

Chemometric Optimization of Superplastic Forming Parameters in SiC-Reinforced Aluminium Matrix Composites via Response Surface Methodology

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The rapid industrial development leads to a variety of complex shapes being manufactured in various fields, i.e., aerospace, automobile, architecture, and medical applications, with high quality at minimum cost. In this virtue, superplastic forming plays an imperative role in the production of complex shapes in aluminum alloys. The superplastic formation exhibits polycrystalline in an isotropic manner the near net shape components production with high tensile elongations. In general, superplastic forming is a long-neck-free elongation under low flow stresses. In this work, Al 7075 is used as a base material, and the SiC particles were reinforced in three various percentages, i.e., 5, 7.5, and 10 weight percentages. After the fabrication of the composites for further improvement of the mechanical properties, the composite was hot-rolled and the grain was recrystallized. The multi-dome test was conducted for the confirmation of superplastic forming in 1mm and 1.5 mm of aluminum alloy sheets, and the strain rate sensitivity index was calculated. The result reveals that 5% of SiC composites have a maximum strain rate sensitivity at the rate of 0.45. The fracture surface analysis indicates that the superplastic deformation is mostly dominated by grain boundary sliding and dynamic recrystallization.

Keywords: Aluminium metal matrix composite; Al 7075; SiC; superplastic forming; polycrystalline

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Aluminium alloys find extensive utilization across diverse industries owing to their lightweight nature and exceptional resistance to corrosion. Nevertheless, there exists perpetual scope for enhancement in the realm of mechanical characteristics to cater to the escalating requisites various modern applications [1]. Aluminium alloy 7075 is recognized for its robustness and endurance against fatigue. To improve the mechanical characteristics of aluminum alloys is by integrating them with ceramic particles such as silicon carbide (SiC), titanium carbide TiC, aluminum oxide (Al₂O₃) and boron carbide (B₄C) etc. [2]. Through the incorporation of SiC particles into this alloy using the stir casting technique the composite material that manifests notably ameliorated mechanical properties. The stir casting methodology encompasses the fusion of the aluminium alloy followed by the introduction of SiC particles, subsequent to which the mixture is stirred to ensure an even dissemination of the reinforcing particles [3]. The introduction of SiC particles into aluminium alloy 7075 has been observed

to substantially boost the hardness, strength, and resistance to wear of the substance. This phenomenon is attributed to the elevated potency and hardness of SiC particles, which function as reinforcements within the composite material. The dispersion of these particles throughout the alloy matrix also serves to impede crack propagation, thereby further enhancing the material's tenacity. Alongside the enhanced mechanical properties, the aluminium alloy 7075-SiC composite material produced via the stir casting method also showcases commendable thermal conductivity and minimal thermal expansion, rendering it apt for applications requiring elevated temperatures [4]. The utilization of SiC particle-reinforced composite materials manufactured through the stir casting method presents a promising avenue for boosting the mechanical properties of aluminium alloys such as 7075. With escalated hardness, strength, and wear resistance, this composite material proves to be well-suited for applications where superior performance and dependability hold paramount significance [5].

A metal matrix composite is typically lightweight, has high strength and stiffness, is incredibly wear-resistant, and is relatively stable at high temperatures, which makes it suitable for structural applications [6]. Nevertheless, a major disadvantage of these materials is their poor formability, which has effectively prevented them from being considered for many potential applications [7]. Furthermore, these composites exhibit a high-strain rate occurrence of superplastic deformation in aluminum-based MMCs thus making these materials more appropriate for industrial applications [8]. Superplasticity is the capacity of specific materials to experience enormous extension at the best possible temperature and strain rate [9]. Under legitimate conditions, these materials can be stressed a few times to their unique length [10]. Several studies have concluded that the high strain rate superplasticity is a result of the fine-grained nature of these materials, while others suggest the existence of a liquid phase at grain boundaries and interfaces during superplastic formation at elevated temperatures enables sliding at these interfaces and grains [11].

