir casting. Here the chemical composition of AZ91 alloy is 9%AL, 0.7%Zn and 0.2%Mn (weiqht percent). Based on their findings, the compressive and tensile strength in the obtained composites was significantly raised compared with the AZ91 alloys with an increasing weight fraction of reinforcements. In another similar work, Khalili et al.[24] created Mg/HA surface metal matrix nano-composites using stir-centrifugal casting in order to design the ideal condition for generating a homogeneous composite as a prospective bone implant. Also, they established a statistical model to anticipate the best parameters of casting and identified effective parameters such as weight percent of HA, mold rotational speed and propeller rotational time. Their finding revealed that the maximum uniform dispersion mechanical behavior could be obtained at 1.8% nano-HA in Mg matrix. In another related study, Aatthisugan and Murugesan[25] studied the effect of silicon addition on magnesium matrix composites prepared by the stir casting process in order to improve the mechanical characteristics and wear performance. Based on their findings, the mechanical characteristics of magnesium matrix alloy are significantly improved after Si element is added. Recently, Saleh et al.[26] produced homogenous AZ91-SiC composites with different weight fractions of particles via the stir casting process. They studied the effect of material parameters on microstructure behavior and mechanical properties. Their findings showed that the mechanical properties of the produced composites improved as the weight fractions of the particle increases.

From the above literature, it can be seen that the effect of stirring parameters on mechanical properties of AZ91 alloy reinforced with SiC particles has not been widely concluded by statistical approach. Also, magnesium composites reinforced with ceramic particles including silicon carbide, aluminium oxide, and titanium carbide are still required to optimize their process parameters compared with more typical materials such as pure metals and alloys. Thus, lightweight composites can benefit from three aspects, including optimizing production parameters, generating dispersion, and using statistical analysis to predict. As a result, in the current work, the statistical investigation for the mechanical properties of stir-cast magnesium matrix composites using response surface methodology (RSM) has been explored. The effect of stirring speed, stirring time, and weight fraction of particles on the mechanical behavior of homogeneous composites has been analyzed using the Minitab software version 19.

1 Materials and Methods

1.1 Materials and fabrication process

In this study, AZ91 magnesium alloy is used as matrix to prepare homogenous composites by the stir casting method, which is widely used in aerospace, automotive, energy, and other fields. The homogenous composites are strengthened by four different weight fractions of SiC particles with an average size of 10 μm, due to its distinct properties such as high hardness and wear resistance. The properties used particles have terribly hardness and high wear resistance, thus adding ceramic particles to the AZ91 matrix alloy can significantly enhance mechanical characteristics and wear resistance.

The stir casting method is used to prepare homogenous composites with a size of 200 mm×150 mm×40 mm. The AZ91 matrix alloy is melted in a graphite crucible at 700 ℃, while the SiC particles are simultaneously heated to 300 ℃ for 2 h. The dried particles are added to the molten metal and stirred for 10, 20, and 30 min at different stirring speeds of 500, 600, and 700 r/min before pouring into the preheat steel die (400 ℃) to obtain samples. In the previous publication[26], the stir casting process is given with more details.

1.2 Tests and analysis techniques

In order to check the particle distribution of the prepared composites, the specimen is cut with a size of 10 mm×10 mm×2 mm by the wire electrical discharge machine. The initial polishing is performed on 320, 400, 800, 1 500, and 2 000 grits by using emery papers. The final polishing is carried out with 0.05 μm liquid alumina to form a mirror surface, and then the boundary is visualized with the acetic-picric solution for 5 s as an etchant. A scanning electron microscope with a field emission (SEM, Sirion 200) is used to monitor particle distribution.

The Vickers hardness test is performed on specimens of the prepared composites with various weight fractions of particles. The HXD-1000TM/LCD is used as a hardness testing machine for a 100 gf load with a dwelling period of 15 s. The average of five hardness values is measured in order to obtain a consistent average value.

