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Effect of Pre-Compaction Stage Parameters on the Defects Occurring During Post-Compaction Stage in Aluminium Foams Fabricated via Powder Metallurgy Using Carbamide Space Holders

Viral D. Panara, Akash V. Vyas*, and Mayur P. Sutaria

CHAMOS Matrusanstha Department of Mechanical Engineering, C. S. Patel Institute of Technology, Charotar University of Science and Technology, CHARUSAT, Changa - 388421, Gujarat, India; akashvyas.me@charusat.ac.in

Abstract

Acceptance of a newly developed material into the industry highly depends upon its characteristics and ability to produce consistently and efficiently based on the needs. Metal foams are the materials facing difficulties getting adopted because of their structural randomness. Metal foam offers very unique property combinations, but it is difficult to maintain consistent production. Foam produced by the powder metallurgy method using the space holder helps achieve the desired pore morphology and characteristics. Every process step demands attention to ensure the quality. The manufacturing process can be divided into two parts based on the state of the raw material, i.e., (1) pre-compaction and (2) post-compaction stage. During the water leaching, a post-compaction stage process, samples exhibiting the spalling issue have direct relevance to the raw material produced at the pre-compaction stage. In this investigation, an attempt has been made to systematically investigate the effects of the pre-compaction stage process on the defects encountered during the post-compaction stage. The mechanisms are discussed in detail, where the root cause for the occurrence of a particular defect lies in the pre-compaction stage process, mainly space holder mixing and die-filling.

Major Findings: *A defect caused by the foam production method and material conditions, spalling is a post-compaction phenomenon with roots in the pre-compaction stage. The overall quality and structural integrity of aluminium metal foam are largely determined by important pre-compaction parameters, such as die filling, time interval, mixing procedure, and binder addition.*

Keywords: *Metal Foam, Powder Metallurgy, Spalling, Space Holder, Water Leaching*

1.0 Introduction

Metal foams, also called porous metals or cellular metals, are a very unique type of metal which contains porosity. This type of metal has diverse and unique properties in comparison to conventional materials. In general, the presence of porosity is not desired in materials, and the material can outperform during its service, but controlled creation of porosity in materials can give very creative and diverse properties. It offers significant alteration in many basic properties such as weight, density, surface area, energy absorption, thermal conductivity, impact strength, sound absorption, etc[1]. This class of novel materials have attracted the attention of many researchers because of such variation in properties and potential applications. Metal foams are classified as open-cell and closed-cell types depending upon the pore morphology[2]. They are made by using either liquid or solid-state metal processing routes[3].

*Author for correspondence

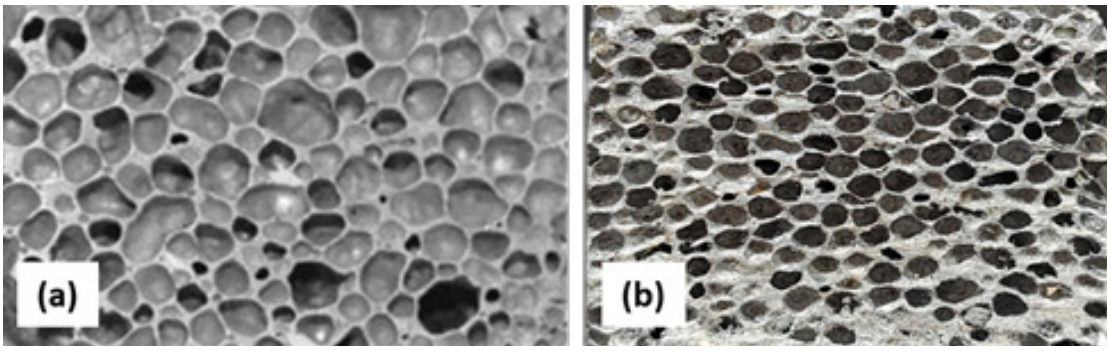


Figure 1. Metal foams produced using **(a)** Liquid state process[8] **(b)** Solid state process by the authors.

In the liquid state metal foaming process, either non-reacting gas is injected into the melt or different blowing agents are introduced into molten metal, which releases the gas and forms bubbles in the melt. Non-reacting gases such as carbon dioxide, argon or nitrogen can be injected into liquid aluminium to produce foams. In another method of liquid state foaming process, different chemicals as a blowing agents like Titanium hydride (TiH_2), Magnesium Hydroxide ($Mg(OH)_2$, Magnesium carbonate ($MgCO_3$), Dolomite ($CaMg(CO_3)_2$), and calcium carbonate ($CaCO_3$) are introduce in the melt to release the gases[4]. In the solid state process, one of the routes to develop metal foam is where the blowing agents are mixed with metal powder, and compaction is carried out[5]. Then these green samples, called a precursor, are heated at the temperature range required for the blowing agent to activate and create porosity[6]. Foams developed using solid state processing have better control over pore morphology and distribution[7] in comparison to liquid state processing, as shown in Figure 1.

In solid state processing, another route where space holders are mixed with metal powder and compaction is carried out to develop the foam. Subsequently, the space holder material is removed either by soaking it into suitable liquid media or by burning it out at higher temperature[9]. The advantage of the space holder technique over gas blowing agent is that more precise control over the size and shape of pores, hence the desired pore density can be achieved[10,11]. Pore morphology of desire shape and size can be achieved in metal foam by selecting the space holder accordingly[12,13]. While in the gas foaming technique, it is very hard to gain control over foaming due to the very high volatility and complex gas mechanics. As shown in Figure 2, the distribution of pores, shape and size is more uniform in samples developed using the space holder as compared to the blowing agent technique. The fabrication of foam with the powder metallurgy method using space holder has several challenges, such as spalling of the sample, loss of green strength and insufficient leaching out of space holder from the green compact. These issues compromise the consistent quality of metal foams, and the causes for such issues must be considered while manufacturing foams.

