Study of internal porous structure formation of the powder metallurgically prepared aluminium foam

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Abstract. The internal pore wall structure formation and density play an important role in improving the mechanical and thermal properties of the closed-cell aluminium foams. The present research work aims to investigate the internal structure formation of the aluminium foam prepared by powder metallurgy and the uniformity of the distribution of the pores when the minimum amount of TiH₂ is added. The foamable precursor of two different aluminium alloys (Al-1050 and A5083) is produced with a TiH₂ gaseous agent of 0.05 wt.%. The parameters analysed include the density, pore wall formations, pore, and metal density distribution inside the structure with the help of X-ray tomography. Furthermore, the image-processing technique has been adopted to produce the 3D surrogate model of the foam for visual inspection and analysis. The obtained results show the importance of the amount of TiH₂ addition and of the foaming furnace temperature in deciding the internal porous structure formation. Further, the pore morphology of lower porosity foams (in the range of 30-40 % porosity) of the two alloys produced at 690 ℃ furnace temperature is investigated with the help of developed surrogate models. The presence of micropores and uniformity of the distribution of pores found brings the idea of choosing the optimized structure of foam for thermal energy storage systems associated with PCM.

1. Introduction
Metallic foams are lightweight cellular structures with a high percentage of porous spaces at macroscopic and microscopic levels. They can be classified as open or closed cell foams according to the manufacturing process. The behaviour of the porous structure is not the same as that of the original base metal, which helps for applying them under thermal and mechanical loading. Foam density and the composition of the alloy used are the main parameters for deciding the foam quality. In addition, foam structure plays a significant role in governing its quality, which is hard to control. Several processing parameters, such as precursor compaction condition, alloy composition, pre-treatment of the blowing agent, foaming temperature profile, etc., play a significant role in the foaming process [1]. The structure and foam quality change based on the change in those parameters.

This work systematically studied the influence of a small amount of foaming agent addition during the foaming of Al-1050 (99.7 %) and A5083 - Al-1050 (99.7 %) + 20 wt.% AlMg5 on pores size, density, and the porous structure of the foam. The foaming agent chosen for this study is 0.05 wt.% TiH₂ for achieving higher density foams [2]. X-ray tomographic characterization is made on the produced foam. The obtained X-ray slice images are used to reconstruct the 3D model of the actual foam sample by
image-processing technique. The reconstruction of the model is performed with the free open-source 3D slicer software. Software such as Ansys workbench and Meshmixer is utilized to construct and analyse the pore distribution of the surrogate models. In addition, pore analysis on the obtained images is performed with the free open-source ImageJ software.

2. Experimental

2.1. Materials and foamed samples

The aluminium foam prepared for this study is based on the PM route by following the procedure mentioned in the literature [3]. Aluminium powder with a particle size of < 63 μm, supplied by KERAMETAL, Ltd., Slovakia, is used to prepare the samples [2]. The manufacturer reports that the aluminium powder consists of 99.7 wt.% Al, 0.11 wt.% Fe and 0.06 wt.% Si [2]. The pre-alloyed AlMg5 powder (99.7 % purity with particle size < 400 μm) is supplied by Mepura (Austria). The foaming agent TiH2 is supplied by Chemetall (Germany), which belongs to grade U with particle size ≤ 45 μm and the average particle size d = 5 ± 1 μm [3]. The 0.05 wt.% foaming agent is admixed with each powder mixture.

Two types of precursors prepared from the following powder mixtures were used: The first one is based on Al-1050 (99.7 %) alloy, and the second one has a composition of A5083 alloy. The second precursor is prepared from the mixture of Al-1050 (99.7 %) + 20 wt.% AlMg5 powders. After mixing the powder, cold isostatic compaction is performed under the pressure of 200 MPa. The compacted billets of dimension 30 mm (approx.) are produced and hot extruded. The extrusion temperature is maintained at 450 °C. The extrusion ratio is kept at 28:1 [2]. The rectangular profile of cross-section 2 × 20 mm² is produced from the billets. The extruded profile is cut into the dimensions of 20 × 40 × 2 mm³. Four pieces of the precursor profile are placed into the steel mould, and the aluminium foam of dimension 40 × 40 × 5 mm³ is foamed by introducing the steel mould into an electric resistance furnace. Foaming is performed under various furnace temperatures in the range of 730 °C – 690 °C. The precursor inside the steel mould is overheated when the furnace temperature is maintained at 730 °C and 720 °C. It leads to the poor quality of aluminium foams. Better foaming has been found by maintaining the furnace temperature in the range of 710 °C and 690 °C. The corresponding time which is taken for the foaming is recorded. Totally fourteen samples are produced in the density range of 0.97-2.24 g/cm³. The foamed samples show good reproducibility. Six samples at each furnace temperature with a density of 1.7 – 1.8 g/cm³ have been chosen for the present study.

