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# Effect of compaction pressure on the structure and mechanical properties of functionally graded aluminium-glass microsphere (FGAGM) foams produced by powder metallurgy process

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**Abstract**

Functionally graded aluminium-glass microsphere (FGAGM) foams were successfully fabricated via powder metallurgy. The influence of compaction pressure on the structural and mechanical properties of the foams was systematically investigated. The study observed that increasing compaction pressure enhanced the green (pre-sintering) density of FGAGM foam, while post-sintering densities exhibited a decreasing trend with higher compaction pressures. The mechanical examination indicated that mechanical properties improved significantly with higher compaction pressures. While higher compaction pressures generally improve mechanical properties, the study identified that excessive pressures could lead to the fracture of GM particles, introducing defects that compromise the foam's structural integrity. Therefore, determining an optimal compaction pressure is crucial to maximize mechanical performance while preserving the integrity of the composite constituents.

## 1. Introduction

Functionally graded materials (FGMs) represent a paradigm shift in materials science, enabling materials design with spatially varying properties tailored to specific engineering applications, which lead to improved mechanical performance and durability. These advanced materials, often denoted as FGMs, are characterized by a gradual and continuous change in composition and microstructure over their volume, resulting in a corresponding gradient in material properties such as elastic modulus, thermal conductivity, and coefficient of thermal expansion [1]. FGMs are particularly useful in aerospace, automotive, and structural engineering applications due to their superior damage tolerance, high energy absorption, and improved stiffness [2,3]. Among FGMs, metal matrix syntactic foams (MMSFs) have been gaining interest as a lightweight material with a trade-off of high strength, thermal insulation, and acoustic damping performance [4]. The development of FGMs was spurred by the need for materials that could withstand extreme thermal gradients in aerospace applications, such as in thermal barrier coatings for rocket engines [5].

A pivotal moment in the history of FGMs was their introduction in 1984 by a group of Japanese scientists [6]. Early research focused on ceramic-metal composites, where the ceramic phase provided high-temperature resistance and the metallic phase offered improved toughness and resistance to thermal shock [7]. Current research indicates that the graded distribution of reinforcement materials, *i.e.,* glass microspheres (GM), is accountable for improved compressive strength as well as energy absorption capacity, making these foams suitable for structural applications [8]. GMs offer low density, good thermal stability, and are commercially available at low cost. Additionally, their spherical geometry enhances isotropic reinforcement and promotes uniform stress distribution in the Al matrix. Several studies [9-12] have demonstrated the effectiveness of GM in syntactic foam systems, which supports their selection [12] in the present study. Functionally graded aluminium-glass microsphere (FGAGM) foam is a promising development in this field, offering an optimum compromise between weight reduction and mechanical efficacy. The addition of GM to an aluminium (Al) matrix results in a significant reduction in density with adequate mechanical strength and impact resistance [13].

Powder metallurgy (PM) has been determined to be a useful technique for the production of FGMs, due to the suitability for achieving controlled porosity, functional gradation, and cost-effective fabrication of metal matrix foams, especially in layered or composite configurations [14]. It enables precise control over composition and microstructural details with little material loss. In addition, compaction techniques and sintering technology have been enhanced, making it possible to produce high-performance FGAGM foams with better porosity and mechanical properties [8]. Compared to spark plasma sintering (SPS), PM offers scalability and better control over porosity,

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which are crucial for foam production. This comparison is now supported by recent literature [15]. The gradual composition change in FGMs helps in minimizing issues such as stress concentration and premature failure, typically encountered in conventional composite materials [16].

Although promising, further research is necessary to optimize the production process, gain more uniform distribution of the reinforcement materials, and enhance the mechanical behavior of FGAGM foams for practical uses. The present research aims to develop FGAGM foams using the powder metallurgy process. The study focuses on investigating the effects of different compaction pressures on the performance of the foam described as compressive strength, hardness and energy absorption.