Aluminium is a non-ferrous metal with low density and a higher strength-to-weight ratio, which leads to a reduction in the weight of the components. In metal foams, many researchers have carried out a significant amount of study to create lightweight aluminium foam[6]. One of the most common methods to produce aluminium metal foam from a metallic powder is powder metallurgy. The

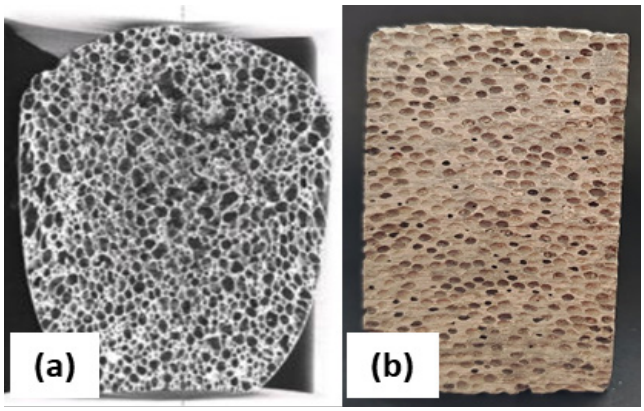


Figure 2. Metal foams produced by powder metallurgy method using **(a)** gas blowing agent (TiH_2),[14] **(b)** Space holder (Carbamide) by the authors.

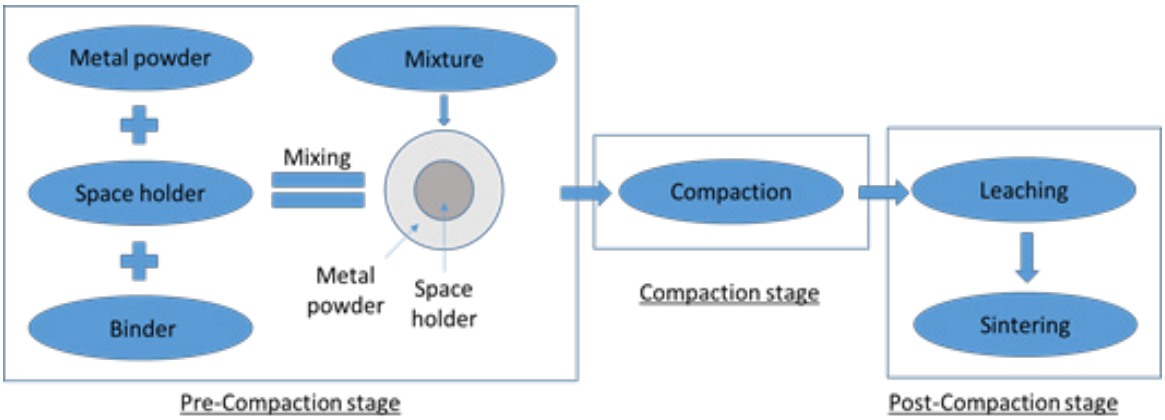


Figure 3. Processing stages of the metal foam fabrication by powder metallurgy using space holder.

method has three process stages, i.e. (a) Pre-compaction, (b) compaction and (c) post-compaction as presented in Figure 3. In this technique, porosities of desired shape and size are created using space holders such as NaCl[15], Carbamide[16], Magnesium sulfate $MgSO_4$[17], Ammonium Bicarbonate[18], Saccharose[19]. These are widely studied space holder materials because of their characteristics and compatibility with metal powders in solid state processes. Carbamide is one of the most used space holders in which researchers have focused more because of low evaporation temperature and excellent solubility in water, which helps significantly during the leaching stage in order to create pores in the compacted green sample[20]. Space holders were mixed with the metallic powder using liquid binders. This mixture was filled into the die and compacted at the desired pressure to generate the green sample, and subsequently water leaching process was carried out to remove space holders from the green sample. After removal of the space holder, the sintering was carried out in which samples were heated to achieve base metal strength[21].

The powder metallurgy process is highly sensitive to parameters at all stages, and defects like spalling and cracking can arise due to multiple factors. These defects are generally attributed to pre-compaction, compaction and post-compaction causes, each contributing uniquely to structural failures. Bafti and Habibolahzadeh[21] have used the powder metallurgy method to create the foams using carbamide as a space holder. They evaluated the effect of compaction pressure, dissolving time of space holder, sintering temperature, and time. They have prepared 30 mm diameter samples, each having a 40 g weight by varying the porosity in the range 40 to 85 %, compaction pressure in the range 200 to 400 MPa and leaching time in the range 2-4 hours. The leaching process was carried out at room temperature. It is observed that at lower compaction pressure (< 300MPa), the powder was not able to create enough green strength to survive in the water leaching process. Aluminium powder gets washed away from the surface and damages the sample as shown in Figure 4.

