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Influence of Water Leaching Process Parameters on Aluminium Foam Manufacturing using Powder Metallurgy Method and Carbamide as a Space Holder

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Abstract

Metal foams are unique materials which have extraordinary property combinations, such as light in weight and high surface area, by introducing pores into them. There are many techniques to manufacture metal foams, and they have various advantages and disadvantages. The end properties of metal foams are highly dependent on pore morphology. To gain control over the generation of desired pore shape, pore size and pore density, the powder metallurgy method using the space holder technique has highly promising results. In this process, samples are made using a mixture of metal powder and space holder, compacted under an enclosed die. These compacted green samples are submerged in liquid media to leach out such space holders. In the present study, foams are made using the powder metallurgy method, where aluminium powder and carbamide space holder were selected as materials. A water leaching process was carried out to remove carbamide. The leaching parameters, such as temperature and submerge time in liquid media, are examined, as they are crucial for obtaining a fully leached space holder and for maintaining the quality of the green sample. The observations reveal that the quality of foam is influenced by factors such as compaction pressure, the quantity of space holder, water leaching time and temperature, or the interaction of all these factors. The spalling is promoted by lower compaction pressure and a higher quantity of space holder, whereas longer submergence times decrease the sample's green strength. In contrast, higher compaction distorts the shape of pores as well as hinders the removal of the space holder. This study has identified a safe range for producing quality foams with a compaction range of 100 to 150 KN, carbamide weightage of 60 to 70%, and water temperatures between 100 and 120 degrees Celsius.

Major Findings: *The process parameters were found to have a high sensitivity on the quality of the aluminium foam and the degree of spalling. Careful regulation of these parameters, especially the leaching duration and temperature, can yield noticeable improvements. The findings highlight the significance of controlling processes and parameter ranges to ensure quality consistency. Overall, this work outlines a practical and systematic approach for producing spalling-free foams.*

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

1.0 Introduction

Modern industrial revolutions have increased the consumption of metals because of their reliable performance based on different applications. The engineers and researchers have to develop and incorporate new ideas and concepts in order to achieve efficient use of metals. For example, the developments in lightweight alloys enabled a reduction in the weight of automotive vehicles, which directly contributed to the enhancement of fuel efficiency. In addition, the giant ships can be made with such engineered materials to facilitate floating with less

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self-weight, resulting in lower fuel consumption. Again, the use of lightweight materials in spacecraft to increase the payload capacity is the key requirement in space applications. Metal foams are one of the materials which have attracted researchers because of their advantages. These are spongy kinds of highly porous materials made out of solid metals with a high amount of voids and holes at the micro or macro level. In general, void or porosity is considered a defect, which causes rejection because of theoretical assumptions. However, uniform and controlled distribution of porosity may give very diverse and unique results for various engineering applications. These foams differ from conventional materials in having distinctive characteristics such as lower relative density and weight, higher compressive strength, higher heat resistance, higher load bearing capacity and higher energy absorption capacity. The noteworthy achievement of these properties was identified in aluminium foams, and therefore, the developments in aluminium foams among all other materials are receiving prime attention nowadays[1]. A number of researchers have worked on the development of metal foams, and a number of techniques are in use.

Metal foams can be produced based on the liquid or solid state of metal. In the liquid state, to produce foams, empty voids or space can be generated by a number of ways, that are injecting pressurised gas bubbles into liquid melt, use of gas foaming agents or using space holders[1,2]. The gas foaming agents have the characteristic that they start producing gas just below the liquidises temperature of the metal. Most common gas foaming agents are metal hydrides or metal oxides such as titanium hydride, boron hydride and calcium carbonate, which produce hydrogen gas or carbon dioxide and create voids or pores in metallic foam[3]. Many researchers have worked on this method and achieved relative densities ranging from 50% to 98% with pore sizes of microns to millimetres for various metals and alloys as per applications using gas foaming agents[1,2]. The use of calcium carbonate as a blowing agent was reported to achieve a fine pore size of 1 to 2 mm[4]. However, in gas injection or gas foaming agent technique, control over pore size, shape, and distribution is very difficult[5]. The reactions of the agents with metal also form the undesirable compounds that affect the mechanical properties and performance of foam[6]. In addition, though this technique is preferable for large-scale production, higher production cost and inconsistent quality also limit its application[7].