## 2. Experimental

Pure Al powder (Ecka Granules, 99.9% purity) was combined with GM particles (Q-CEL) at four distinct volume fractions to fabricate four-layered density-gradient syntactic foams. The GMs used in this study are hollow. This feature contributes significantly to the reduced density and improved energy absorption capability of the composite foams. To produce a composite sample, a constant Al-GM mass of 2.5 g per layer was maintained throughout the preparation, with various volume fractions of GM, as detailed in Table 1. The prepared mixtures were sequentially stacked within a 22 mm diameter tool steel die, which was lubricated with magnesium stearate suspended in acetone. To ensure a smooth surface and maintain horizontal uniformity across each layer, the die and its contents were gently vibrated after the addition of each layer. Subsequently, the mixtures underwent uniaxial cold compaction at pressures of 300 psi, 400 psi, and 500 psi. A two-step sintering process was employed under a nitrogen atmosphere, as illustrated in Figure 1. This process involved initial preheating of the samples to 400°C for 1 h, followed by heating to a target sintering temperature of 600°C for 2 h. Gradual cooling to room temperature within the furnace was then performed. This controlled sintering approach was implemented to prevent thermal shock, which could otherwise lead to crack formation and compromise the structural integrity of the specimens.

The relative density ($\rho^*$) of FGAGM foam samples is calculated as shown in Equation (1).

$$\rho^* = \left(\frac{\rho_a}{\rho_t}\right) \times 100 \tag{1}$$

Where $\rho_a$ and $\rho_t$ are the actual and theoretical densities of samples, respectively. The actual density of foam sample was measured using Archimedes' principle with water as the immersion fluid. The theoretical density of the FGAGM sample can be calculated using the basic rule of mixtures, based on the volume fraction of Al and GM in each layer. The different layer compositions were considered independently and weighted accordingly in the final theoretical density computation. In this study, the densities of Al and GM, reported to be 2.69 $g\cdot cm^{-3}$ and 0.11 $g\cdot cm^{-3}$, respectively, are used to find the theoretical density of 2.66 $g\cdot cm^{-3}$ [17,18].

To characterize the foam microstructure, samples were bisected along their mid-plane using a high-precision cutting machine, followed by standard metallographic preparation for Al samples. Microstructural examination of both the raw materials and the foam samples was conducted using a JEOL JSM-IT300 Scanning Electron Microscope (SEM), equipped with Energy Dispersive X-ray Spectroscopy (EDS) for phase identification. The particle size and distribution of both Al and GM were analyzed using a MALVERN Mastersizer 3000 laser particle size distribution analyzer. Compression tests were performed using a Shimadzu AG-X Plus universal mechanical testing machine. Foam samples were deformed to approximately 80% strain at a constant crosshead speed of $5 \times 10^{-5}$ $m\cdot s^{-1}$. For each compaction pressure condition, three samples with comparable GM compositions were tested. The energy absorption capacity ($E$) was calculated as the area under the stress-strain curve, as defined by Equation (2).

$$E = \int_0^{\varepsilon} \sigma d\varepsilon \tag{2}$$

Where σ and ε are stress and strain, respectively. The HM-200 Mitutoyo microhardness tester, with the applied load of 100 kgf and holding time of 15 s, was used to determine the microhardness of foam samples.