Michailidis and Stergioudi[22] have observed that the lower compaction pressure promotes the spalling, and at higher compaction pressure, cracks are formed as shown in Figure 5. Insufficient compaction pressure leads to weak inter-particle bonding, resulting in green compacts that are prone to disintegration or spalling during subsequent processing stages. Conversely, excessive

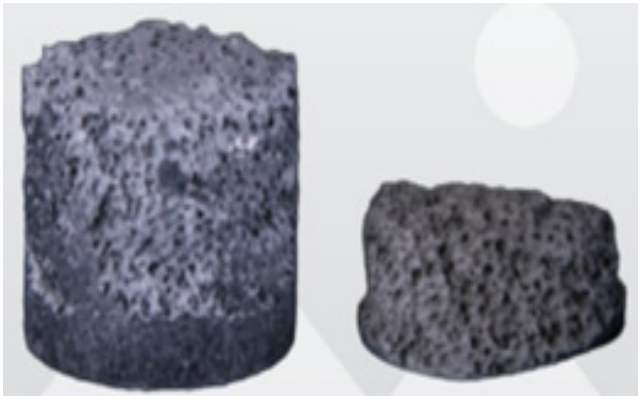


Figure 4. Spalling over sample surface[21].

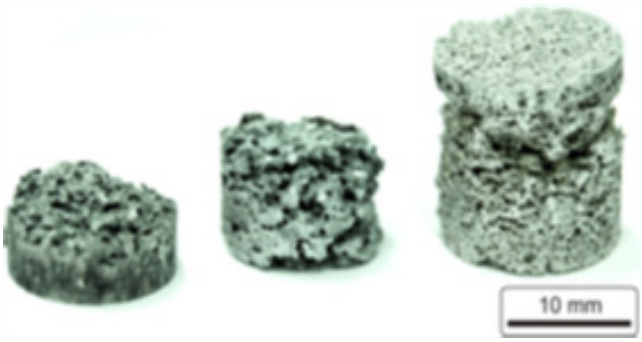


Figure 5. Spalling over the sample surface along with cracks[12].

compaction pressure may deform or fracture the carbamide particles, leading to irregular pore structures and microcracks within the aluminium matrix. These microcracks can propagate during sintering, culminating in surface spalling and compromised mechanical properties.

In another study, using the Sintering and Dissolution process (SDP), Zhao and Hana[23] noticed the same type of spalling during the water leaching process (4 hours of leaching time at 90°C temperature) in a few samples of aluminium foam with NaCl space holder material as presented in Figure 6. In the SDP route, the space holder is not removed before sintering. Instead, the compact is sintered with the space holder in place, and leaching is done afterwards to create porosity. While this approach has some benefits, like improved dimensional stability during sintering, it also introduces a significant risk of crack formation. During sintering, aluminium particles begin necking and bonding. However, the space holder still occupies volume and acts as a temporary structural support. During the subsequent leaching process, these supports are suddenly removed, but the foam's structure may not have fully densified or stabilised. This sudden loss of constraint can cause collapse or cracking, especially in regions with thin walls[24].

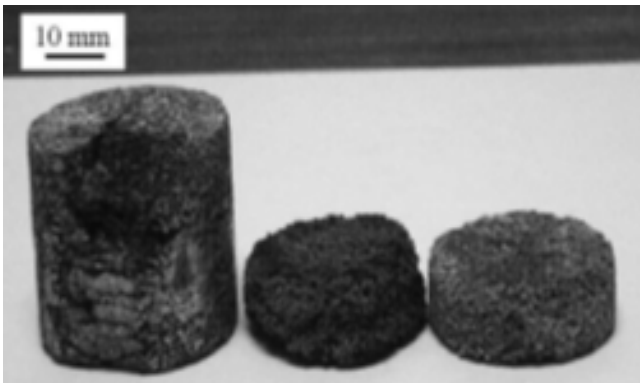


Figure 6. Spalling over the sample surface along with cracks[23].

The majority of past works in the literature focus on compaction and post-compaction parameters like compaction pressure[25], sintering time and sintering temperature[26], pore density[27,28], leaching time and leaching temperature[29], etc. There are other significant factors related to the pre-compaction stage which play an important role in obtaining consistent and quality products, like powder and space holder mixing process, loading of mixture in the die process, removal of space holder process and metal powder to binder ratio (inadequate or excessive binders). In this work, a systematic study on the pre-compaction stage processes has been carried out. The mechanisms for the occurrence of defects have been discussed in detail, where the root cause lies in the pre-compaction stage processes.

2.0 Materials and Methods

In the present study, aluminium powder of 65 micrometre size is used to develop the samples. Carbamide particles as a space holder are selected with a 2 mm to 3 mm diameter range. The total weight of the mixture is kept at 60 grams to prepare a cylindrical sample with a diameter of 32 mm alcohol with 2 wt. % of the mixture is used as a liquid binder, and the mixture was carried out manually in a stainless steel bowl to ensure homogeneous distribution. Before die filling, graphite powder is applied to the surfaces of the die and punch, which act as a solid lubricant, minimising friction during the compaction and while removing the sample from the die. Manually die is filled with a mixture after positioning it in a unidirectional hydraulic compaction press. Compaction loads of 100 KN, 200 KN, and 300 KN are applied, each with a dwell time of 5 minute[3,6]. Carbamide proportions of 60 wt. %, 70 wt. %, and 80 wt. % were selected[9,20]. Water leaching is performed to remove the space holder material (carbamide). The water leaching is carried out at a controlled temperature of 60 °C for a duration of 120 minutes[12].

3.0 Results and Discussion

Despite being manufactured under controlled and consistent conditions, some samples exhibited abnormal behaviour during the water leaching process. Specifically, in certain cases, aluminium powder was partially expunged along with the dissolution of the carbamide space holder. Furthermore, a few samples experienced a significant loss of green strength following leaching. This indicates that, although the post-compaction parameters were adequate for sample formation, the root cause likely lies within the pre-compaction stage. Consequently, each step of the pre-compaction process was thoroughly examined, and specific observations were made to identify and mitigate the underlying issues.

