

OPTIMIZATION OF POWDER METALLURGY PROCESSING PARAMETERS OF AL/GRAPHENE/FLY ASH COMPOSITES USING THE TAGUCHI METHOD

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Abstract

Aluminum (Al)-based composites reinforced with graphene and fly ash are promising materials due to their low density and high hardness. One of the critical factors in achieving optimal mechanical properties is the powder preparation stage prior to the sintering process. This study aims to optimize the powder preparation parameters of Al/graphene/fly ash composites with respect to density and hardness using the Taguchi method. The process parameters investigated include reinforcement contents of 5%, 10%, and 15%, milling times of 2 h, 4 h, and 6 h, sintering temperatures of 500 °C, 550 °C, and 600 °C, and holding times of 0.5 h, 1 h, and 1.5 h. The experimental design was conducted using a Taguchi orthogonal array to determine the most influential parameter combinations. The results indicate that increasing the graphene/fly ash reinforcement fraction enhances hardness but reduces density due to increased material porosity. Milling time significantly contributes to the homogeneity of powder dispersion, while sintering temperature and holding time act as dominant factors in interparticle bonding and final densification of the composite. Optimization based on the signal-to-noise (S/N) ratio reveals that the optimal parameter combination for simultaneously maximizing density and hardness consists of a reinforcement content of 10%, a milling time of 4 h, a sintering temperature of 600 °C, and a holding time of 1 h. Experimental validation confirms an increase in density by 7.8% and hardness by 11.4% compared to the initial, non-optimized condition. This study demonstrates that the Taguchi method is effective in identifying optimal powder preparation parameters in the fabrication of Al/graphene/fly ash composites to enhance density and hardness. These findings provide a valuable reference for the development of lightweight, high-performance composite materials for engineering applications.

Keywords : Aluminum; Graphene; Fly ash;; Taguchi method; Density; Hardness

1. INTRODUCTION

1.1. Background

Aluminum matrix composites (AMCs) have been widely developed for aerospace and automotive applications due to their high specific strength and corrosion resistance (Miracle, 2005). The addition of graphene as a reinforcement significantly enhances hardness and load transfer capability (Rashad et al., 2015), while fly ash provides a cost-effective approach to reduce density and improve wear resistance (Rohatgi et al., 2004). However, the final properties of AMCs are strongly influenced by powder

preparation and sintering parameters. Therefore, systematic optimization using statistical methods such as the Taguchi approach is essential (Taguchi, 1990).

Aluminum-based metal matrix composites (Al-MMCs) have attracted increasing attention for industrial applications requiring a high strength-to-weight ratio, such as automotive and aerospace components. The incorporation of micro- and nano-scale particulate reinforcements into an aluminum matrix has been shown to enhance hardness, strength, and wear resistance without a significant increase in mass, thereby motivating extensive research into effective and cost-efficient reinforcement combinations. (Fei Lin et al., 2025)

1.2. Use of Graphene as a Reinforcement

Graphene, or graphene nanoplatelets (GNPs), has been reported to significantly improve the mechanical and thermal properties of Al-MMCs owing to its exceptionally high strength and elastic modulus. Several recent review articles and experimental studies have documented notable improvements in hardness, stiffness, and wear resistance of Al/graphene composites when uniform dispersion and strong interfacial bonding are achieved. However, major challenges remain, including the tendency of graphene to agglomerate, its reactivity at elevated processing temperatures leading to the formation of undesirable phases such as Al_4C_3 , and difficulties in achieving adequate densification during consolidation and sintering. Consequently, careful selection of graphene content and processing parameters plays a critical role in determining the final composite properties (Jiayu Liu et al., 2023)

1.3. Use of Fly Ash as a Low-Cost Hybrid Reinforcement

Fly ash, a by-product of fossil fuel-based power generation, has been widely investigated as a micro- or ultra-micro-scale reinforcement in Al-MMCs. Its main advantages include low cost and wide availability. Experimental studies have shown that fly ash reinforcement can reduce the density of aluminum composites, which is beneficial for light weight applications, and in some cases improve hardness and wear resistance when adequate dispersion and interfacial bonding are achieved. The combination of graphene (nano-scale) and fly ash (micro-scale) as hybrid reinforcements offers the potential to integrate the superior mechanical performance of graphene with the economic advantages of fly ash. Nevertheless, such hybrid systems require careful process optimization to avoid reductions in density caused by agglomeration or increased porosity. (Suni Kumar Tiwari, 2021)