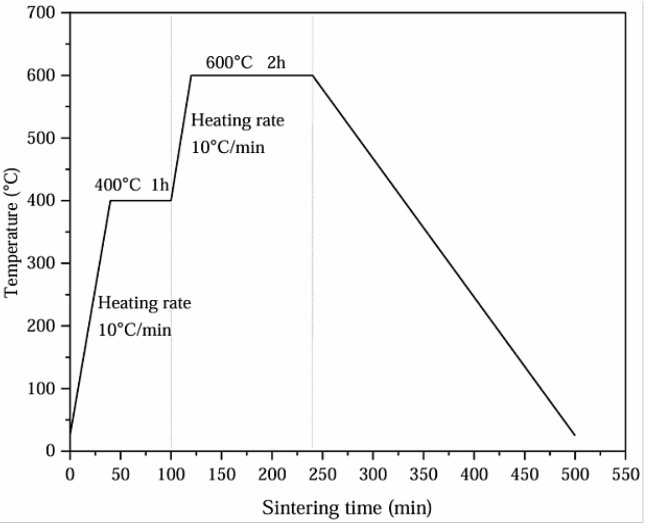

**Figure 1** Sintering profile of FGAGM foams

**Table 1.** Total mass distribution of powder mixture in each layer.

| Sample | Vol% of Al | Vol% of GM | Mass of Al | Mass of GM |
|---|---|---|---|---|
| 4th layer | 50 | 50 | 2.4021 | 0.0979 |
| 3rd layer | 60 | 40 | 2.4339 | 0.0661 |
| 2nd layer | 70 | 30 | 2.4571 | 0.0429 |
| 1st layer | 80 | 20 | 2.4748 | 0.0252 |

## 3. Results and discussion

Figure 2 displays the morphologies of the Al powder and GM particles. The Al powder exhibited an irregular shape with a rough surface, characteristic of an air-atomized process. In contrast, the GM particles were spherical with smooth surfaces and consistent sizes. Notably, no significant aggregation of either Al powder or GM particles was observed across all samples. Table 2 presents the particle size distribution of both the Al powder and GM particles; the mean particle size of GM was approximately half that of the Al powder. EDS analysis of the GM particles revealed the presence of major elements, including oxygen (42.11%), silicon (26.21%), carbon (19.06%), and sodium (12.61%), suggesting a silica composition.

Figure 3 illustrates the effect of compaction pressure on the relative density of the FGAGM foams, both before and after sintering. A clear trend emerged as the density of the as-compacted FGAGM foam increased with increasing compaction pressure. Conversely, the density of the sintered FGAGM foam decreased as compaction pressure increased. For all compaction pressures, the pre-sintering densities were higher than their post-sintering counterparts. The initial increase in density is attributed to improved particle packing and a reduction in porosity within the green foams. The observed decrease in post-sintering density may be due to reduced sintering activity at higher initial compaction pressures. This could potentially arise from limited particle rearrangement or alterations in pore structure that hinder densification, indicating that while higher compaction pressures enhance green density, they might adversely affect the subsequent sintering process. It has been shown that excessive compaction pressure can limit particle rearrangement and create closed pores, thereby inhibiting densification during sintering [19,20]. Therefore, optimizing compaction pressure is critical to achieve a balance between initial compactness and effective sintering behavior, thereby ensuring the desired final material properties.

Figure 4 presents the microstructure of the sintered FGAGM foams. The black particles observed are remnants of the GM particles and are confirmed via EDS analysis. Major cracks in some GM particles were observed. The GM particles were found to be evenly dispersed within the Al matrix under all compaction pressures, demonstrating the efficacy of the powder mixing process. Significantly, no major pores or other macroscopic defects, such as flaws or cracks, were evident in the microstructure, suggesting a fully developed sintering process that resulted in dense and rigid composite foams. The reduced density post-sintering is likely attributed to sintering shrinkage and potential trapped gas porosity that may form during heating. Although no major macropores are visible in the microstructure, micro-voids and incomplete bonding at certain particle boundaries can contribute to a drop in density. In addition, minor defects in the form of fractured GM particles were occasionally observed, particularly at higher compaction pressures. This suggests that the application of excessive pressure during powder consolidation can induce fracture or damage in the GM particles, which may compromise load transfer and energy absorption capabilities.