The production of foams using liquid metal and space holder is not popular because of limited materials options that can meet high process temperature requirements[8]. Whereas, in solid state metal processing, powder metallurgy is observed as a most reliable method[9]. The mixture of metal powder and space holders is compacted inside a metal die, then after leaching is carried out to remove space holders and these green compacts are sintered at elevated temperature to produce foams. Thermocol, PPMA, carbamide and plastic are the most common artificial space holders[10], while sea salt and sugar cane crystals are naturally available space holders used for metal foam production[11,12]. The space holder can be removed by various means, such as by shaking, leaching or pyrolysis[13]. The shape and size of pores can be controlled with the appropriate selection of space holder in this method helps to develop foams with different tailored properties. Aluminium is one of the most common materials which is being used in metal foams, and the most common space holders used in aluminium metal foams are salt[14], sucrose[12], NaCl[15] and carbamide[16]. Water leaching is a process where a green compacted sample is submerged in water to leach out the space holder. The space holder should be removed in a controlled manner without damaging the pore surface. The carbamide is observed in prime use as a space holder due to its lower cost and lower melting point for easy removal during leaching[17].

Jiang[13] have prepared aluminium metal foams using urea as a space holder with compaction pressure of 300 MPa to obtain defined porosity of 50%, 70% and 80%. The cylindrical samples of 20mm diameter and 20mm height were soaked in water at 80°C for 5 hours to leach out urea. It was observed that the samples with porosity of 70% or more can be leached out completely by the water leaching process, while below 70% porosity residues trapped inside the sample. In addition, though the complete leaching can be achieved for samples above 80% porosity, the green strength was observed to collapse significantly. This study was extended by the same authors, where the foam samples were prepared for the same porosity range at a compaction pressure of 400 MPa. This increase in compaction pressure helped to improve green strength without deviating the pores' shape and size[18,19]. However, higher compaction pressure hinders the complete removal

of the space holder from the samples. A study was carried out where samples were prepared with a pressure range of 300 MPa to 500 MPa and soaked in a hot water bath at 70°C and for 1 h. It was observed that in a case of 500 MPa pressure, carbamide residues were present in the sample after a leaching duration of 1 hour. It indicates that the 1 h of leaching time is not sufficient for the samples prepared with higher compaction pressure[20]. In another study, aluminium foam samples (10 mm diameter and 16 mm height) with 30% porosity were prepared using carbamide at a compaction pressure of 200 MPa. Though dissolving samples in hot water for 3 h at 80°C, carbamide residues of 1.76 % of total weight were found[21]. Similarly, Sathaiah[21] have reported in their work that the leaching time of up to 5 h at 80°C temperature helped in complete and easy removal of carbamide in Al foams with 70% porosity. It indicates an increase in leaching time, only is not sufficient enough for the complete removal of the space holder. Similarly, the use of carbamide was also explored to create 316L steel foams and water leaching was studied for the compaction pressure range of 100 MPa to 500 MPa and a mixture of 70 vol.% carbamide and 30 vol.% 316L. It was also observed that longer leaching times resulted in significant spalling of foams. Sazegaran[22] has adopted multi-stage leaching to avoid spalling under long leaching periods. The compacted foam samples were submerged in hot water sequentially with a short interval of time. It was also noted that the rate of carbamide removal was faster at the starting stages and further reduced gradually[23].

The carbamide emerged as a promising and cost-effective space holder for aluminium foam production. Previous studies have reported internal pore surface damage and spalling observations, and attempts were made to minimise the same[24]. The water leaching was observed as a critical step in defining the quality of pores and minimising the spalling. In the present work, intensive focus is made on controlling the leaching process parameters for the successful production of aluminium foams using carbamide space holder[25].

2.0 Experimental Procedure

In the present work, powder metallurgy with the space holder method was used to produce metal foams. As a metal, 99.9% pure aluminium powder produced by Loba Chemicals Pvt. Ltd., having a particle size in the range of 65 mm, was selected as the main framework material of foam. The carbamide granules, spherical in shape and with a size in the range of 2-2.5 mm, were selected as space holders. The 70% carbamide of the total weight of aluminium powder is selected for foam preparation. The ethanol with 2% vol. was sprayed on carbamide before mixing with aluminium powder. This helps to improve the adhesion of aluminium powder with carbamide and favours uniform mixing without causing agglomeration. This homogeneous mixture of 30 g was compressed uniaxially in a high-carbon steel die with a steel punch to produce a cylindrical foam sample with a 32 mm diameter using a press setup. The holding time for compaction was set to 2 min for all the samples. The pores were created after the water leaching step, where the compacted green sample was soaked in water. The entire process of foam formation is presented graphically as shown in Figure 1.