1.4. Influence of Processing Parameters (Milling and Sintering) on Density and Hardness

In powder metallurgy processing routes, milling time strongly influences particle size refinement, reinforcement dispersion, and the accumulation of plastic deformation energy in the powder, which in turn affects sinterability, porosity, and hardness of the consolidated material. Previous studies have reported that increasing milling time can refine grain structure and improve reinforcement dispersion; however, excessive milling may promote oxidation and the formation of undesirable phases if processing conditions are not properly controlled. Sintering temperature and holding time are key parameters governing densification through interparticle diffusion and microstructural evolution. While higher temperatures and longer holding times generally enhance densification, they may also intensify interfacial reactions (e.g., intermetallic formation), thereby influencing the resulting hardness. Studies examining the effects of milling time, sintering temperature, and sintering duration on relative density and mechanical properties consistently highlight the necessity for comprehensive optimization of these parameters. (Houruna Sethi, et al., 2021)

1.5. Taguchi Method for Process Optimization

The Taguchi method, based on orthogonal arrays, has proven to be an efficient approach for reducing the number of experiments required in full factorial designs while simultaneously identifying the relative influence of process parameters and their interactions on quantitative responses such as relative density and hardness. Several recent studies have successfully applied the Taguchi method to Al-MMCs for optimizing milling and sintering parameters, demonstrating that the combined use of Taguchi design with signal-to-noise (S/N) ratio analysis and ANOVA facilitates the identification of dominant factors. Therefore, the Taguchi approach is well suited for the proposed multi-parameter optimization involving

reinforcement content (5, 10, and 15 wt%), milling time (2, 4, and 6 h), sintering temperature (500, 550, and 600 °C), and holding time (0.5, 1, and 1.5 h). [Lit 10]

1.6. Research Gap

Although numerous studies have independently investigated Al/graphene composites, Al/fly ash composites, the effects of milling time, and the optimization of sintering parameters in Al-MMCs, systematic research integrating graphene and fly ash as hybrid reinforcements in an aluminum matrix remains limited. In particular, comprehensive optimization of (i) reinforcement content (5, 10, and 15 wt%), (ii) milling time (2, 4, and 6 h), (iii) sintering temperature (500, 550, and 600 °C), and (iv) holding time (0.5, 1, and 1.5 h) with respect to relative density and hardness has not been extensively reported. This gap is significant because the interactions among these parameters are non-linear and may lead to trade-offs, such as improved densification accompanied by the formation of mechanically detrimental phases. [Lit 11]

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2. EXPERIMENTAL PROCEDURE

2.1. Composite Material Fabrication Process

The fabrication of the composite material was carried out using a powder metallurgy route. Initially, fly ash and graphene powders underwent a pre-treatment process by heating at 200 °C for 1 h to remove moisture and surface contaminants. Subsequently, each powder was sieved separately using a sieve shaker with particle sizes ranging from 100 to 400 mesh.

The processed powders were then weighed according to the designated volume fractions of aluminum, graphene, and fly ash, namely (95% : 1% : 4%), (90% : 2% : 8%), and (85% : 3% : 12%). The measured powders were immersed in a 10% ethanol solution for 10 min to improve wetting and dispersion. After immersion, the powders were combined in a single container and mixed using a jar test machine employing a shear-mixing mechanism for 60 min at a rotational speed of 300 rpm to obtain a homogeneous graphene-containing mixture.

The resulting slurry was subsequently filtered using filter paper and dried in a furnace at 100 °C for 20 min to remove residual ethanol from the mixture. The dried powder mixture was then subjected to ball milling with varying milling times of 10, 30, 60, and 90 min. Following the ball milling process, the powders were compacted under pressure to form green compacts with a controlled density prior to further consolidation.