The tensile test is prepared according to ASTM E8M on the Universal tensile testing machine (Model: UTM4294X) on both AZ91 alloy and composites to check the distribution of reinforcement particles. The tensile test is conducted with a strain rate of 5×10^{-4} s^{-1} at 25 ℃.

1.3 Response surface methodology

In the current study, stirring speed, stirring time, and hard particle weight fraction are chosen as significant parameters to determine the optimum values of stirring parameters on mechanical properties using RSM. Because RSM confirms and optimizes the design parameters of the empirical model, as well as recognizes the existence of various factors that affect these parameters by a polynomial formula for experimental results. And it also describes the conduction of the data set[27].

Three preparation parameters are selected with different levels, as seen in Table 1. Twenty experimental runs are constructed for the chosen three parameters. Polynomial equations are prepared for the account of the measured responses (hardness and tensile strength) as a function of the test parameters with six coefficients.

Table 1 Preparation parameter values and their levels

Parameter	Level			
Stirring speed/ $(r\cdot m^{-1})$	500	600	700	
Stirring time/ min	10	20	30	
Weight fraction/%	0	2.5	5	10

2 Results and Discussion

2.1 Microstructure evaluation

Among all parameters, the distribution of hard particles inside the soft matrix plays a vital role in improving the characteristics of the prepared composites. As the ceramic reinforcements added into the magnesium matrix alloy have a good impact on strengthening the mechanical characteristics of the manufactured composite. This is because AZ91 is a soft matrix, and the solid particles are harder, which helps to improve the characteristics of the prepared composite. However, the constraint to plastic deformation relies on the SiC particle dispersion inside the matrix. In case the particle distribution is obscured in a certain region and absent from a specific spot, the characteristics of the generated composites will alter considerably. Therefore, it is vital to seek for better dispersion of particles inside the matrix by adjusting the stirring conditions. Fig.1 illustrates the microstructures of magnesium matrix composites (MMCs) formed at various stirring speeds (500, 600, and 700 r/min) under a constant stirring duration of 20 min. It can be noted that the stirring speed effects the distribution of particles inside the Mg matrix alloy and the SiC particles are approximately homogeneous at a stirring speed of 700 r/min. Also, SEM pictures in Fig. 1 reveal that there are many SiC agglomerations at 500 and 600 r/min of stirring

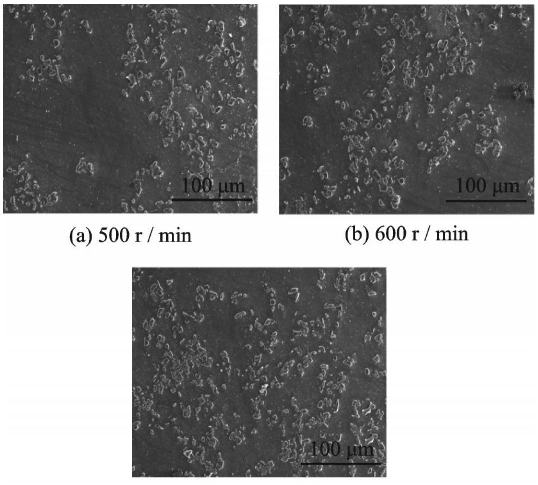

(a) 500 r / min (b) 600 r / min
(c) 700 r / min

Fig.1 Microstructures of MMCs for different stirring speeds at stirring time of 20 min

speed, where many particles clump together. This leads to a weaker contact between solid particles and the AZ91 matrix alloy, which results in a deterioration in the characteristics of the manufactured composites.