Initially, the number of aluminium foams was prepared at various compaction pressures in a range of 50 KN to 300 KN to determine the preferred pressure. The foams produced at 50 KN, 150 KN and 300 KN are presented as shown in Figure 2. It was observed that at compaction of 50 KN, the green strength was not adequate, and as a result, severe spalling was observed as shown in Figure 2(a). While increasing compaction pressure to 150 KN, the foam was observed in good condition with sufficient bonding in the green state as shown in Figure 2(b). The pores were also found in uniform shape and size at the same pressure. Further, the increase in compaction up to 300 KN resulted in strong bonding without any presence of spalling as shown in Figure 2(c). However, extremely close packing at the same pressure resulted in to formation of cracks and also water penetration in the sample for leaching was not satisfactory. As a result, the sample failed to form the necessary pores for foams.

Similarly, a number of samples were leached at a temperature range of 50°C to 150°C to determine the preferred leaching temperature. The samples leached out at temperatures of 50°C, 100°C and 150°C are presented in Figure 3. At a leaching temperature of 50°C, the process was slow and time-consuming, which led to softening of the sample due to loss in green strength and ultimately, spalling was observed as shown in Figure 3(a). Whereas at 100°C temperature, samples with uniform pore formation as shown in Figure 3(b) were observed, that indicates effective leaching out of carbamide particles. Again at 150°C, the leaching occurred fast due to rapid dissolution of carbamide. However, the process becomes

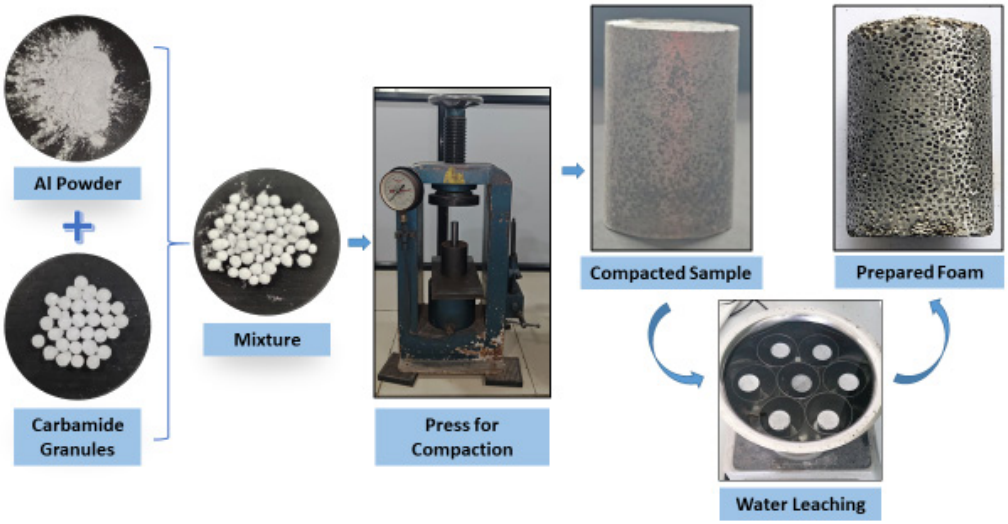


Figure 1. Processing steps of aluminium foam fabrication.

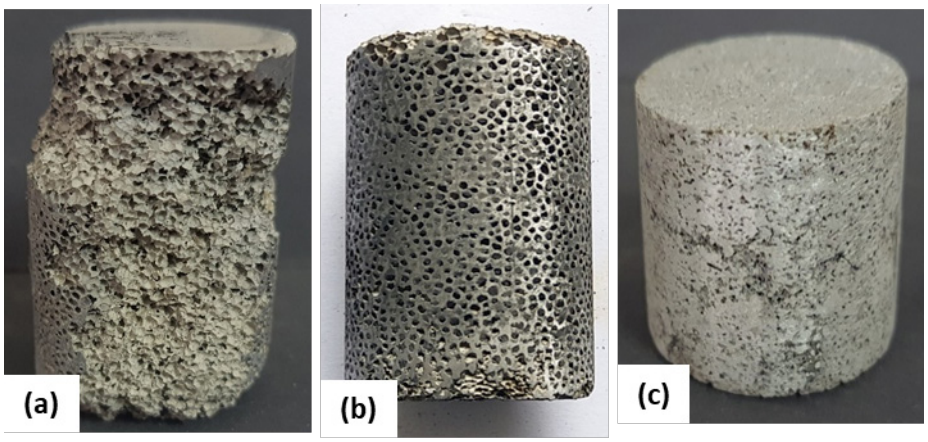


Figure 2. Foams produced at compaction pressure **(a)** 50 KN, **(b)** 150 KN, **(c)** 300KN.

so fast that bubble formations start and cause the sample surface to be damaged with abrupt spalling as shown in Figure 3(c). This is happening because of the evaporation of carbamide, as the melting point of carbamide is 135°C.