3.1 Binder Addition

The selection of a suitable binder is based on several key criteria, including evaporation characteristics at elevated temperatures, chemical compatibility with both the metal powder and space holder, and the stability of its binding properties. Water and alcohol are the most commonly employed binders for such applications[30]. Upon thorough evaluation of each stage in the manufacturing process, the author identified the binder addition and its quantity used during the preparation of the aluminium powder and carbamide mixture as a potential contributing factor to spalling, among other possible causes.

The sequence in which the binder is introduced, whether into the powder, the space holder, or a pre-mixed combination, significantly influences the homogeneity and quality of the mixture. Direct addition of the binder to the aluminium powder often results in lump formation, which hinders uniform mixing with the carbamide particles. Similarly, introducing the binder into a pre-mixed powder and space holder blend tends to produce a non-uniform mixture, as the wetted powder adheres to the walls of the mixing bowl. Moreover, agglomeration between space holder particles and between aluminium powder can occur under these conditions, as illustrated in Figure 7 (a) and Figure 7 (b).

The most effective mixing approach was witnessed while spraying a fine mist of binder over the space holder before the gradual addition of aluminium powder. This approach yielded the most uniform and stable mixture, thereby reducing the likelihood of defects such as spalling. The required amount of binder varies depending on the proportions of metal powder and space holder, as well as environmental conditions. For instance, alcohol readily evaporates into the atmosphere, whereas water can be absorbed by carbamide due to its hygroscopic nature. According to the literature, 2 wt. % alcohol is commonly used for manual mixing[22,23]. Furthermore, excessive binder promotes agglomeration of carbamide particles by causing them to stick together, forming conjugated pores with extremely thin cell walls, which may be prone

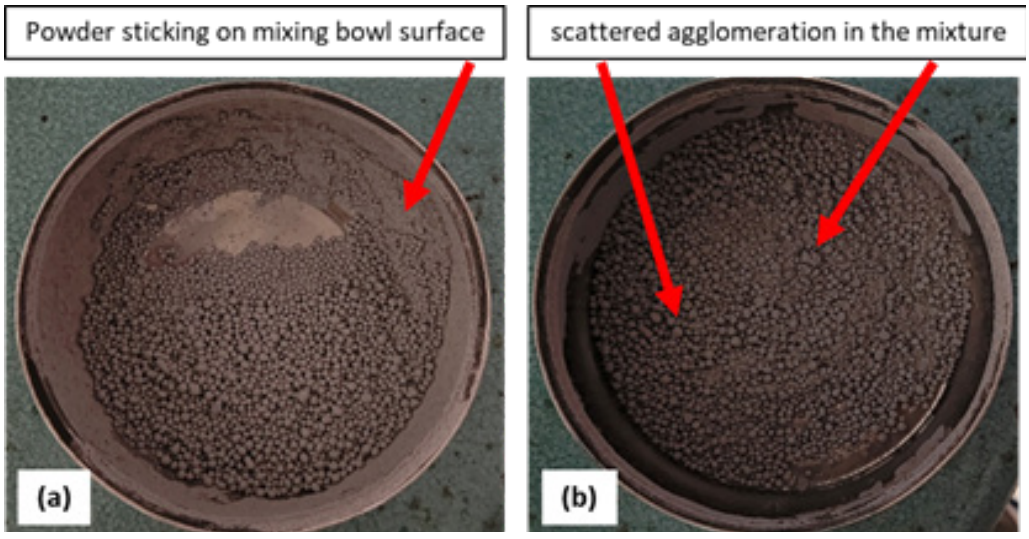


Figure 7. Addition of binder **(a)** in premix (aluminium and carbamide) **(b)** in aluminium powder.

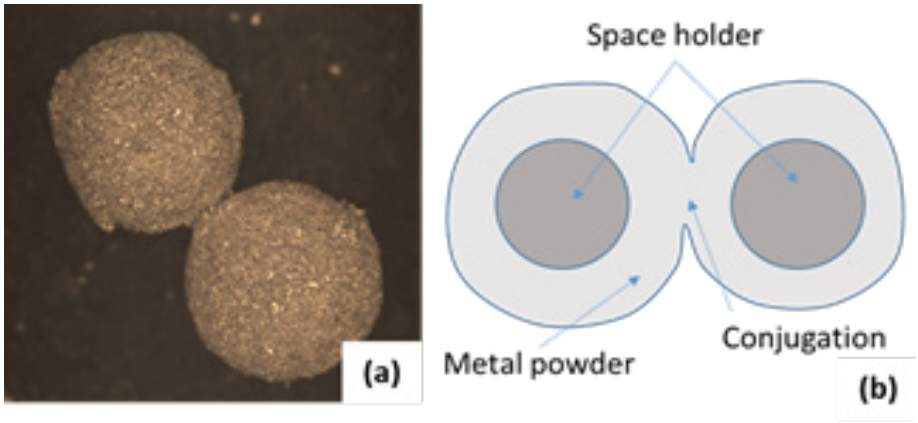


Figure 8. Effect of a higher amount of binder.