In general, the foam porosity is calculated by the given equations below [4]:

$$\text{Porosity} = 1 - \frac{\rho_s}{\rho_b}$$ \hspace{1cm} (1)

$$\text{Porosity} = \frac{V_p}{V_T}$$ \hspace{1cm} (2)

where $\rho_s$, $\rho_b$ and $\rho_r$ are the volumetric, bulk, and relative density, $V_p$ is the volume of the pores, $V_T$ is the total volume of solid and pores [2]. The foaming conditions and structural parameters of the foamed samples are presented in Table 1. The porosity calculated is based on equation (1).

The aluminium oxide layer of the nano-scale is covered at the foamable parts that gives them a metallic appearance. Among the six foamed samples, the higher porosity samples such as X3 and X6 samples are studied in detail in this work. Both the samples are of the same porosity level (approx. 36 %) and foamed at 690 °C furnace temperature with good reproducibility. Moreover, it helps to evaluate the structure of two different aluminium alloy foams having the same porosity. The study on their internal structure formation could help future studies on PCMs based thermal energy storage applications where high-density foam is desirable [2].

**Table 1. Foaming conditions and the structural parameters of the foam (TiH2-0.05 wt.%).**

| Aluminium alloy | Sample | Precursor weight, (g) | Furnace temperatu | Foaming time, (s) | Volumetric density of the | Porosity, (%) |
|-----------------|--------|-----------------------|-------------------|------------------|--------------------------|---------------|
| Al-1050         | 1      |                       | 710 °C            | 90               | 1.7                      |               |
| Al-1050         | 2      |                       | 690 °C            | 120              | 1.8                      |               |
| Al-1050 + 20%   | 3      |                       | 710 °C            | 150              | 1.7                      |               |
| Al-1050 + 20%   | 4      |                       | 690 °C            | 180              | 1.8                      |               |

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2.2. X-ray tomography characterization

There are various visualization, segmentation, and morphometry methods employed to investigate the internal structure of foams. The inner structure of the foamed samples is observed with X-ray tomography images. The characterization is performed with X-ray Phoenix/X-ray Nonatom 180. CXZ device (Manufacturer—G & E, Institute of Materials and Machine Mechanics—Slovak Academy of Sciences, Bratislava, Slovakia) [2]. It is one of the effective methods of characterizing the distribution of metals and pores. The details about the tortuosity of the solid and gas phases, the density of the metal distribution, and pores distribution can be easily found with the X-ray slice images [5]. The X-ray tomography was made on the samples X3 and X6, foamed at 690 °C. The number of slice images made along the x-axis, the y-axis, the z-axis direction, and the corresponding foam at 96 × 96 dpi resolution is shown in Table 2.

Table 2. Number of X-ray images and their corresponding resolution.

| Plane | x-axis | y-axis | z-axis |
|-------|--------|--------|--------|
| X3 images | 1613 | 246 | 1637 |
| Resolution (mm) | 148 × 486 | 464 × 486 | 464 × 148 |
| X6 images | 1620 | 242 | 1626 |
| Resolution (mm) | 146 × 489 | 467 × 489 | 467 × 146 |

Figure 1. View of X-ray tomography images of the sample X3 along (a) x-axis, (b) y-axis, (c) z-axis, and (d) full view of the actual foamed sample X3 (36.25 % porosity).

The slice image view in three directions and the 3D view of the sample X3 position, which is placed for X-ray tomography imaging, are shown in figure 1.
2.3. Creation of surrogate model using image-processing technique

The X-ray slice images of the experimentally developed sample are used to create the surrogate model of the foam. A vast number of points in the numerical point cloud can be generated with the X-ray images that develop the need for computer resources [6]. However, it leads to a high computation time. The surrogate model of dimensions $7.5 \times 7.5 \times 5 \text{ mm}^3$ is developed to simplify the computation time for investigating the structure with numerical modelling. It can be achieved by selecting the 330 slice images in the X direction, 330 slice images in the Z direction, and 220 images in the Y direction. The slice images obtained from the foam samples X3 and X6 are investigated in this section. The images selected for creating the model are cropped from the original slice images with the help of 3D slicer software. The X-ray slice images are converted into the "*.stl file" by thresholding the images in the segment editor tool. The scale factor is set to 1, and the default smoothing factor is maintained at 0.5. The thickness of the actual foam sample (5 mm) is kept constant. The accuracy of the developed surrogate model depends mainly on the space between slicing planes kept for producing the X-ray slice images [7]. It sets the minimum observable pore size and defects, especially on the nano-scale. The creation of the 3D model from the cropped slice images in 3D slicer software is shown in Figure 2.