From these initial experiments, the compaction pressure and leaching temperature were determined as 100, 150 and 200 KN and 80, 100 and 120°C, respectively, for the subsequent experiments. The leaching time was selected as 50, 70 and 90 min, respectively. Considering a full factorial for the selected three process parameters and three levels, a total of 27 foam samples were prepared as shown in Figure 4.

To assess the effectiveness of set parameters on leaching, the depth to which the formed pores are visible will be calculated. This can also be attributed to the depth of water penetration to dissolve carbamide successfully. These depths can be measured along with compaction direction (height direction – h) and perpendicular to the centre axis (radial direction - d) as shown in Figure 5. These depths can be denoted as "Y" and "X", respectively.

3.0 Results and Discussion

The penetration depth was measured as stated in the previous section, and radial depth was considered for the study on the effect of leaching parameters, *i.e.*, compaction pressure, leaching temperature and leaching time on depth of penetration. The powder mixture gets compacted tightly in the axial direction as the pressure exerted by the punch is direct as compared to the radial

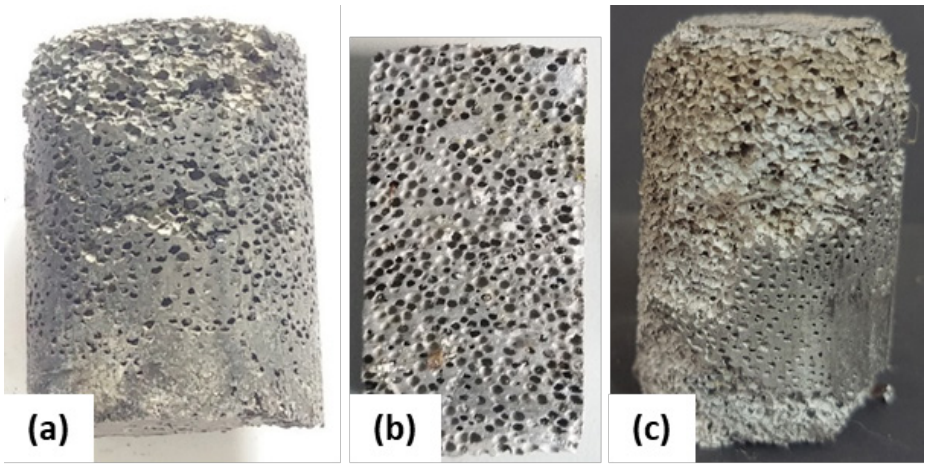


Figure 3. Foams produced at leaching temperatures **(a)** 50°C, **(b)** 100°C, **(c)** 150°C.

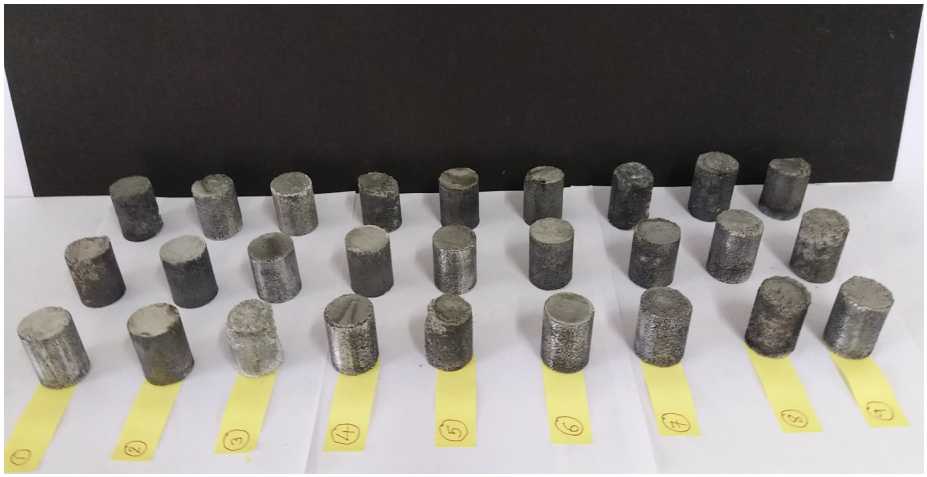


Figure 4.. total of 27 foam samples were prepared for selected parameters.