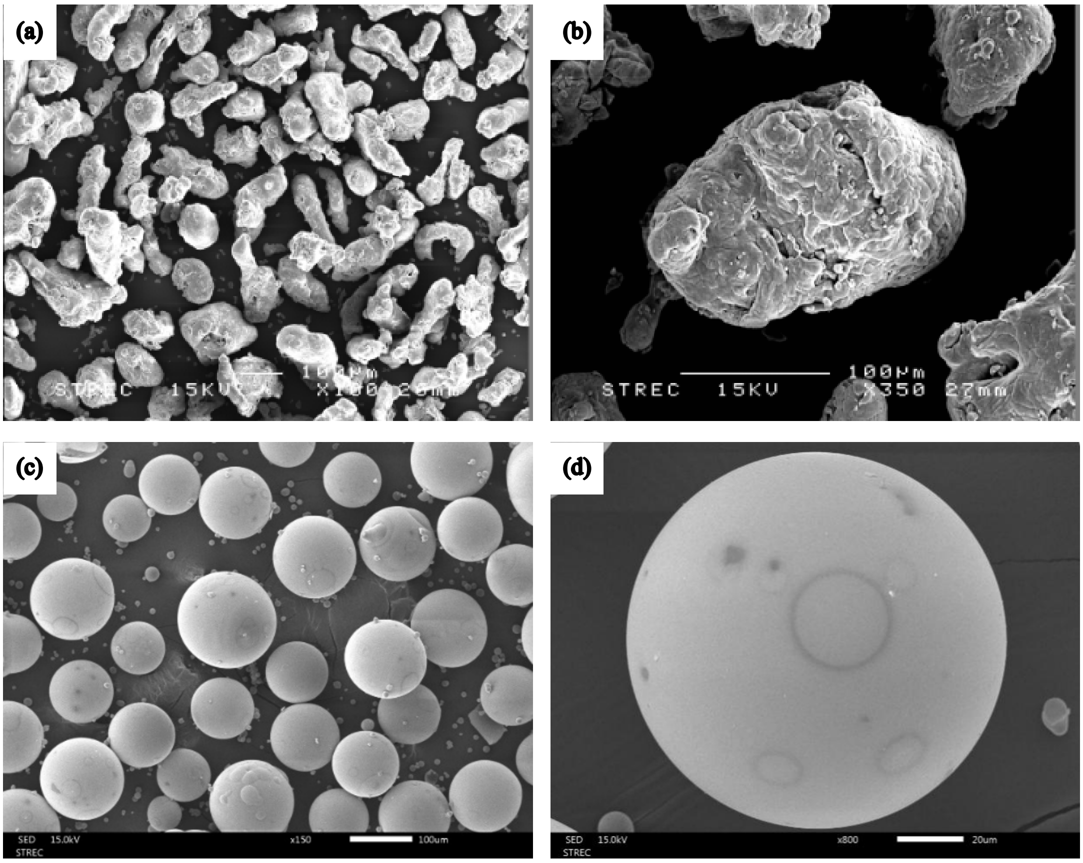

**Figure 2.** SEM images showing morphologies of (a), (b) Al powder and (c), (d) glass microsphere particles.

**Table 2** Particle size distribution of Al powder and GM particles

| Materials | Purity [%] | $D_{10}$ [µm] | $D_{50}$ [µm] | $D_{90}$ [µm] | Mean diameter [µm] |
|---|---|---|---|---|---|
| Al | 99.7 | 45.62 | 101.55 | 184.10 | 108.44 |
| GM | 99.9 | 25.25 | 54.89 | 97.22 | 59.63 |