The literature investigations shows that the enhanced mechanical qualities and thinning factor of AA7075/SiC composites may be used in the aerospace, automotive, marine, and military industries. The phenomenon of superplasticity has been thoroughly investigated in both the pure form of AA 7075 alloy and in its composite state with SiC particles [12]. The AA 7075 alloy demonstrates superplasticity under specified parameters of temperature and strain rate, with the most favorable elongations achieved at a temperature of 530 °C and a strain rate of 10^{-3}s^{-1} . The AA 7075 alloy, when coupled with SiC particles, exhibits exceptional superplasticity at high strain rates. It achieves elongations of around 315% when subjected to an initial strain rate of 5 s^{-1} at a temperature of 793K [13]. Moreover, the composite materials exhibit refined grain structures, which contribute to their exceptional superplastic characteristics. The findings emphasize the promise of AA 7075 and its composites for applications that demand superplastic behavior, demonstrating its adaptability and mechanical qualities under various testing situations [14]. According to the research, AA7075/SiC composites demonstrate their potential for demanding industrial applications with encouraging results in multi-dome tests.

MATERIALS AND METHODS

Stir Casting Method of Al7075-SiC Composites

The Al7075-SiC composite material is made in the stir casting process; the materials were prepared simultaneously in the stir caster and the muffle furnace, for the amalgamation of the materials. In the stir caster, 1 kg of aluminum is heated with 5 g of coverall flux to 800°C for 50 minutes. The impurity removal, the addition of 5 g of degasser, and holding

at 800°C for 5 minutes. Subsequently, 5 g of nucleant is added, and then reheating it into 800°C for an additional 5 minutes, and finally, the addition of another 5 g of coverall flux with composite in molten stage. The SiC powder is preheated in the muffle furnace, the SiC is concurrently heated to 1000°C to improve wettability and reduce thermal shocks. The preheated SiC is introduced into the molten Al7075 aluminum, resulting in an Al-SiC mixture that is mechanically stirred at 125 rpm to achieve uniform distribution of reinforcement particles. Simultaneously, die is preheated to 800°C, into which the stirred mixture is poured and permitted to solidify, yielding the Al7075-SiC composite material.

Thermo Mechanical Treatment for Hot Rolling of Synthesised Composites

Thermo mechanical treatment (TMT) consists of solution zing, averaging, and hot rolling of 7075 Aluminium alloy. The process of TMT results in the formation of a microstructure with a small and uniform grain size, measuring less than $10\mu\text{m}$. The AA7075 material was purchased as a sheet of $30 \times 30 \times 5\text{mm}$ in thickness [15]. After undergoing the hot-rolling process, the thickness of the sheet was reduced to 2mm using the technique by the following steps. The sequence is formulated to regulate the microstructure, distribution of precipitates, and grain structure of the material, with the aim of attaining particular mechanical qualities like as strength, ductility, and perhaps texture for anisotropic behavior. The method involves a comprehensive thermo-mechanical treatment.

Step 1: Solution Treatment; The temperature is 500 degrees Celsius with the timeframe: 60 minutes, State: Furnace temperature decreasing to 380°C This process entails raising the temperature of the material to a significant degree (500°C) in order to incorporate alloying materials into the aluminum matrix by dissolution. The gradual decrease in temperature of the furnace to 380°C enables the precise formation of certain phases by controlled precipitation.

Step 2: Over Aging; the process of aging beyond the typical or expected timeframe, the temperature reached throughout the process of 380°C and it lasted for a duration of 2.5 hours. The condition at the end of the process was furnace cooling to 190°C. This phase facilitates the proliferation of solid particles, which may lead to their enlargement. Prolonged exposure to this temperature and subsequent gradual cooling can result in a more resilient microstructure.

Step 3: Warm Rolling, the temperature is set at 180°C with the severity of 65-85% decrease in thickness. This phase entails subjecting the material to high temperatures in order to induce deformation. The substantial decrease in thickness (65-85%) results in a considerable amount of strain, which has the potential to impact the strength and texture of the material.