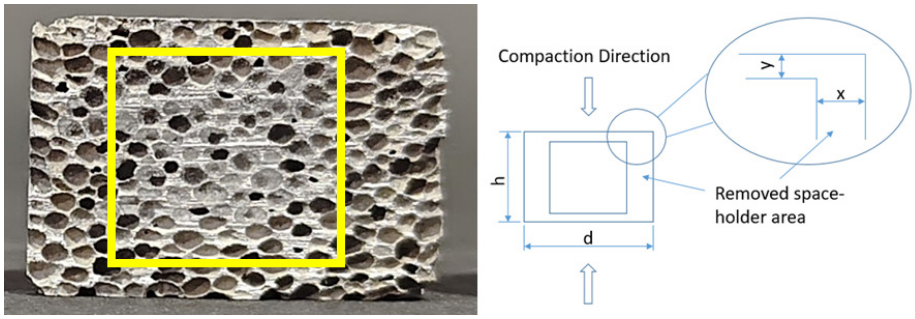


Figure 5. Water penetration in compaction direction (height direction – h) and perpendicular to the centre axis (radial direction – d).

peripheral direction. As a result, in the radial direction, compaction is not as close to the axial direction, and the strength of the sample wall is also less. So, water enters easily from the peripheral of the sample and leaches out carbamide fast compared to the axial direction during the water leaching process. These are the reasons for selecting radial depth for the comparison of the leaching penetration study. The regression analysis was carried out

considering all 27 samples where the R-sq value is >80% and the pvalue is <0.05, resulting obtained a confidence level of 95%. From the adjusted mean square percentage contribution of 10.37%, 8.99% and 80.48% were identified for parameters compaction pressure, leaching temperature and leaching time, respectively. It indicates that the leaching time has having maximum influence on the depth of penetration.

The graphs are plotted, indicating the effect of leaching parameters on the depth of penetration as shown in Figure 6. It can be seen that with an increase in leaching temperature and time, the depth of penetration increases. In contrast, an increase in compaction pressure hinders the leaching depth. It can also be observed from the bar chart presented in Figure 7 that with the increase in compaction pressure, the depth of penetration is decreasing at constant leaching temperature *i.e.* 80°C, and the time required to leach out the required depth is also increasing. The reason can be attributed to greater resistance to water penetration through the close-packed particles at higher compaction pressure. However, samples prepared with lower compaction pressure did not exhibit good integrity as softening and spalling were observed due to a lack of strength, irrespective of leaching temperature, as shown in Figure 8. Samples prepared with

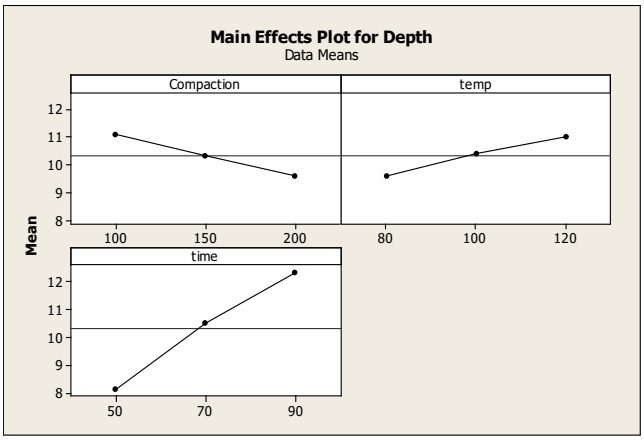


Figure 6. Plots indicating the effect of compaction pressure, leaching temperature and leaching time on penetration depth.

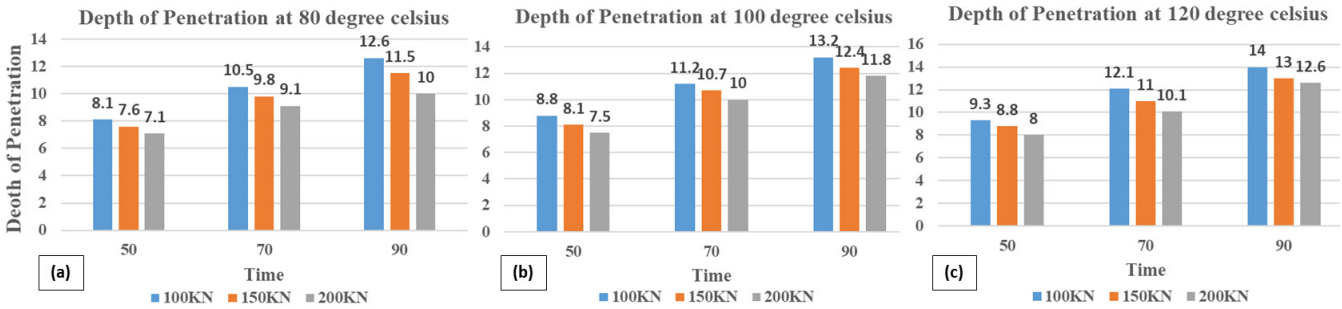


Figure 7. Comparative charts presenting the effect of compaction pressure and leaching time on depth of penetration at leaching temperatures of **(a)** 80°C, **(b)** 100°C, **(c)** 120°C.