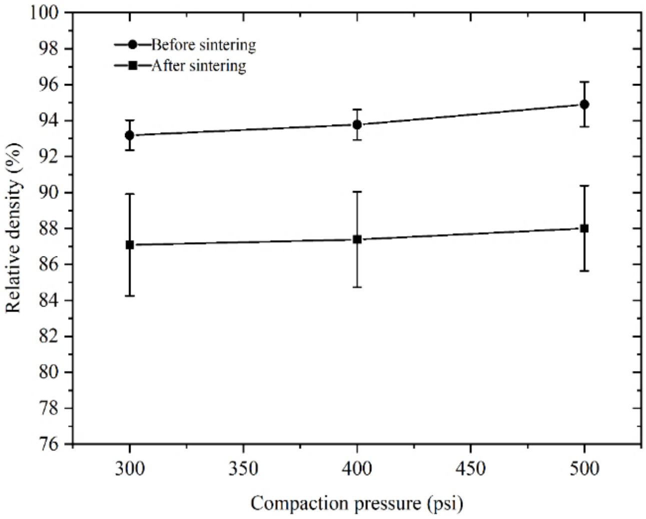

**Figure 3.** Relative density of FGAGM foams before and after sintering

During sintering, diffusion bonding occurs between the Al matrix and the surface of the hollow glass microspheres. The ceramic-metal interface is maintained without significant reaction, preserving the integrity of the microspheres. The limited chemical bonding between aluminosilicate glass and Al under solid-state sintering conditions is reported [20]. Mechanical interlocking and limited interfacial diffusion contribute to the load transfer mechanism, consistent with prior studies [21].

Figure 5 illustrates the mechanical properties of the FGAGM foams, specifically their compressive yield strength and energy absorption capacity. In all cases, both the yield strength and energy absorption of the foams consistently increased with increasing compaction pressure. The application of higher compaction pressure during the powder metallurgy process leads to enhanced particle rearrangement and increased packing density. The densification results in smaller and more uniformly distributed residual pores in the green body, which, after sintering, contributes to a more continuous matrix structure. The reduction of porosity within the green compact is paramount for superior mechanical performance. Lower porosity translates to fewer voids and defects, thereby improving the material's load-bearing capacity and, consequently, its strength and energy absorption. Furthermore, higher compaction pressures bring GM particles into closer proximity, increasing their contact area. This increased proximity facilitates improved diffusion during sintering, leading to stronger metallurgical bonds between particles. Such enhanced bonding contributes significantly to the overall integrity of the material, augmenting its resistance to deformation and failure under applied loads.