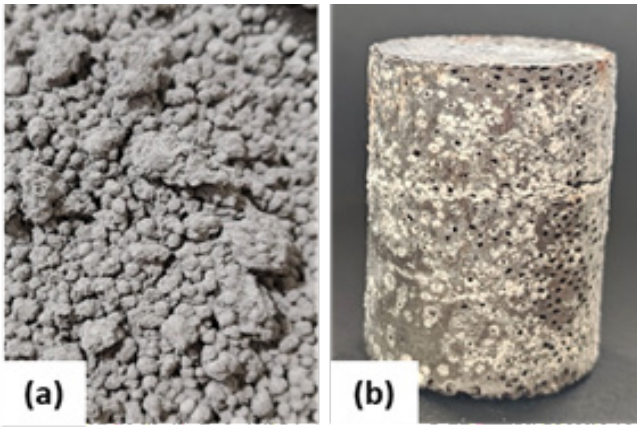


Figure 9. Higher amount of binder **(a)** agglomerated mixture and **(b)** cracks in green sample.

to spalling Figure 8 (a). As shown in schematic Figure 8 (b) conjugated location has the least distance between the space older, which may further reduce during compaction.

Additionally, the presence of an excessive amount of binder in the mixture, which agglomerates carbamide particles as shown in Figure 9 (a), can lead to binder extrusion under compression. This binder leakage forms undesirable macro-channels within the green compact, which can result in cracking, as seen in Figure 9 (b). These types of cracks can be present on the surface or internal locations of the sample. Such surface cracks are visible after water leaching.

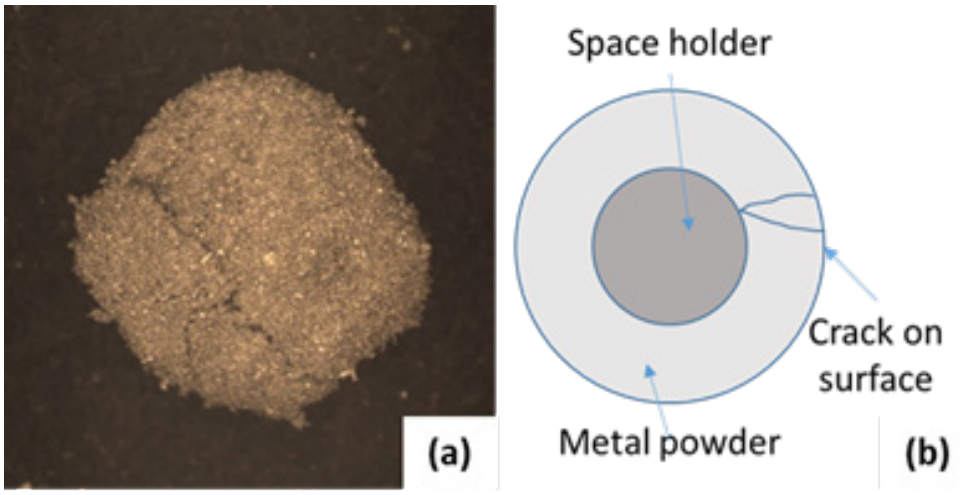


Figure 10. Presentation of crack over the surface of the coating in **(a)** actual and **(b)** schematic.

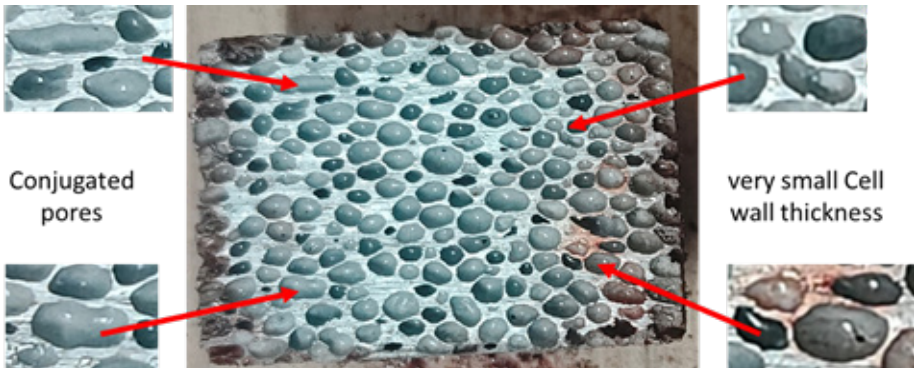


Figure 11. Various sites with conjugated pores and thin cell walls.

In cases where the mixture contained an adequate amount of binder, another issue was observed: surface cracks on the powder coating, as shown in Figure 10(a). These cracks may result from abrupt or rapid drying of the mixture due to sudden changes in atmospheric humidity. Such cracks can expose the carbamide particles during compaction, increasing the likelihood of direct contact between adjacent particles, which ultimately leads to coupled or merged pore formation, as seen in Figure 11.

The observations discussed above may reflect the influence on the formation of conjugated pores in the sample, whether considered individually or in combination as shown in Figure 11. Such locations have having high probability for spalling because of thin cell walls, which do not provide sufficient green strength.

3.2 Mixing Process

Mixing is the initial and crucial step in preparing the raw material for metal foam fabrication. During the mixing process, aluminium powder sticks to the surface of the space holder, which defines the probable cell wall thickness. The metal powder and the space holder are blended to produce a uniform mixture suitable for die filling. In general, mixing is performed either manually or using a ball mill[10]. Manual mixing is particularly effective during the development phase, where the optimal combination of constituents is being identified. One of the key advantages of manual mixing is the ability to immediately address issues such as agglomeration caused by excess binder, which can be resolved by breaking up lumps or allowing them to dry. Similarly, insufficient binder can be visually detected and compensated for in real-time during the mixing process. The ideal combination of metal powder, space holder, and binder depends on several factors, including ambient temperature, relative humidity, and the evaporation rate of the binder. Controlling these factors is essential to prevent agglomeration, achieve uniform binding quality, and ensure consistent powder coating thickness. The use of a ball mill becomes advantageous once the formulation is well established and the influence of environmental and processing parameters is well understood and controlled. Additionally, stepwise addition of small quantities of binder and powder during mixing has been found to enhance mixture uniformity and overall consistency.