Step 4: Recrystallization, It is process involves the materials alteration the crystal structure at a temperature of 500°C for a duration of 30 minutes. The rapid cooling made by using the water for the purpose of this stage is to initiate recrystallization of the distorted structure resulting from the heated rolling process. Elevated temperatures facilitate the creation of fresh, unstrained grains. The water quench rapidly decreases the temperature of the material, which may result in the preservation of certain alloying components that are dissolved in the solution.

Step 5: Aging, the temperature is set at 180°C and the duration is 1 hour, the water quenching made. This final stage facilitates the regulated formation of strengthening phases by precipitation. The water quenching process at the conclusion of the treatment preserves the microstructure that was formed throughout this treatment.

The TMT process results in the formation of a microstructure with a small and uniform grain size of 35µm. The cast composites possess a thickness of 5 mm. Following the hot rolling procedure, the thickness of the specimens was decreased to 1mm and 1.5mm. Figure 1 displays the samples of the rolled specimens. Subsequently, it was let to cool in order to undergo natural aging. During the aging process, it was noted that Mg₂Si precipitates occur as finely scattered phases. These phases act as anchors within the matrix and hinder deformation, leading to a substantial enhancement in strength. The process of natural ageing results in an increase in the yield strength of a material. The sheets were stored for a

period of three days following the rolling procedure to allow for natural aging.

Biaxial Superplastic Forming

In the present investigation, hot-rolled composites were subjected to biaxial superplastic forming of multi-dome test experiments. This has been used to analyse the strain rate sensitivity index as shown in Figure 1. The setup has one split type furnace the temperature can be obtained up to 1000°C along with a temperature display unit and a temperature control unit; the required temperature can be set up in the furnace through the temperature control unit. The die sheet has three holes with 5 mm, 10 mm, and 15 mm and is placed at the centre of a split-type furnace and it is connected with steel pipes for blowing of air and connected with a compressor with a capacity of 7 bar. The specimen is fixed with the die sheet at the bottom of the Die, the top and bottom die were tightened without any leakage of air and fixed inside the furnace. Depending on the pressure, the control unit controls the pressure. The required temperature and pressure through the die the components were formed. After finishing the multi-dome test, the components were taken from the die and it was measured through a venire height gauge. The dome height of 15 mm, 10 mm and 5 mm was observed. To measure the thickness anvil setup is available; the formed component was placed on the top of the anvil and the thickness was measured. The bottom of the anvil end will be at the bottom of the component and the same place top the dial gauge was placed, through this method, the thickness of the formed component is measured.



Figure 1. After recrystallization (Hot rolled) specimen.

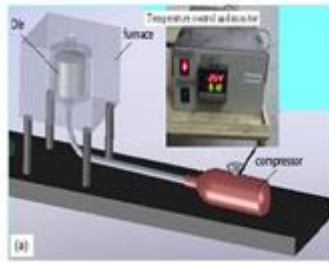


Figure (2-a). Schematic view of multi dome test.



Figure (2-d). Temperature Control unit.



Figure (2-b). Half section view of split type furnace.



Figure (2-e). Measuring instrument Zero setting.



Figure (2-c). Setup with Split type furnace.



Figure (2-f). Measuring instrument and setup views.

RESULTS AND DISCUSSION

Microstructure of Synthesised Composites

In order to acquire a micrograph of the composite, samples were taken from the cast and rolled composite. The samples were affixed using a heated Bakelite mounting method, the samples were produced according to the specifications set by ASTM (2004). The samples were sequentially polished using emery sheets of 1/0, 2/0, 3/0, and 4/0 grades, followed

by a wet polishing disc using Alumina powder as the polishing medium.

The samples were treated with Keller's reagent and images were captured at a 100X magnification [16, 17]. The Biovis program was used to measure the average grain size in the rolled specimens. The mean grain size of the cast specimen was 75µm, whereas the cast composites of the rolled sheets had a mean grain size of 30µm. It was noticed that the agglomeration of SiC particles was seen in Figure 3.