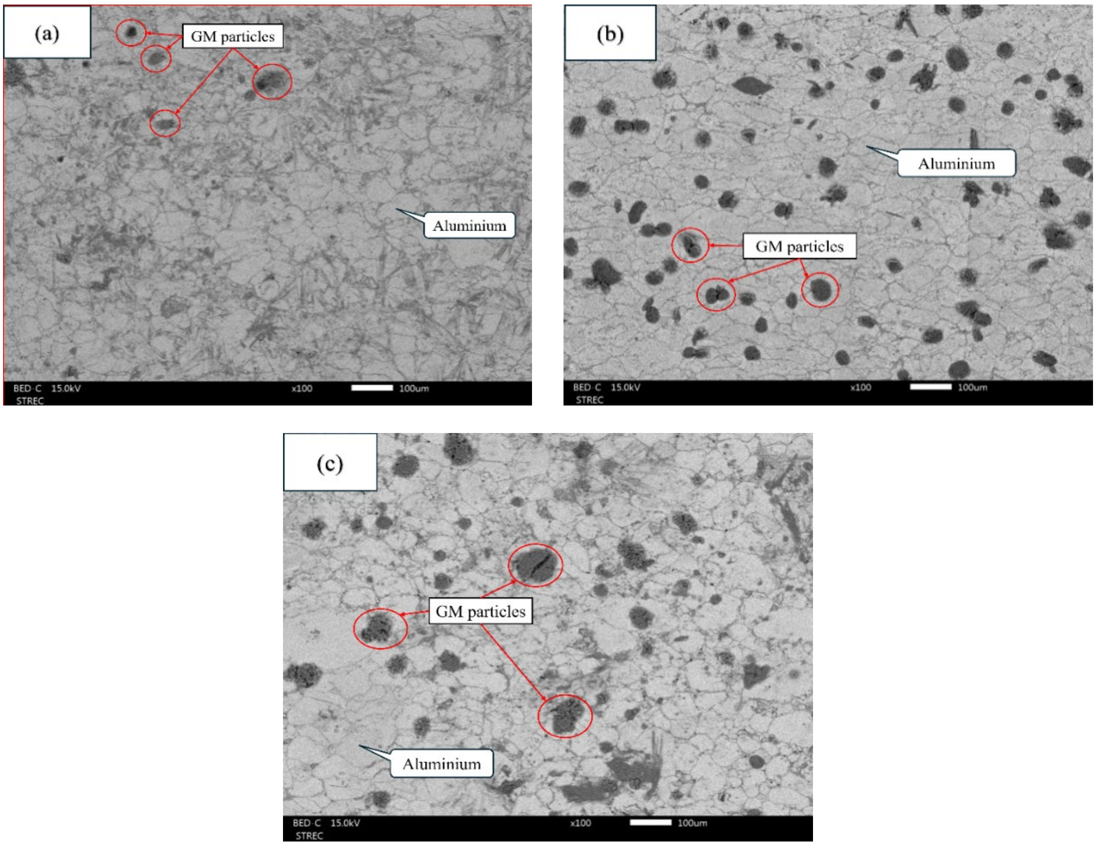

**Figure 4**. Back-scattered mode SEM images showing FGAGM foams, after sintering, produced at compaction pressures of (a) 300, (b) 400, and (c) 500 psi.