3.3 Mixture Utilisation Timeframe

The time interval between mixture preparation and its usage can also determine the binding characteristic of the prepared mixture. As shown in Figure 12 (a) and Figure 12 (b), 2 wt. % alcohol proportion is sufficient to produce a homogeneous and uniformly layered powder coating for a 60-gram mixture containing 80 wt. % carbamide.

However, if the prepared mixture is exposed to open air for an extended period, the bonding between the carbamide and aluminium powder weakens due to alcohol evaporation. When the mixture was prepared and subsequently used for die filling, the powder initially adhered to the carbamide and began to detach as time progressed, as shown in Figure 13 (a) and Figure 13 (b).

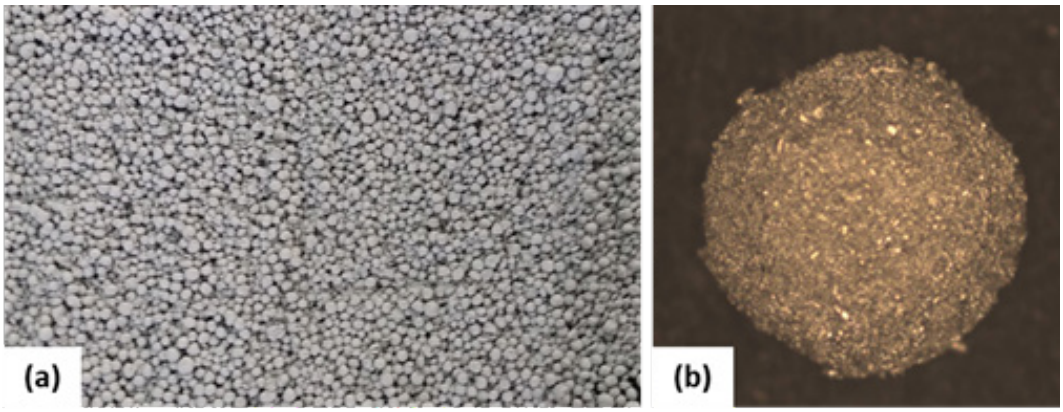


Figure 12. Use of 2 wt% % alcohol binder indicates **(a)** uniform mixture, **(b)** individual particle.

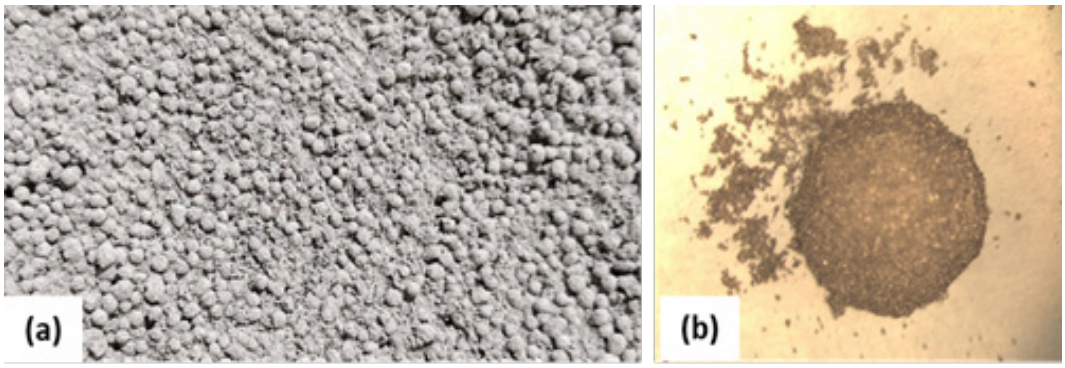


Figure 13. Mixture after 30 minutes **(a)** loose powder in a pool of mixture **(b)** individual particle.



Figure 14. **(a)** loose powder detached from the coated particle. **(b)** localised spalling in the foam sample after water leaching.

Therefore, during the mixing process, it is essential to ensure mixture uniformity through frequent visual inspection, especially considering the effect of binder evaporation. The gradual addition of small quantities of binder can help maintain the desired consistency and improve the stability of the mixture.

During exposure to the open atmosphere, the combined effect of imbalances in temperature and

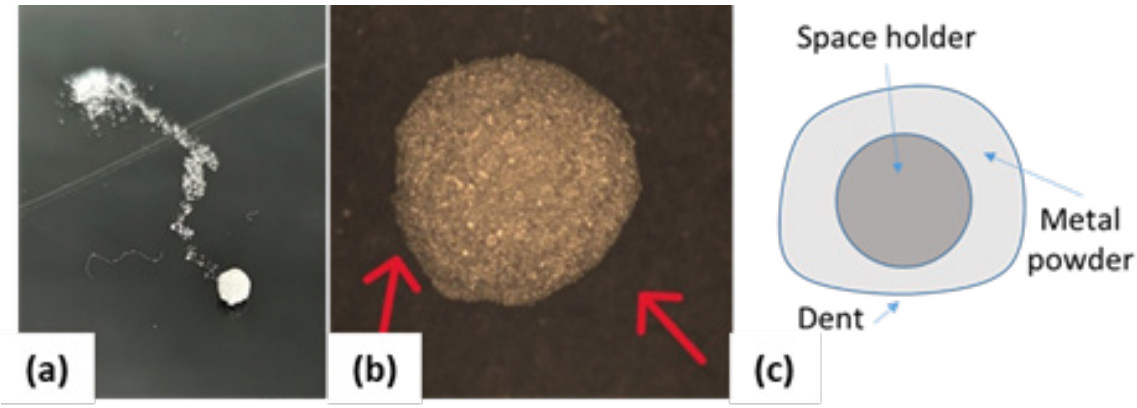


Figure 15. **(a)** powder traces after roll over of coated particle on surface **(b)** dent on the surface of coated particle disturbing circularity because of falling from height **(c)** schematic representation of dent over surface.