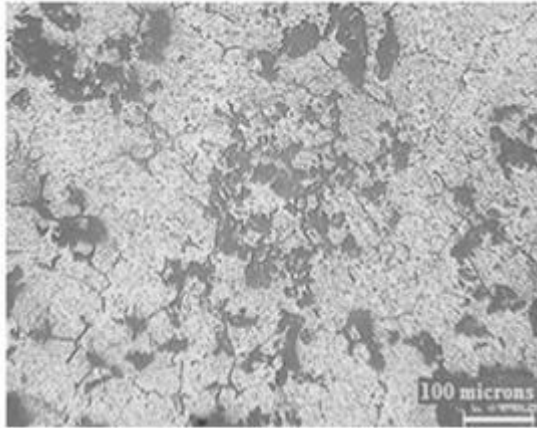


Figure (3-a). Microstructure of cast specimen before rolling.

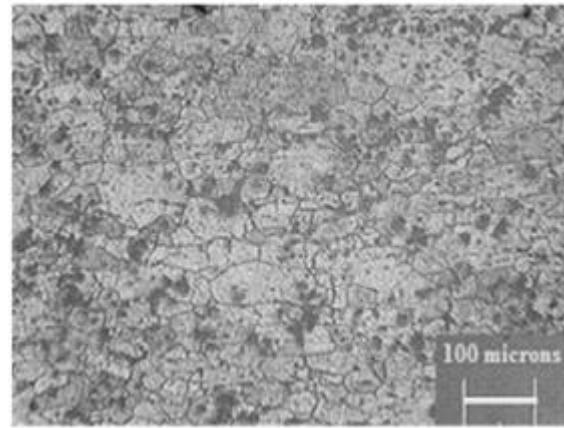


Figure (3-b). Microstructure of cast specimen after rolling.

Multi Dome Analysis of AL7075 Alloy

In the present investigations, Al7075 alloys of 1.5mm thickness sheet were tested to find the strain rate sensitivity index [18].

By using the above parameters, the multi-dome test was performed. It was observed that at 0.5 MPa pressure and the temperature of 450° C and for 60 seconds the components were formed as shown in Figure 4 (a-b).

Table 1. Al7057 / SiC Strain rate sensitivity index for 1.5mm thickness sheet.

Forming parameters		Diameter of a hole, mm	Bulge Height, mm	Effective flow stress, σ Mpa	Strain ϵ	Strain rate sensitivity Index, m
1	Initial thickness (1.5 mm)	15	3.617	1.968	0.209	0.6791
2	Pressure (0.5 MPa)					
3	Forming time (60 sec)	10	1.881	1.443	0.132	

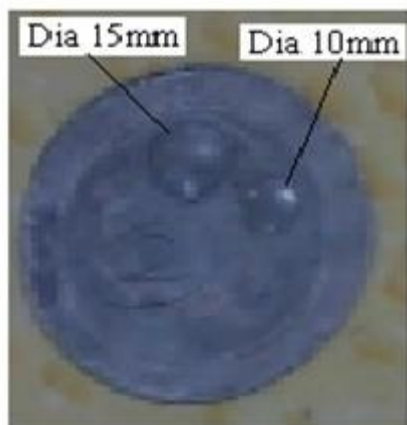


Figure 4 (a). Multi Dome Analysis of AL7075 Alloy of formed component.



Figure 4 (b). Multi Dome Analysis of AL7075 Alloy of test sample.

Table 2. Al7057 / SiC Strain rate sensitivity index for 1mm thickness sheet.

Forming parameters		Diameter of the hole, mm	Bulge Height, mm	Effective flow stress, Mpa	Strain 'e'	Strain Rate sensitivity Index, 'm'
1	Initial thickness (1 mm)	15	1.525	1.9999	0.0405	0.301
2	Pressure (0.3 MPa)					
3	Forming time (60s)	10	0.31	4.0633	0.0038	



Figure 5. Multi dome of Al-SiC composites.

The height and flow of stress were tabled as shown in table 1. In the 15 mm diameter hole, a dome height of 3.6 mm was formed and 1.88 mm was formed on the 10 mm hole. The measurements of the dome height, diameter of the hole, flow stress, and strain allowed for the determination of the strain rate sensitivity index, which was found to be 0.6.