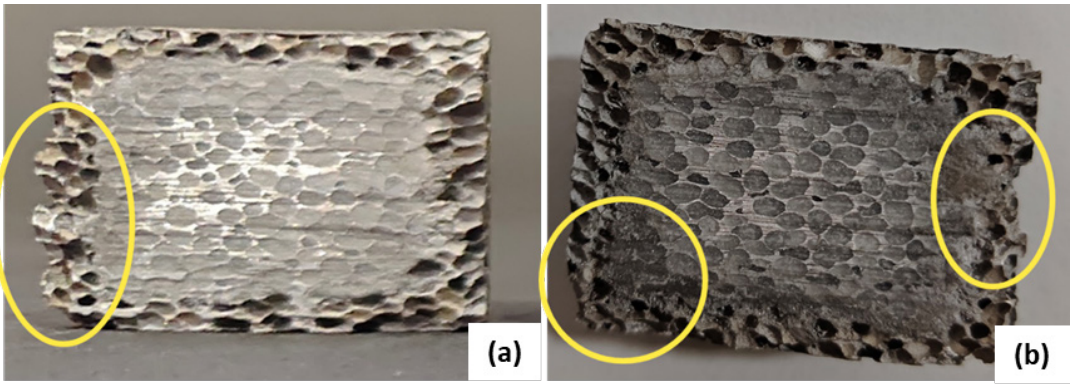


Figure 8. Foams prepared at 100KN compaction and leaching time of 50 min, indicating **(a)** softening effect at 80°C, **(b)** spalling effect at 120°C.

100 N compaction pressure and 80°C temperature get softened from the edges as shown in Figure 8(a). When the cross section was manually cut, the softening of samples was verified, where edges and overall shape were distorted. On the other hand, increasing the temperature to 120°C causes the carbamide to dissolve quickly at the same compaction pressure. As seen in Figure 8(b), the leaching at high temperatures, which creates bubbles from water vapour and bursts, and the sample with lower compaction of 100KN shows loss in green strength, which has caused surface damage and spalling. Therefore, samples should be compacted at higher pressure, though it inhibits water penetration to leach out carbamide.

Now, samples were prepared with a compaction pressure of 200 KN and water leaching temperature of 100°C, as higher temperature creates problems. These samples required a longer time to be leached out at this temperature due to the close bonding of the metal powder and carbamide at higher compaction pressure. The samples leached for 50, 70 and 90 min at 100°C are presented in Figure 9. It is observed that at the 50, 70 and 90 min time, the achieved depth of penetration is 7.5, 10 and 11.8 mm, respectively. Again, intense submergence of the sample in water also raises the problem of softening. In addition, the pore shapes also get distorted from a circular shape to an elliptical shape due to higher compaction, which also influences the mechanical properties of foams. In the next attempt, the samples were compacted with an intermediate pressure of 150 N and leached for 50, 70 and 90 min at 100°C. It was observed that successful leaching was observed creating pores to a greater depth at a leaching time of 90 min, as shown in Figure 10. The samples with 150 KN and 100°C have achieved depths of 8.1, 10.7 and 12.4 mm for leaching times of 50, 70, and 90 min, respectively. A significant improvement in water penetration can be observed in the case of compaction with 150 KN as compared to 200 KN up to a leaching time of 90 min.

In summary, leaching at 80°C takes a longer time and softens the sample, whereas 120°C temperature speeds up dissolution, but that could start spalling. As demonstrated in Figure 11, the edges softened after a lengthy 150 min leaching period for the compaction of 200 KN and temperature of 120°C.

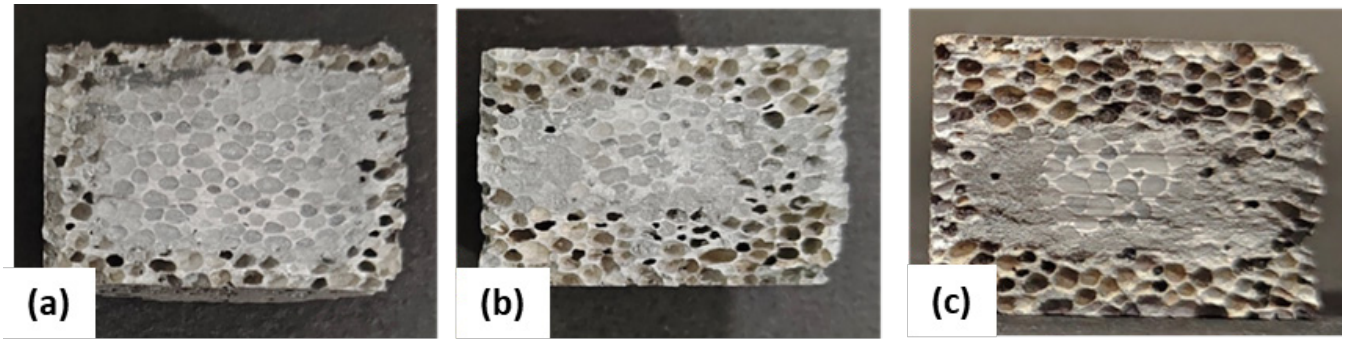


Figure 9. Foams prepared at 200 KN compaction, 100°C leaching temperature and leaching time of **(a)** 50, **(b)** 70, **(c)** 90 min.