Fig.2 demonstrates the microstructure of MMCs formed at varying stirring durations (10, 20, and 30 min) with a constant stirring speed of 700 r/min. It can be noticed that the stirring time impacts the distribution of particles inside the AZ91 matrix alloy, and the solid particles are practically uniform at a stirring time of 20 min. The SiC particles are agglomerated in some local places, and there are blank areas lacking SiC particles, as demonstrated in Fig. 2. Thus, the stirring duration can impact the distribution of particles, where the shortest stirring time leads to the non-homogeneous distribution of the particles, while the longest stirring time can lead to the particles to accumlate in certain areas, as shown in Fig. 2. Even though the stirring duration influences the distribution of the particles inside the AZ91 matrix alloy and then the control of the mechanical characteristics, the stirring speed has the most notable impact on the distribution of the particles in the manufactured composites.

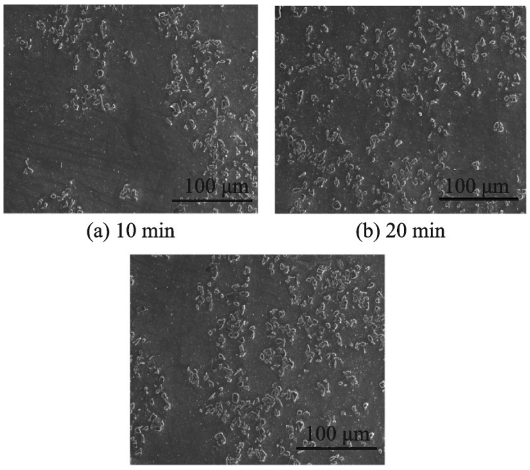

(a) 10 min (b) 20 min
(c) 30 min

Fig.2 Microstructures of MMCs for different stirring time at stirring speed of 700 r/min

2.2 Analysis of variance and model fitting

Table 2 displays the experimental data of three parameters (stirring speed, stirring time, and weight percent) with their levels for two output responses of mechanical characteristics of the fabricated composites. Analysis of variance (ANOVA) has been used to study the effects of output factors to obtain the ideal values for the main parameters and to assess the competency of the present models. The values of R^2 and Adjusted R^2 for models are provided at the end of Table 2, and these values are found closer to each other, confirming that the developed full-quadratic models are efficient in linking the responses to the components. The regression coefficients produced from the significance tests are utilized to build the models and displayed in the following Eqs.(1) and (2). The models created reveal that linear term, square term, and interaction term have an influence on the mechanical characteristics of the produced composites. The equations reveal that not only the terms of the main components are relevant but also those particular interactions and quadratic terms are proved to be significant.

$$\begin{aligned}\text{Hardness(HV)}=&37.1+0.045A+1.75B+5.7C-\\&0.000\,04A^2-0.043B^2-0.272\,4C^2+\\&0.000\,9AC+0.002\,33BC\end{aligned}\qquad(1)$$

$$\begin{aligned}\text{Strength(MPa)}=&69.5+0.14A+4.6B+7.9C-\\&0.000\,08A^2-0.113B^2+0.052C^2-\\&0.001\,8AC-0.019BC\end{aligned}\qquad(2)$$

where A is the stirring speed (r/min); B the stirring time; and C the weight fraction of particles.

The mechanical results of the uniform composites developed by the stir casting method can be determined within the range of parameters explored when measured values of variables in Eqs.(1) and

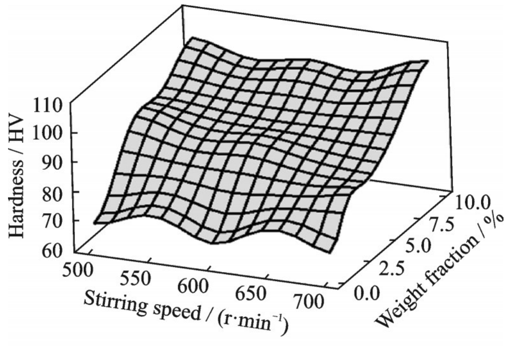
Table 2 Experimental parameters in actual units and experimental mechanical responses