Multi Dome Analysis of AL7075/SiC Composites of Varying Weight Proportions

Multi-dome experiments for the processing of Al7075/SiC aluminium alloy composites with different process parameters were investigated. The dome with a height of 6 mm was achieved using a die with a diameter of 15 mm and a pressure of 5 bar [19]. Al/SiC composites 1.0mm thickness sheet were tested to find the strain-rate sensitivity index shown in Table 2.

From table 2, it was found that the strain rate sensitivity index obtained at the same temperature

and the pressure was 0.3 Mpa for 60 sec. The strain rate sensitivity index was obtained at 0.3. The reduction in pressure reduces the formability characteristics.

Thinning Factor of Al7057 Alloy

Table 3 lists the thinning factor of the superplastic formed components thinning factor values for Al 7075 with SiC in various weight percentages. The thinning factors values were measured at various locations of the Dome at different positions for 1mm and 1.5mm thickness sheets [20-22]. Table 3 lists the thinning factor of Al -SiC, 2mm thickness sheet, thickness variation at different positions. From the outcome of the tested results the higher thinning factor were occurs in the 15 mm sheets, in all the SiC reinforcements, there is need to find the optimum point of prediction and develop the mathematical model to find appropriate level of change of reinforcement and thickness these requirement leads to use of RSM analysis [23-25].

Table 3. Thinning factor value of Al7075 with SiC.

Sample	SiC	Sheet Thickness	Thinning at various locations of the Dome			Average Thinning (mm)	Thinning Factor
	%	mm	Point 1	Point 2	Point 3		
Sample A	5	1	1.73	1.89	1.862	1.8273	0.9567
Sample B	5	1.5	1.721	1.695	1.74	1.7187	0.9862
Sample C	7.5	1	1.765	1.828	1.892	1.8283	0.9654
Sample D	7.5	1.5	1.721	1.695	1.74	1.7187	0.9842
Sample E	10	1	1.842	1.891	1.905	1.880	0.9824
Sample F	10	1.5	1.889	1.922	1.938	1.92	0.9875

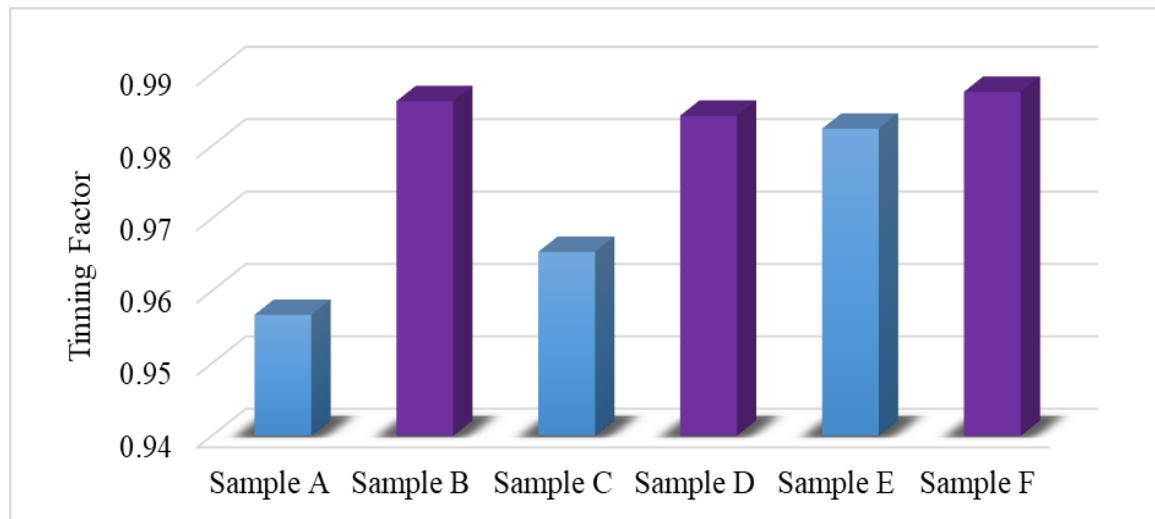


Figure 6. Thinning factor value for Al7075 with SiC in various weight percentages.