![Actual foam slice image, cropped slice image view, and developed 3D surrogate model](image)

**Figure 2.** 3D surrogate model of the sample "X6" developed from the cropped slice image in 3D slicer software, (a) 78th slice image view made along Z direction, the cropped slice image view along, (b) Z-direction (Front view), and (c) X-direction (Right view), (d) Y-direction (Bottom view), and (e) generated 3D surrogate model of the actual foam in segment editor module.
The surrogate model of X3-S and X6-S is developed in 3D slicer software, and the same has been compared with the actual polished foam sample, shown in figure 3. The porosity of the developed 3D surrogate model is calculated with equation (2). The volume of the solid part is found by the stability tool of the Meshmixer software. The volume of the pores is the difference between the volume of the developed model and the solid model of the same dimensions. The structural parameters of the developed model are presented in Table 3.

| Surrogate model | Volume, \(V_s\) (mm\(^3\)) | Total surface area, \(A_s\) (mm\(^2\)) | Porosity, (%) |
|-----------------|-----------------------------|---------------------------------|--------------|
| X3-S            | 186.794                     | 513.074                         | 33.58        |
| X6-S            | 194.336                     | 838.072                         | 30.9         |

The porosity values obtained based on equations (1) and (2) are different. Equation (1) helps to calculate the foam porosity when density is known. Equation (2) is used to calculate the surrogate model porosity developed at the particular area of the foam shown in Figure 2 when the volume of the model is known.

### 3. Results and discussion

#### 3.1. Pore space measurement

The X-ray tomography characterization shows pores formed inside the closed-cell aluminium foams are spherical, with interconnected pore walls having microcracks [8]. The resulting X-ray images have shown that the pores are of indefinite shape and the pore wall ruptures are visible at a particular point of the foam. Moreover, the developed 3D model through the image-processing technique gives a detailed visualization of the pore morphology, which is hard to visualize only with the slice images. The 3D surrogate models help to find the structural parameters of the inner pores. It can be verified by measuring the pore wall distance of the X6-S surrogate model at a particular point of the pores in both the slice image (with the help of ImageJ software) and the 3D model (with the help of the measurement tool of the Ansys workbench Spaceclaim module). The measured pore wall distance is shown in Figure 4 below.
3.2. Pores distribution

The X3-S and X6-S models are imported into the Meshmixer software to study their pores distribution. Both the surrogate models are reconstructed and convert into the solid model with the help of the "Make solid" tool. The cell size of the ".stl" file is kept as 1 mm. The reconstructed model is in the form of both solid and mesh files, which gives a clear view of pore distribution inside the structure shown in Figure 5.

This approach could be helpful to understand the pores distribution in microscale and the depth of pores which is hard to visualize with the solid surrogate model. By comparing both the models in this way, it has been found that the presence of micropores is higher for lower porosity foams [2]. Furthermore, by X-ray tomography characterization, the pore distribution's uniformity is higher for the X3 sample made of pure aluminium alloy. Moreover, the X-ray slice images of the samples foamed at lower foaming temperature (690 °C) lead to an increase in uniform pore distribution inside the structure.

Overall, the methodology used seems very useful to study the internal pore and pore wall structure formation and the defects in micro and macroscale. Furthermore, it gives the idea of using the developed surrogate models to conduct the numerical heat transfer study to analyse the heat conduction and heat storage capacity when PCM is impregnated into the aluminium foam.
4. Conclusion

The research study helps to understand the efficiency of the X-ray tomographic characterization and image-processing technique to evaluate the pores distribution in the internal structure of high-density foams produced through the PM route. The foamable precursors of two different aluminium alloys such as Al-1050 (99.7%) and A5083-Al-1050 (99.7%) + 20 wt.% AlMg5, mixed with 0.05 wt.% TiH₂ are utilized for the study. In addition, the quality of foamed aluminium samples by keeping the furnace temperature ranging from 730 °C to 690 °C is investigated. The quality of the obtained foams is better at 690 °C than the produced foam at 730 °C. However, higher furnace temperature leads to the poor quality of foam production due to the overheating of precursor inside the mould.

The image-processing technique is adopted, which helps to develop the surrogate model of real foams (30-40 % of porosity) obtained from both the alloys foamed at 690 °C. The investigation made on the slice images and developed surrogate model shows a massive number of micropores, which is the result of the addition of 0.05 % TiH₂. The pore space measurement at the definite point of the slice image and 3D model gives a very good agreement. It helps to understand the efficiency of the proposed approach in finding pore morphology and its structural parameters.

Further, the pores' distribution uniformity is analysed, which is higher for the pure aluminium alloy foamed at 690 °C than for A5083 foam. The obtained results help for further research on utilizing high density aluminium foam for mechanical and thermal applications.

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