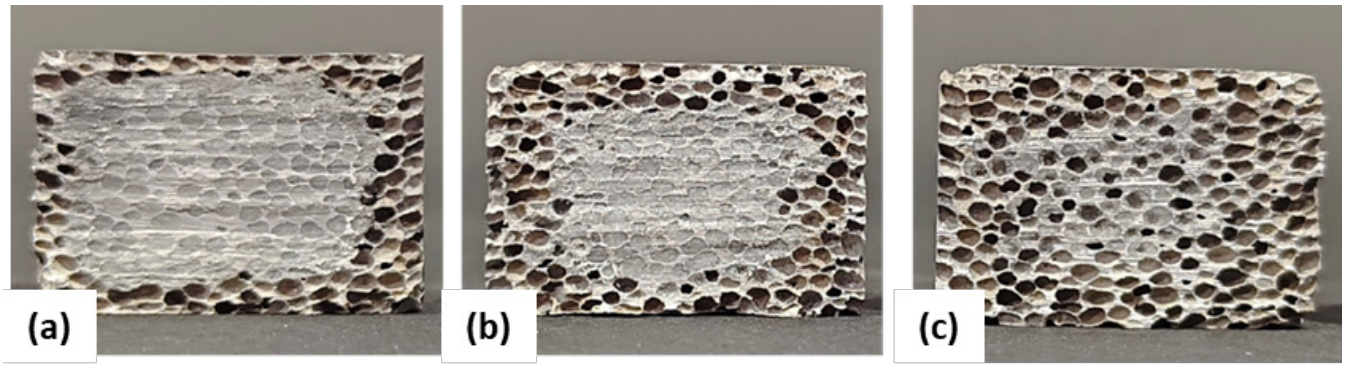


Figure 10. Foams prepared at 150 KN compaction, 100°C leaching temperature and leaching time of **(a)** 50, **(b)** 70, **(c)** 90 min.

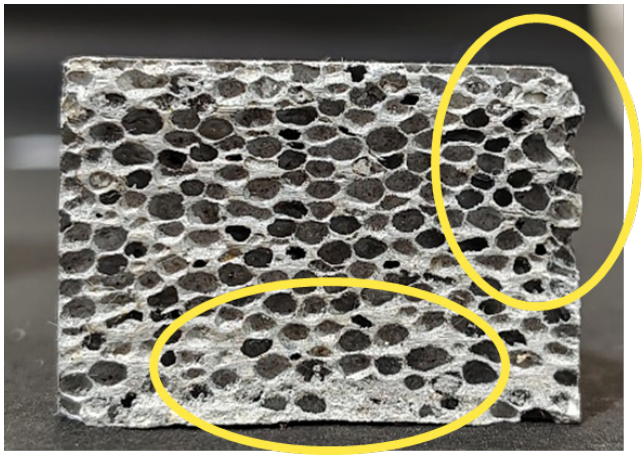


Figure 11. Softening of foam sample prepared at 200 KN compaction, 120°C temperature and 150 min leaching time.

Based on the observation discussed, the preferable range of process parameters was determined as compaction pressure of 150 KN to 200 KN, leaching temperature of 100°C to 120°C for leaching time of 90 to 120 min for successful leaching without any damage. In the next set of experiments, these determined parameters were used to produce foams with different space holder amounts, i.e., 60 wt.% and 80 wt.%, as shown in Table 1.

The samples prepared with 200 KN compaction pressure and leaching were carried out at 120°C for 150 min. In both cases, complete penetration was achieved as shown in Figure 12.