RESPONSE SURFACE METHODOLOGY (RSM)

In this research work, the superplastic formation is components thinning factor values for Al 7075 with SiC in various weight percentages. The thinning factors values were measured at various locations of the Dome at different positions for 1mm and 1.5mm thickness sheets. The Design-Expert 13 software is utilized for conducting response surface methodology (RSM)

analysis. Based on the analysis of the experimental findings, it is recommended that the quadratic formation analysis is suitable for the present study. This recommendation is supported by the fit summary value, where the R² value for the quadratic model is 0.95.

Choose the polynomial with the maximum degree where the extra terms have a meaningful impact and the model is not affected by aliasing.

Table 4. RSM analysis Fit Summary.

Source	Sequential p-value	Adjusted R ²	Predicted R ²	
Linear	0.0899	0.6657	-0.1095	
2FI	0.0502	0.9509	0.7347	Aliased
Quadratic	0.1382	0.9954	0.9508	Suggested

Table 5. Sequential Model Sum of Squares.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	5.73	1	5.73			
Linear vs Mean	0.0007	2	0.0003	5.98	0.0899	
2FI vs Linear	0.0001	1	0.0001	18.42	0.0502	Aliased
Quadratic vs 2FI	0.0000	1	0.0000	20.55	0.1382	Suggested
Residual	7.500E-07	1	7.500E-07			
Total	5.73	6	0.9548			

Table 6. Model Summary Statistics.

Source	Std. Dev.	R ²	Adjusted R ²	Predicted R ²	PRESS	
Linear	0.0074	0.7994	0.6657	-0.1095	0.0009	
2FI	0.0028	0.9803	0.9509	0.7347	0.0002	Suggested
Quadratic	0.0009	0.9991	0.9954	0.9508	0.0000	Suggested

Table 7. ANOVA for Response of Tinning Factor.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0008	4	0.0002	273.92	0.0453	significant
A-SiC	0.0002	1	0.0002	243.00	0.0408	
B-Sheet Thickenss	0.0005	1	0.0005	633.68	0.0253	
AB	0.0001	1	0.0001	198.45	0.0451	
A ²	0.0000	1	0.0000	20.55	0.1382	
B ²	0.0000	0				
Residual	7.500E-07	1	7.500E-07			
Cor Total	0.0008	5				

Table 6 presents the Model Summary Statistics, with the primary objective of the model being to maximize the Adjusted R² and the Predicted R². It confirms the suggestion of the quadratic equation formation of mathematical model development.

Table 7 displays the ANOVA results for the Tinning Factor's Response. In this model, the F-value is 273.92, indicating its importance and suggesting that there is only a 4.53% probability that such a significant F-value could be attributed to random variation. A P-value below 0.0500 shows that the model terms are statistically significant. Regarding this matter, it is worth noting that A, B, and AB are highly important model terms. The values exceed 0.1000, indicating that the model terms lack significance. The Predicted R² value of 0.9508

is a realistic estimate, considering the Adjusted R² value of 0.9954. The discrepancy between the two values is smaller than 0.2. The adequacy accuracy quantifies the ratio of the signal to the noise. A ratio over 4 is preferable. The ratio of 39.592 suggests that the signal is sufficient. This paradigm is applicable for navigating the design space.

Final Equation in Terms of Coded Factors

$$\text{Tinning Factor} = + 0.9748 + (0.0068*A) + (0.0089*B) - (0.0061*AB) + (0.0034*A^2) + B^2$$

The equation, expressed in terms of coded components, allows for making precise predictions regarding the reaction for certain quantities of

each element. The default coding assigns a value of +1 to represent high levels of the components, while a value of -1 is used to represent low levels. The encoded equation is valuable for determining the relative influence of the components by comparing the coefficients of the factors.

From figure 7 it is observed that the maximum thinning factor observed at 1.4 mm thickness of the sheet with 9.8 weight percentage of SiC with the aluminium alloy 7075. The 3D surface plot Thining

factor is drawn from sheet thickness with Sic in various percentages. Figure 8 shows the 3D surface plot Thining factor, it is observed that 9.8 weight percentage of SiC with the aluminium alloy 7075 in 1.4mm thickness of the sheet is an optimized combination. By optimizing material and process parameters for SiC-reinforced aluminum components used in various parts in manufacturing sectors, energy and thermal storage systems, chemometric optimization using RSM can improve efficiency and stability of energy systems. [26-28].