humidity levels of both the mixture and the surrounding environment, the cohesive bonding between the aluminium powder and the space holder begins to weaken. As a result, when the mixture is filled into the die, loosely bound powder particles accumulate in undesired locations, leading to non-uniform pore distribution. From Figure 14 (a), the detachment of aluminium powder disrupts the uniformity of cell wall thickness, adversely affecting the pore morphology. The presence of numerous thin cell walls concentrated in localised regions lacks the mechanical integrity to withstand the forces applied during water leaching. Consequently, localised spalling of powder from the green compact occurs, as shown in Figure 14 (b).

3.4 Die Filling

Another observation during die filling revealed that particles often roll or slide across surfaces, causing a small amount of powder to adhere to those surfaces, as shown in Figure 15 (a). This phenomenon disturbs the uniformity of the powder coating on carbamide particles. Moreover, when the mixture strikes the die surface, it can leave dents in the coating, disrupting circularity and creating uneven thickness, as shown in Figure 15 (b) and Figure 15 (c). Therefore, the falling height of the mixture inside the die plays a significant role in determining the coating integrity. These deformations occur even when sufficient binder is present. The impact of particles striking the die surface can dislodge powder, further deteriorating the mixture and quality.

In a case of insufficient binder content, poor particle bonding can be observed, which may cause powder to remain loose. When such poorly bonded powder mixtures are filled into the die, the aluminium powder tends to separate from the mixture. This separation, along with inadequate mixture, leads to a non-uniform arrangement within the die, resulting in localised regions with imbalanced concentrations of constituent materials as presented in Figure 16.

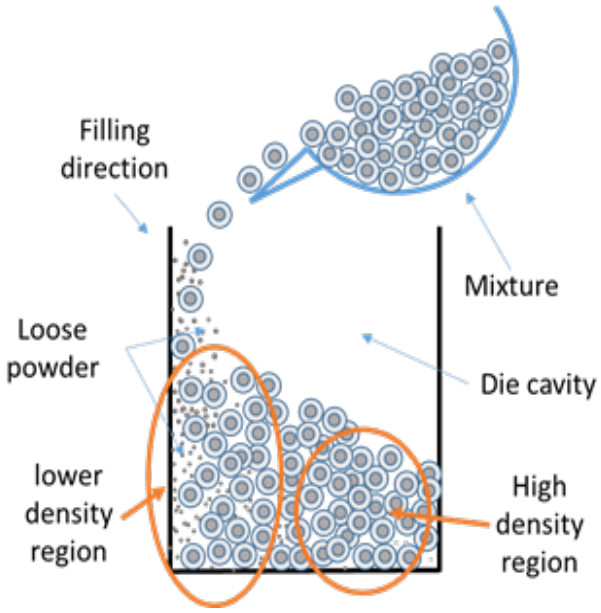


Figure 16. Movement of a mixture containing loose powder and coated particles while die filling.