No.	Main parameter			Mechanical response	
	Stirring speed/ $(\mathrm{r \cdot min^{-1}})$	Stirring time/ min	Weight fraction/ %	Vicker hardness/HV	Tensile stength/MPa
1	350	10	2.5	73	174
2	350	10	10	93	217
3	300	5	10	88	208
4	300	15	0	60	155
5	250	10	10	90	213
6	300	5	5	78	172
7	350	10	0	64	164
8	250	10	0	63	162
9	300	15	5	79	173
10	250	10	5	82	179
11	350	10	10	93	217
12	250	10	5	82	179
13	300	15	0	60	155
14	250	10	0	63	162
15	300	15	5	79	173
16	300	5	0	60	155
17	300	15	5	79	173
18	300	10	0	65	168
19	250	10	5	82	179
20	350	10	0	64	164
	R^2/%			99.91	99.65
	Adjusted R^2/%			99.86	99.47

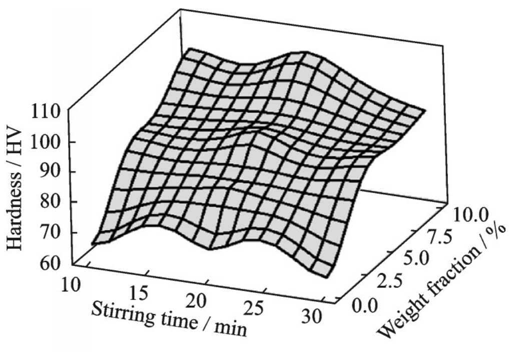
(2) are substituted.

2.3 Influence of stirring parameters on hardness

Material hardness is a fundamental characteristic that expresses the mechanical properties of surface behavior. Fig.3 presents the Vickers hardness surface response plots depending on the stirring parameters (stirring speed and stirring time) and weight fraction of particles. The surface plots also show that the Vickers hardness increases by increasing the weight fraction of particles within the matrix alloy. It is observed that the Vickers hardness performance is improved by 21.68%, 30.11%, and 38.09% for AZ91/SiCp composites for 2.5%, 5%, and 10% of hard particles, respectively, compared with AZ91 matrix alloy. This may be because the increased SiC particles surround a softer matrix, which continues to withstand wear by preferring more plastic behavior. Moreover, Fig.3 shows a sharp increase in the value of Vickers hardness at a higher level of the stirring speed and a middle level of the stirring speed. Furthermore, the effect of stirring time is much more pronounced at the middle level compared with other factors.



(a) 20 min

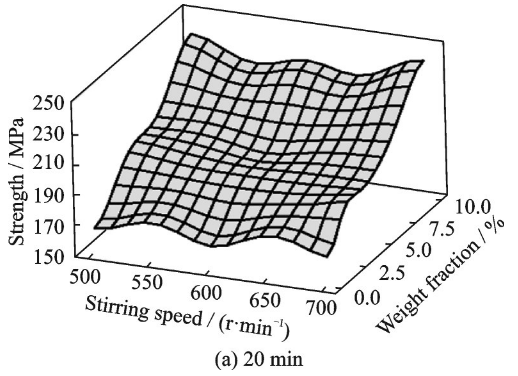


(b) 700 r / min

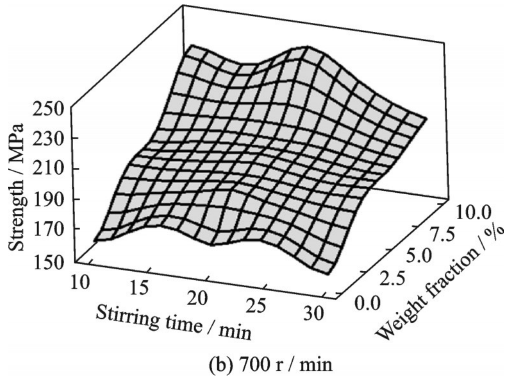
Fig.3 Effect of stirring parameters and weight fraction on hardness of MMCs