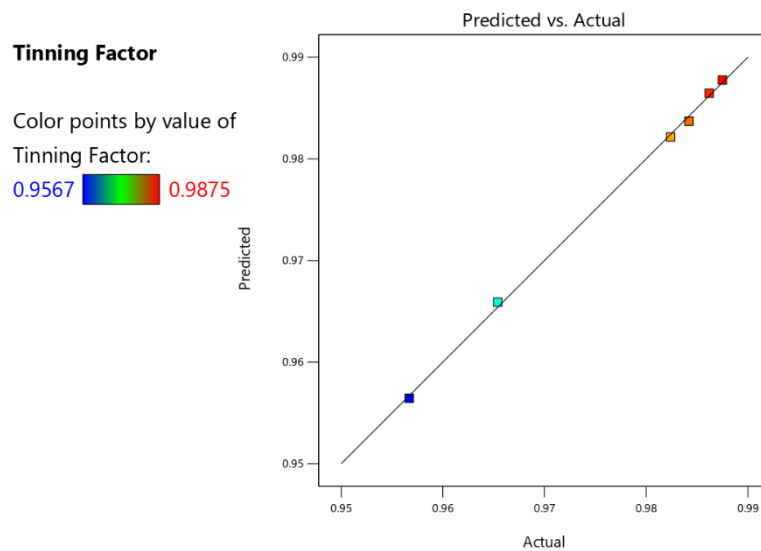


Figure 7. Contour plot for thinning factor.

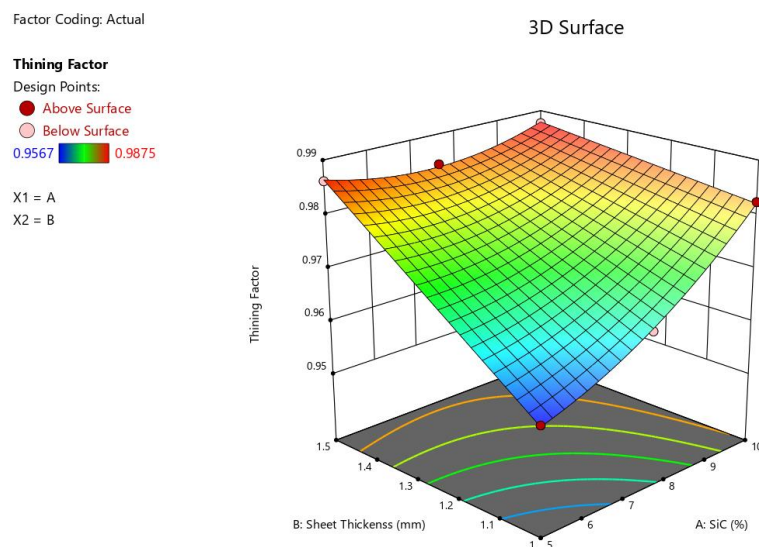


Figure 8. 3D surface plot Thining factor.

CONCLUSION

The multi-dome tests were conducted with different pressures; a Maximum 0.67 was obtained at the pressure of 5 bar. An increase in pressure increases the dome height; Maximum dome height of 5.8 mm was obtained at the pressure of 5 bar. If the pressure increased to 6 bar the component failed. The thinning factor of the composites was measured; an increase in the percentage of SiC particles increases the thinning factor. After the superplastic forming, the hardness was measured it was reduced, and because of the annealing process, the superplastic forming processes keep the component at a high temperature, and cooling slowly leads to the hardness reduction. In the RSM analysis of the research work made in Design-Expert 13, the fitness statics of the model was suggested quadratic equation of the mathematical model and coded tinning factor quadratic equation found. The RSM analysis suggested the point of optimum value is 1.4mm thickness with a 5.5 Sic percentage is the best combination for the good thinning factor of 0.98.

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