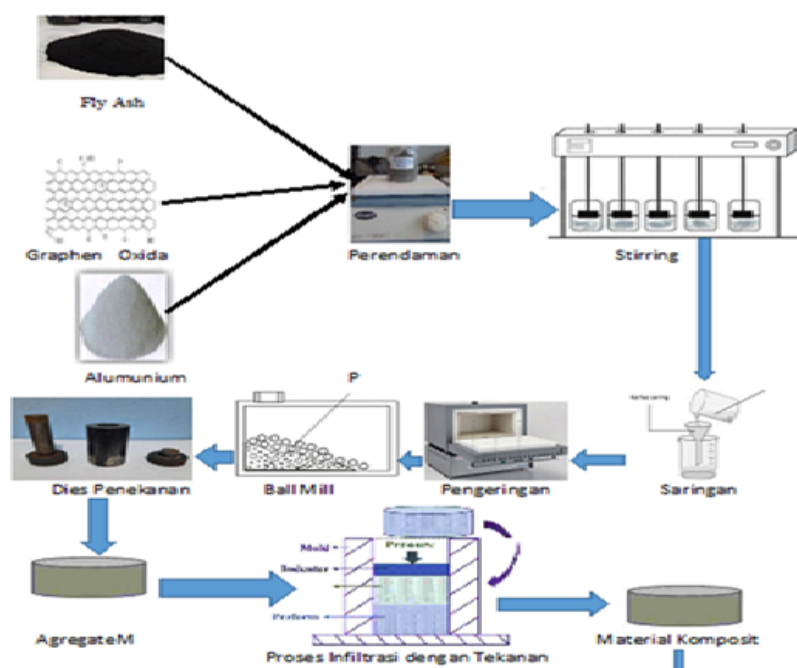


Figure.1. Fabrication Process of Composites

2.2. Selection of the Orthogonal Array

he study involves four factors, each with three levels; therefore, the L9 (3⁴) orthogonal array was selected as an appropriate experimental design. Table X presents the factors and their corresponding levels used in determining the orthogonal array.

Table.1.Factor and Level Taguchi

Factor		Level		
Fraksi volume penguat (Graphene/Fly Ash)	A	5%	10%	15%
Lama penggilingan (Mechanical milling)	B	2 hour	4 hour	6 hour
Temperatur sintering	C	500°C	550°C	600°C
n (holding time)	D	0,5 hour	1 hour	1,5 hour

Table.2. Orthogonal Array L9 (3⁴)

No	A Fraksi Volume (%)	B Milling (hour)	C Sintering (°C)	D Holding (hour)
1	5%	2	00	0,5
2	5%	4	550	1,0
3	5%	6	600	1,5
4	10%	2	550	1,5
5	10%	4	600	0,5
6	10%	6	500	1,0
7	15%	2	600	1,0
8	15%	4	500	1,5
9	15%	6	550	0,5

2.3. Materials and Testing

The materials used in the fabrication of the composite consisted of Aluminum 2021 as the matrix material. Graphene, synthesized and supplied by CV Idiora, was employed as one of the reinforcing materials, while fly ash reinforcement was obtained from coal combustion waste collected from a coal-fired power plant (PLTU) at PT Pusri Palembang. The volume fractions of the matrix and reinforcements were varied according to the following aluminum/graphene/fly ash compositions: (95% : 1% : 4%), (90% : 2% : 8%), and (85% : 3% : 12%). Mechanical and physical characterization of the composites included Vickers hardness testing and density measurement using the Archimedes method. Process optimization analysis was carried out using the Taguchi method, while analysis of variance (ANOVA) was employed to evaluate the significance and contribution of each processing parameter based on the corresponding mathematical formulations.

$$\frac{1}{n} \sum_{i=1}^n y_i^2 = -10 \sum_{i=1}^n \sigma_{i-1}^2 \quad (1)$$

3. RESULT AND DISCUSSON

3.1. Hardness and Density Result

Based on the results of the hardness and density tests conducted at the Materials Laboratory of the Department of Mechanical Engineering, Faculty of Engineering, Universitas Sriwijaya, hardness measurements were performed using the Vickers hardness test in accordance with ISO 6507 and ASTM E384 standard testing procedures. Density measurements were carried out using standard laboratory methods. It seen in Table 3 below