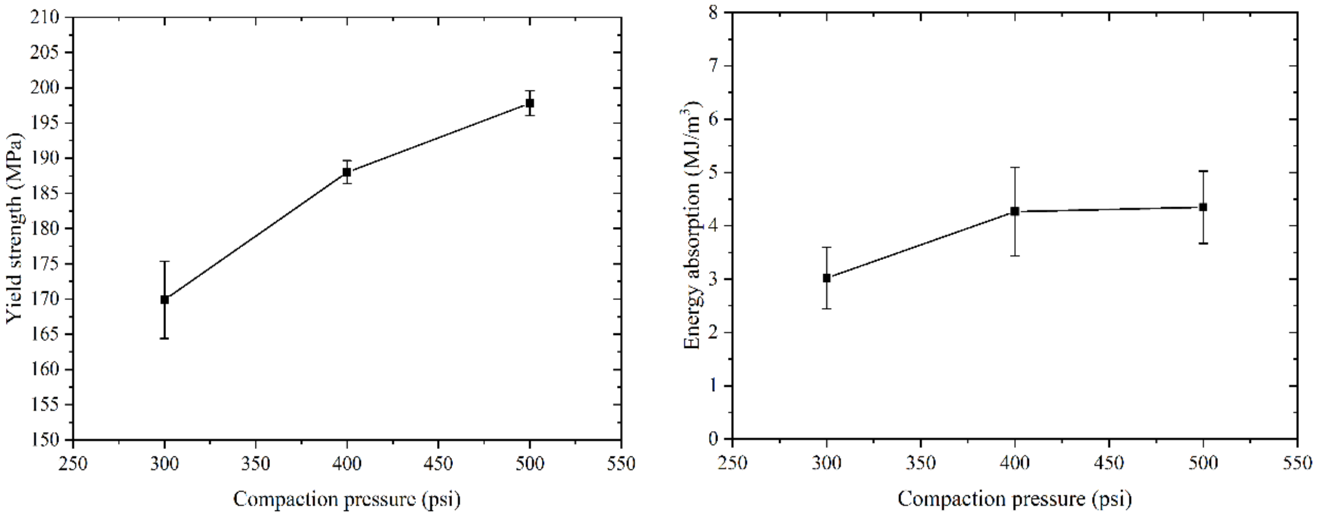

**Figure 5** Yield strength and energy absorption of FGAGM foams produced at different compaction pressures.

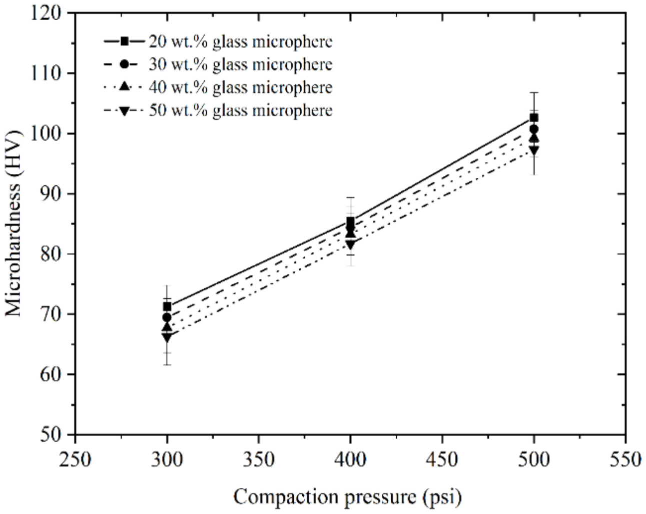

**Figure 6.** Microhardness values for FGAGM foams produced at different compaction pressures.

Figure 6 displays the microhardness measurements of the sintered FGAGM foams for each GM composition layer across different compaction pressures. In all instances, an increase in compaction pressure resulted in an increase in the foam hardness, signifying enhanced densification and reduced porosity within the material. It is worth noting that the hardness of GM is approximately 108 HV (kg·mm$^{-2}$). Another observation revealed that hardness decreased with increasing GM content. This can be attributed to several interrelated factors. GM particles are inherently brittle and possess lower hardness compared to the Al matrix. GMs, particularly of the hollow type used in this work, are inherently prone to fragmentation or incomplete packing during compaction, especially at higher concentrations. As their volume fraction increases in a given layer, the likelihood of interparticle voids, poor interfacial bonding, and trapped air rises, all of which contribute to a greater total porosity. This correlation between increasing GM content and porosity has been consistently reported in prior studies [9], where a rise in microsphere volume fraction led to increased void formation and decreased composite density. As their volume fraction increases, GMs occupy a larger portion of the composite, effectively reducing the overall hardness of the foam specimen. Additionally, incorporating a higher content of GM introduces more voids into the Al matrix, increasing the overall porosity of the foam. Similar trends in decreasing hardness with increasing GM content have been observed in Al-syntactic foams by [22]. The softening effect is attributed to lower load transfer, due to weak interfaces and the hollow nature of GMs. This heightened porosity leads to a decrease in the effective load-bearing area, rendering the material more susceptible to deformation under applied loads.

While increasing compaction pressure generally improves mechanical properties, there exists an optimal range beyond which additional pressure may not yield significant benefits and could potentially introduce defects. Excessive pressure might lead to the crushing of GM or the introduction of residual stresses. Therefore, identifying the optimal compaction pressure is crucial to balance densification and maintaining the integrity of the FGAGM foams.

Compared to alumina or SiC-filled syntactic foams, the Al-GM system shows lower density but also reduced stiffness. However, energy absorption and formability are enhanced due to the deformability of the hollow GMs. The optimum condition, based on combined mechanical strength, energy absorption, and microhardness analysis, was found at the compaction pressure of 400 MPa. This level provided adequate densification without crushing the hollow GMs excessively and maintained a beneficial gradient structure.

## 4. Conclusion

This study provides a significant insight into how compaction pressure influences the microstructure and mechanical performance of FGAGM foams. Increasing compaction pressure enhances the green (pre-sintering) density of FGAGM foams due to improved particle rearrangement and reduced porosity. However, post-sintering densities exhibited a decreasing trend with higher compaction pressures, attributed to hindered sintering dynamics at elevated pressures. The enhancement of strength, hardness and energy absorption in FGAGM foams with increased compaction pressure is primarily due to improved densification, reduced porosity, and better particle bonding. However, it's essential to optimize the compaction pressure to maximize these benefits without compromising the structural integrity of the composite. In this study, the optimum condition, based on combined mechanical strength, energy absorption, and microstructural uniformity, was found at the compaction pressure of 400 MPa. These findings suggest potential for FGAGM foams in lightweight energy-absorbing structures, and future optimization could explore tailored gradient architectures and alternative sintering routes.

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