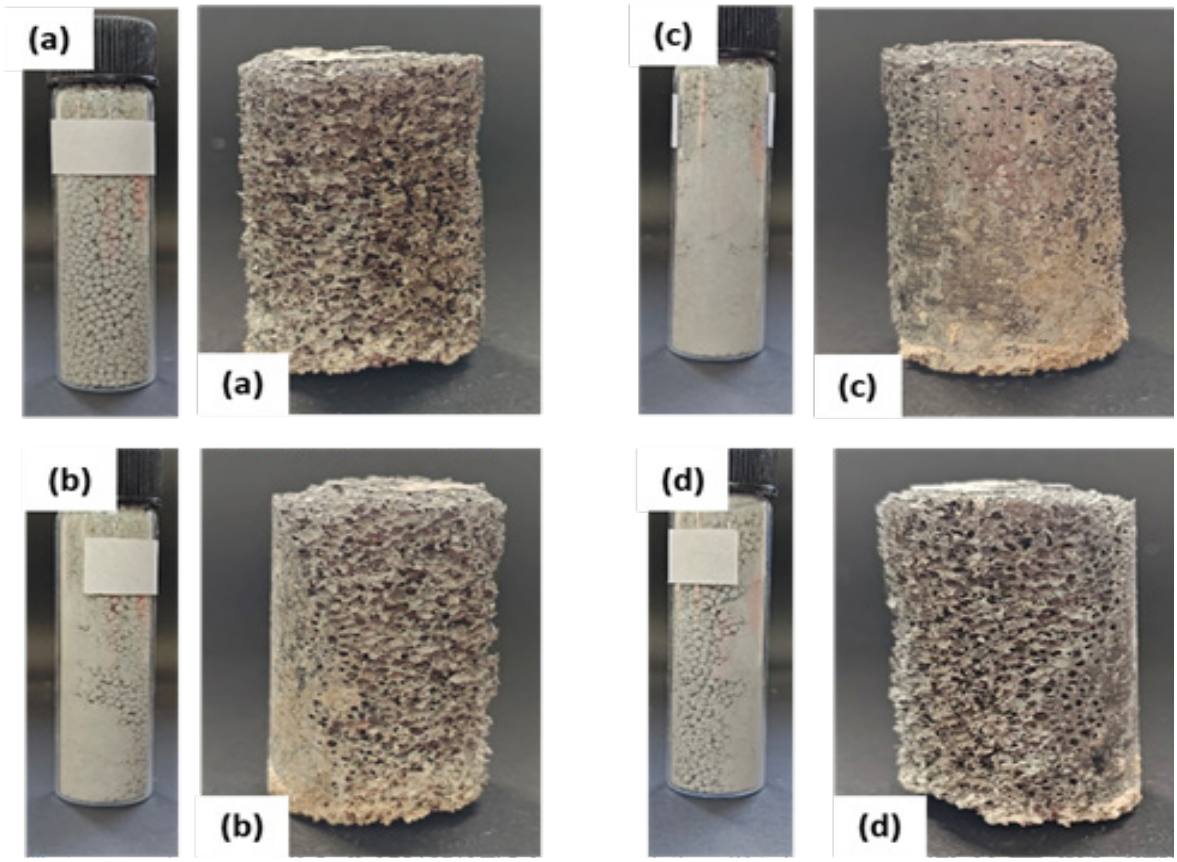


Figure 17. Surrounding visuals of foam surfaces **(a)** front side, **(b)** left side, **(c)** back side, **(d)** right side.

During die filling, it was observed that when the mixture particles move into the die, loose powder starts accumulating due to movement at one side where they strike initially in the die. As shown in Figure 17, locally separated regions of loose powder and mixture are formed. So the region containing loose powder gains higher green strength as compared to other areas. During the water leaching process, water starts penetrating the sample by creating small micro channels between pores. Water penetration is higher into such areas where cell wall thickness is less, which leads to higher transpiration of water compared to other tightly packed regions. So there are high chances of spalling into such areas where there is a lack of green strength. It depends on the uniform mixture having uniform powder coating thickness and filling into the die. In Figure 17, the effect of die filling on spalling from all four sides is presented for a single sample. It can be seen clearly that one side has high

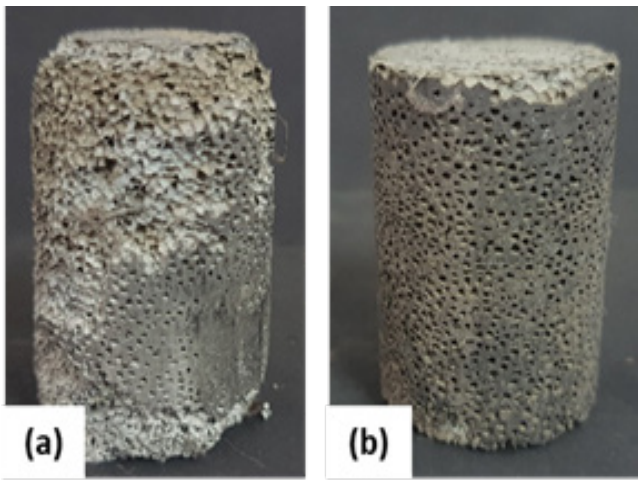


Figure 18. Both samples were made with 150KN compaction pressure and 60 wt% % carbamide space holder. **(a)** Sample with abrupt spalling **(b)** spalling-free sample.

green strength due to the presence of a high amount of loose powder compared to the other side. A clear boundary can be seen in the sample after water leaching, indicating surface spalling occurred due to improper die filling.

Figure 18 shows that two identical samples were fabricated under different conditions. One sample exhibited severe spalling, characterised by notable weight loss and distortion of geometric integrity. In contrast, the second sample remained structurally sound, effectively resisting spalling failure. This improvement is attributed to the deliberate and systematic consideration of the critical factors observed and discussed before. Therefore, the spalling effect, which is observed in post-compaction, has its roots lying in the pre-compaction stage process, such as binder amount, mixing process, time and die filling technique. The integrity of the final product was significantly enhanced through careful control and optimisation of these parameters.

4.0 Conclusion

This study analysed the manufacturing process of aluminium metal foam, focusing on the key parameters that influence the quality and structural integrity of the final product in the post-compaction stage. The findings highlight the critical role of pre-compaction parameters, are the binder addition, mixing process, time interval and die filling, in determining the outcome of the foam quality.

The amount and distribution of binder are of particular importance, as they directly affect the cohesion between powder and space holder and, consequently, the occurrence of defects such as spalling. Environmental factors like atmospheric temperature and humidity were found to significantly influence the binder's behaviour. The lead time of the mixture, as well as the method of die filling, also play critical roles in maintaining the quality of the final product. These pre-compaction factors contribute to maintaining the uniformity of powder distribution and the integrity of the post-compacted foam structure.

Primary defects at the pre-compaction stage, like surface cracks, dents, powder detachment, and particle agglomeration, are results of variations in binder content and the dynamics of the mixing and die-filling processes. Careful attention to timing, focusing upon binder quantity, as well as the method of mixture preparation and die filling, is essential for preventing these issues.

The results emphasise the importance of careful process control, particularly during the pre-compaction stages, to ensure uniform powder distribution and avoid defects. Future studies should focus on developing quantitative methods to optimise these pre-compaction parameters and simplify the manufacturing process, thereby improving the consistency and performance of metal foams for various industrial applications.

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