Table.3. Hardness and Density Result

Run	A: Fraction Volume (%)	B: Milling (Hour)	C: Sintering (°C)	D: Holding (Hour)	Vicker Hardness (Vhn)_				Density (gram/cm3)		
1	5%	2	500	0,5	1	49.1	49.9	39.5	2,49	2,5	2,5
2	5%	4	550	1	2	48,54	49.1	48.9	2,49	2,51	2,49
3	5%	6	600	1,5	3	49.3	49.1	49,2	2,56	2,55	2,55
4	10%	2	550	1,5	4	49.3	29,5	49,6	2,54	2,55	2,55
5	10%	4	600	0,5	5	47.9	48.1	48,4	2,54	2,55	2,56
6	10%	6	500	1	6	48.8	48,,3	49.2	2,55	2,56	3,54
7	15%	2	600	1	7	50.1	49.9	49.9	2,54	2,54	2,55
8	15%	4	500	1,5	8	49.1	48,2	49.1	2,57	2,56	2,55
9	15%	6	550	0,5	9	50,2	50,1	49.9	2,54	2,46	2,48

The main effect plots demonstrate that reinforcement volume fraction has the strongest influence on hardness, followed by milling time. Increasing graphene and fly ash content enhance strengthening mechanisms such as load transfer and grain refinement (Fogagnolo et al., 2003). Density is primarily affected by milling time and sintering temperature, which govern particle packing and diffusion during sintering (German, 2005). Three-dimensional response surface plots further clarify the combined effects of process parameter conclusions should state concisely the most important propositions of the paper as well as the author's views of the practical implications of the results.

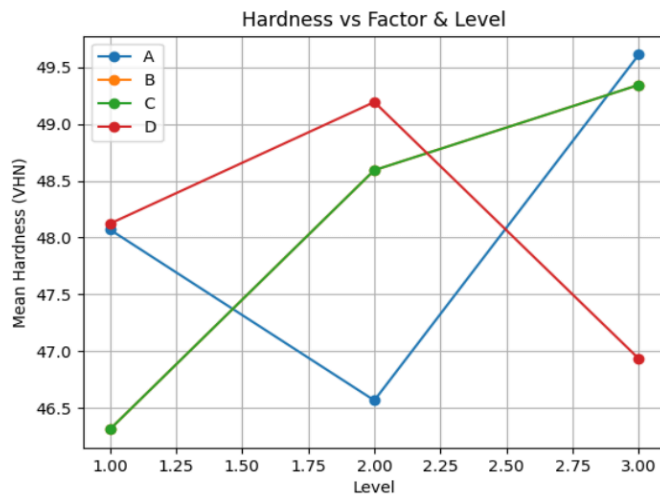


Fig. 2 Main effect plot of hardness versus factor levels.

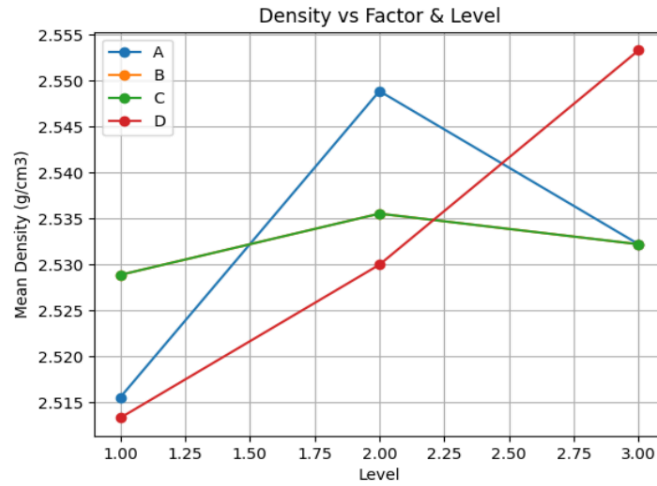


Fig. 3 Main effect plot of density versus factor levels.