2.4 Influence of stirring parameters on tensile strength

The strength of the composite is determined by many factors such as matrix type, weight fraction, size, and type of particles, as well as bonding characteristics between matrix and particles. An increased particle reinforcement usually enhances the tensile characteristics of composites due to the strength of the SiC particles. According to the findings, there is a clear trend of enhancing tensile strength by increasing the content of the SiC particles from 0 to 10% regardless of the stirring parameters. It can be noted from Fig.4 that the tensile strength of the prepared composites enhances with the increase of the weight fraction of particles regardless of the stirring parameters. The increase in tensile strength is mainly due to the presence of hard particles in the AZ91 matrix alloy, which create a very smooth, compact, and defection-free surface due to the uniform distribution of particles produced by the stirring parameters. Also, the 3D plots have clearly shown that the enhanced tensile strength is due to the presence of solid particles, which increases the capacity to bear loads and reduces the matrix deformation by minimizing the movement of dislocation, regardless of whether they are stirring parameters, as shown in Fig.4. Additionally, Fig.4 further shows nonlinear growing contour lines that the variation in tensile strength is a function of the main parameters and the effect of stirring speed being high prominent at the higher level compared with other parameters. Furthermore, the increase of tensile strength with increasing the weight fraction of particles in the prepared composites may be due to the reduction of grain size due to the presence of particles and the increase of the effectiveness of the mechanically mixed layer in AZ91 alloy due to the uniform distribution of particles.



(a) 20 min

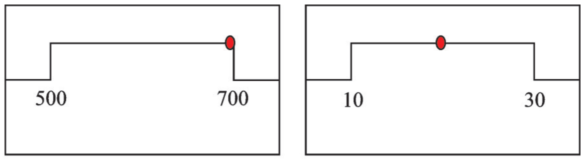


(b) 700 r / min

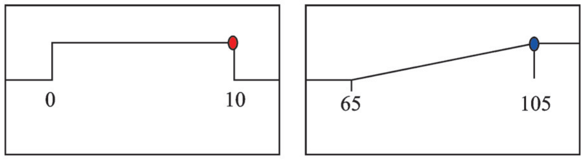
Fig.4 Effect of main parameters on tensile strength of MMCs

2.5 Numerical optimization

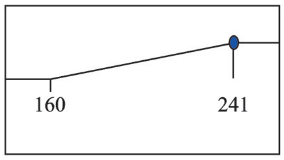
The desired objective has been selected for each parameter and response from the menu using the Minitab software version 19 for numerical optimization. The potential objectives include range, target, minimize, maximize (only for responses), and set an exact value (only for parameters). The main parameters have been set within the experimental ranges, and the responses have been set to obtain the maximum mechanical properties of the composites. In Fig.5, the point on each ramp shows the response prediction of the optimal solution for the maximum mechanical properties of the prepared composites. The optimal parameter conditions for



(a) Stirring speed=696.102 r / min (b) Stirring time=19.889 min



(c) Weight fraction=9.961% (d) Hardness=105.005 HV



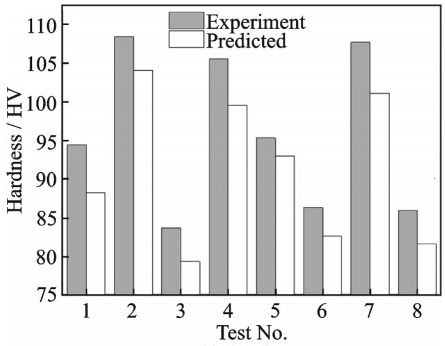
(e) Tensile strength=241.038 MPa

Fig.5 Desirability ramps for optimum values of main parameters to achieve the best mechanical properties for composites the maximum mechanical properties based on desirability function methodology are achieved at the stirring speed of 696.102 r/min, the stirring time of 19.889 min, and the weight fraction of particles of 9.961%. These optimal main parameter values are one of 91 suggestions obtained by regression models to obtain the best mechanical properties of the produced composites; thus, depending on the nature of the application, any range of these suggestions can be used to create a combination of experimental parameters.