The sample with 60 wt.% space holder has no sign of spalling, while the sample with 80 wt.% space holder has faced more damage. During water leaching, space holders are removed by dissolving into the water that comes into contact through tiny channels. The size of these channels completely relies on the orientation of space holders and the packing density of particles. During the compaction stage, the mixture is squeezed in an enclosed space, which determines the location of space holders. During compaction at high pressure, the powder and space holder mixture is being packed more closely together. Thus, the possibility increases for space holders to occupy locations nearer to one another. As the samples submerge in water, urea begins to dissolve upon contact with it. Water attempts to penetrate the sample by forming extremely small channels. Urea particles that are situated within the

Table 1. Water penetration depth at 130 and 150 min indicates sample conditions

Compaction Pressure (KN)	Temperature (Celsius)	Time (minutes)	Carbamide Wt%	Depth (mm)	Remark
200	120°	130	60%	14	
		150	60%	15.5	Good condition
200	120°	130	80%	15.1	
		150	80%	16	Deformation and spalling

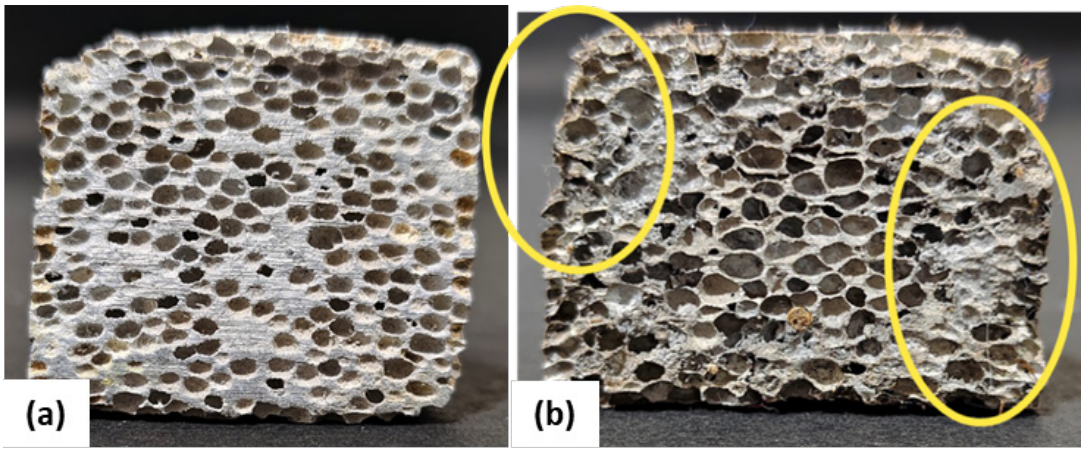


Figure 12. Foams prepared at 200KN compaction, 120°C temperature, and 150 min leaching time with carbamide weightage of **(a)** 60%, **(b)** 80%.

samples will leach out through these paths. The higher space holder amount (80 wt.%) indicates that the space holder will be more closely packed together compared to the lower space holder amount (60 wt.%), which directly suggests that the cell wall thickness in the 80 wt.% sample is smaller than that in the 60 wt.% sample. As a result, the green strength in the case of 60 wt.% will be higher than compared of 80 wt.%. Figure 12(a) demonstrates that a complete depth leach-out was achieved after 150 min for the sample containing 60 wt.% carbamide, which remained intact. In contrast, the sample with 80 wt.% carbamide faced severe spalling as shown in Figure 12(b).

In summary, out of the samples with 60 wt.%, 70 wt.%, and 80 wt.% carbamide, the samples with 60 wt.% and 70 wt.% exhibited more promising results in the compaction range of 150 KN to 200 KN, using water leaching temperatures of 100°C and 120°C.

4.0 Conclusion

Aluminium metal foams have been successfully manufactured using the powder metallurgy method and carbamide as a space holder. In this study, the primary focus was to understand the green sample behaviour during water leaching and its effect on foam quality. The following key conclusions have been derived from the present work.

(i) It was observed that it is very tough to control the space holder's location in a sample to generate uniform density, wall thickness and porosity because of the high probability of randomised orientation.

(ii) Compaction pressure can influence the water penetration inside the foam sample. Lower compaction induces the spalling, and higher compaction promotes the cracks as well.

(iii) To increase the speed of dissolution and achieve more depth, an increase in water temperature has been observed to be helpful. Lower water temperature needs more time for complete leaching of carbamide compared to high temperature at the same compaction pressure and carbamide weightage. A longer time of sample inside water deteriorates the green strength and promotes the spalling. While lower time is observed to be insufficient for complete leaching out of carbamide.

(iv) The change in wt.% of space holder also affected the spalling for constant compaction pressure, leaching time and water temperature. Foams with a higher amount of carbamide have been observed with lower green strength due to thinner cell walls, while lower amounts have higher cell wall thickness, which increases the time for full leach out.

From the overall study, it can be concluded that compaction pressure between 150KN to 200KN, amount of space holder between 60 wt.% to 70 wt.%, and water leaching temperature of 100 to 120°C are found promising to produce aluminium foams with inherent quality.

5.0 References

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