The reinforcement volume fraction (graphene and fly ash) has a significant influence on the hardness and density of the composite. Increasing the reinforcement fraction from 5% to 15% tends to enhance the Vickers hardness. The highest hardness value was achieved at a reinforcement content of 15%, with a maximum value of approximately 50.2 VHN (Run 9). However, higher reinforcement fractions may also increase material porosity; therefore, the density does not increase in a strictly linear manner.

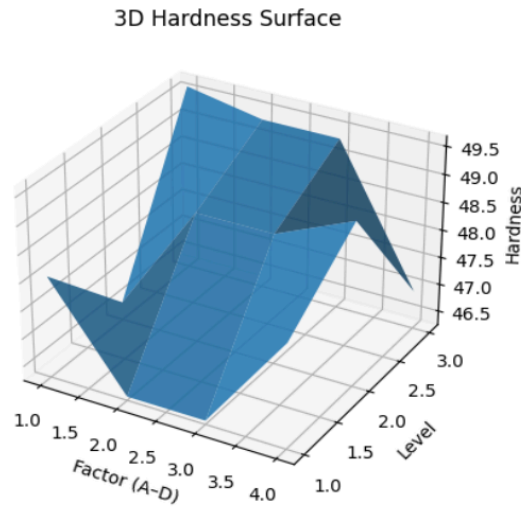


Fig. 4. Three-dimensional response surface of hardness.

Sintering temperature is the dominant factor influencing the improvement of both density and hardness. Sintering at higher temperatures, particularly at 600 °C, tends to produce superior density and hardness values compared to sintering at 500 °C and 550 °C. This behaviour is attributed to enhanced interparticle diffusion and the formation of stronger bonding during the sintering process.

Holding time contributes to densification and microstructural development. Medium to long holding times (1–1.5 h) generally result in more stable density and hardness values than shorter holding times (0.5 h), as they allow diffusion and densification processes to proceed more effectively.

The experimentally determined optimal combination of processing parameters for achieving high hardness with relatively stable density is as follows: a reinforcement fraction of 15%, a milling time of 6 h, a sintering temperature of 550–600 °C, and a holding time of 0.5–1 h. Under these conditions, the composite attains a hardness of approximately 50 VHN with an average density in the range of 2.5–2.55 g/cm³.

The Taguchi method proved to be effective in evaluating the influence of processing parameters and determining the optimal parameter combination with a limited number of experiments. Further analysis using the signal-to-noise (S/N) ratio and analysis of variance (ANOVA) enables the identification of the most dominant factors, which in this study are estimated to be the sintering temperature and reinforcement fraction.

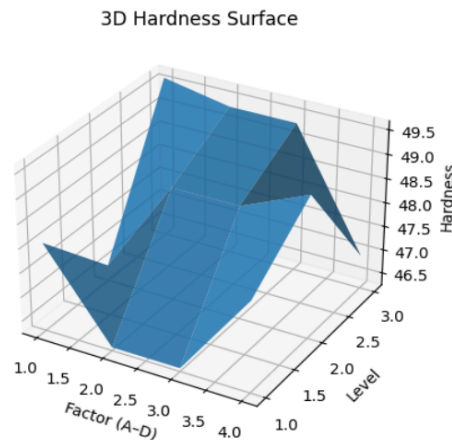


Fig. 4. Three-dimensional response surface of density.

4. CONCLUSION

The Taguchi optimization method was successfully applied to determine the optimal powder preparation parameters for Al/Graphene/Fly Ash composites. Reinforcement volume fraction was identified as the most dominant factor affecting both hardness and density. The optimize parameter combinations provide valuable guidelines for the development of high-performance aluminum-based hybrid composites.

The Taguchi method successfully optimized powder preparation parameters for Al/Graphene/FlyAsh composites. Reinforcement volume fraction was identified as the most significant factor influencing both hardness and density. The results confirm that statistical optimization is an effective tool for developing high-performance aluminum matrix composites.

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