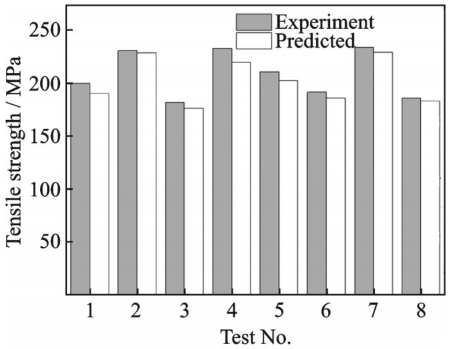
It was substantial to conduct confirmation runs after performing the mechanical tests of the fabricated MMCs. The confirmation tests are carried out with the following set of parameters to validate and ensure the adequacy of the regression models built to estimate mechanical results. Confirmation tests have been carried out for new levels of parameters other than the levels obtained in the design of the experiment table, as shown in Table 3, and in the built models, the same set of parameters is replaced for responses. The error is within 7% in all cases, which guarantees that the developed model adequately relates the main parameters to the response and effectively predict the mechanical properties of the fabricated MMCs under any parameter without actual testing, as shown in Fig.6 and Fig.7.

Table 3 Main parameters of confirmation tests with new levels

No.	1	2	3	4	5	6	7	8
Speed/ $(\mathrm{r \cdot min^{-1}})$	550	575	625	650	675	700	500	600
Time/min	10	20	30	15	25	12.5	27.5	15
Weight percentage/%	5	10	2.5	7.5	5	3	10	2.5



(a) Hardness



(b) Tensile strength

Fig.6 Experiments and predicted results for confirmation tests

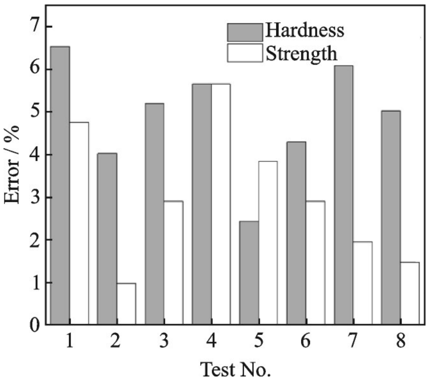


Fig.7 Statistical error comparison for confirmation tests

3 Conclusions

The empirical models are developed to predict the mechanical properties of the homogeneous composites prepared at the different production parameters. These models are constructed using RSM for several parameters, including stirring speed, stirring time, and weight fraction at different levels. The significance of all terms in the models is checked using ANOVA, and non-significant terms are removed from these models. All empirical quadratic models are found to be adequate at a confidence level of 95%. The optimal parameter settings for the maximum mechanical properties of the homogeneous composites are obtained at the stirring speed of 696.102 r/min, the stirring time of 19.889 min, and the weight fraction of particles of 9.961% based on desirability function methodology. Also, the error between the regression and the experimental values is within 7% in all cases, which guarantees that the developed model adequately relates the production conditions to the response and effectively predict the mechanical properties of the homogeneous composites in any parameter without actual testing. Finally, the composites prepared in this study have superior mechanical characteristics and are ideal for applications that require high mechanical properties, such as automotive applications.

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用响应面法优化搅拌铸造镁基复合材料的搅拌参数

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摘要：采用响应面法研究搅拌参数对镁基复合材料力学性能的影响。使用不同的搅拌速度（500、600和700 r/min）、搅拌时间（10、20和30 min）和碳化硅颗粒的重量分数（0、2.5、5和10%）制备复合材料。实验结果表明，700 r/min、20 min是获得最佳力学性能的最佳条件。根据可取性函数法，在696.102 r/min、19.889 min和9.961%时，复合材料的力学性能达到最优。

关键词：复合材料；搅拌铸造；镁；响应面方法；优化