Manufacturing of Open-Cell Metal Foams by the Sponge Replication Technique

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Abstract. Different manufacturing techniques for open-cell metal foams, such as investment casting and space holder casting, are compared with the sponge replication technique that is originally used for the preparation of ceramic foams. Processing of aluminum foams by the sponge replication technique has been reviewed. Based on the results obtained for aluminum open-cell foams, the feasibility of copper foam manufacturing with the sponge replica technique using an aqueous copper dispersion was demonstrated.

1 Introduction

Wood or bones are one of the common cellular materials in nature. Highly porous materials including metal and ceramic foams possess a combination of properties such as high stiffness-to-weight ratio, low specific weight, high energy and acoustic absorption [1–3]. Due to these combinations of properties porous materials have found application in nearly every sector of technology as aerospace industry (noise reduction), architecture (light-weight constructions and thermal/sound insulation materials), automotive industry (engine brackets, gear-wheel with an anti-vibration layer of aluminium foam), filters, heat exchangers, biomedical prostheses (bone implant material) and vibration dampers [1–6].

There are different types of metal foams: open and closed cells metal foams. These metal foams need different manufacturing processes and are characterized by their different properties. Closed-cell metal foams are used predominantly as constructional materials (automotive industry, architecture, and more). At the same time, open-cell metal foams have received a great deal of attention as functional materials such as heat exchanges for adsorption-driven heating/cooling applications [7], as catalyst support materials [5], pressure regulators, fuel cell components [8], battery components [9, 10] and filters, due to combination of properties such as thermal/electric conductivity, high specific surface area and liquid/gas permeability [11, 12]. These types of metal foams are used as a template for deposition or coating with active materials to enable chemical reactions, adsorption properties, mechanical stability or electrical conductivity [4, 5, 11]. Open cell metal foams for the above mentioned application are made from aluminium [9, 11, 13], nickel [8, 14], copper [10, 15] and titanium [16] or alloys such as stainless steel [17], Ni-Fe [18], Ni-Cr and Ni-Fe-Cr [19].

The most common conventional methods to manufacture open-cell metal foams are investment casting, casting around hollow spheres, and space holder or infiltration casting [20–22]. There are other methods such as layer by layer electro deposition on a polymer pre-form [19] or direct foaming [13], etc to obtained open-cell metal foams.
2 Review of manufacturing techniques for open cell metal foams

2.1 Manufacturing of open-cell metal foams by investment casting

The processing of open-cell metal foams consist of four main manufacturing steps, which are schematically illustrated in figure.1. On the first step, a reticulated polyurethane foam (PU) template is filled under pressure with a mould slurry of heat resistant material such as ceramic or gypsum (plaster) suspension. After drying the mould material, the PU foam is removed (burned out) and molten metal is filled into these open voids. The last steps are removal of the mould material with high pressure water or dissolving it with acids or leaching. The obtained metal foam is a replica of the shape of the original PU foam.

![Figure 1. Schematic illustration of the manufacturing of open-cell metal foams by investment casting.](image)

The advantage of the investment casting is the manufacturing of open-cell foams from different metals and a wide spectrum of desired cell sizes, relative density and porosity, which are determined by the polymer foam (porosity may range from 80% to 97%). However the problem of this method is no uniform filling of the mold with molten metal; therefore, the filling of the mold is carried out under high pressure in a gravity field and preheating of the mold [23–25]. In ref. [23], it was shown that changing the gravity coefficient from 300 to 900 enhanced the structural integrity of pores, surface morphology, and thickness of the cell struts of aluminum foams. Also, authors successfully manufactured open-cell metal foams with different cell sizes (from 4.5 mm to 0.1 mm) in a super-gravity field with the gravity coefficient of 500. However, it was found that removing the plaster materials was not effective for the foams with the cell sizes of 0.1 mm [23].

As a result, the manufacturing of large open-cell metal foams is problematic due to the difficulty of controlling the uniform heating of the mold form and its filling with liquid metals that has considerable influence on the macrostructure and phase distribution of metal alloys [25]. Also removing the heat resistant mold is not only complex but also can damage the foam structure. In addition, foams manufactured this way are characterized by low yield and have high prices [26].

2.2 Manufacturing of open-cell metal foams by casting with space holders

Manufacturing of open-cell metal foams by the space holder casting is a technique in which, instead of PU template, different space holder particles such as sodium chloride, sugar, carbonates or sand are used. The manufacturing route is shown in figure 2. The packing of these space holders are infiltrated with a liquid metal. After solidification space holders are removed by acids or leaching in solvents [27].
A high surface tension of liquid metals can be problematic during wetting of the space holders, which leads to an incomplete filling of the voids between the space holder particles. Therefore, for the uniform casting of space-holders with molten metal high pressure melt infiltration, vacuum and pre-heating of space holders are required to avoid solidification of the molten metal until complete filling the pre-form with liquid metal. It is possible to manufacture aluminium, magnesium, zinc, lead, tin foams, etc by space holder casting [27]. The main advantages of this manufacturing way are fabricating foams with a desired geometry and controllable pore size which depends on the size of space holders [29, 30]. At the same time, the maximum value of porosity is limited by the size of space holders; it is laborious to achieved foams with porosity >90%. The shape of cells in these foams is not uniform and repeats the shape of space holders.

Numerous research is focused on optimizing the manufacturing parameters to obtain stable foams with the desired functional properties. In ref. [30], the influence of vacuum, space holder size and compacting pressure of space holders on the cell structure and compressive strength of foams was investigated. Also, a super-gravity field was used to fill a pre-form by molten metal uniformly (figure 3) [29]. It is seen that the metal had completely filled the pre-form at higher centrifugal pressure.

**Figure 2.** Schematic illustration of manufacturing of open-cell metal foams by space holder casting.

2.3 Manufacturing of open-cell foams by the sponge replication technique

The sponge replication technique was initially used for the manufacturing of ceramic foams [31], which consists of three manufacturing steps (figure 4). The first step is the coating of a PU template with an aqueous suspension of metal or ceramic powder mixed with a binder. The second stage is burning out the PU template and the binder. The last step is sintering or heat treatment of the foams.

**Figure 3.** Typical cross-section images of aluminum foams with a pore size of 600 μm fabricated with different centrifugal pressures: (a) 24 kPa and (b) 32 kPa [29].
The foams prepared by the sponge replication technique have controllable pore size (90%) which depends on the pore size of the initial PU template. The foams are characterized by a high surface-area-to-volume ratio; therefore, this manufacturing way is attractive for preparing metal foams used in chemical engineering/industrial chemistry (catalytic reactions, supports for different coatings, filters, etc) [20]. One of the limitations of the sponge replication technique for metal foam manufacturing is the presence of thin oxide layers on the metal powders used as starting materials; the oxide layers are often difficult to disrupt (depending in the metal), and this leading to an inhomogeneous microstructure of the struts due to incomplete powder particle sintering. As a result, metal foams may have a low compression strength and low thermal/electric conductivity [32, 33]. As an example, a natural thin alumina film and high oxidation rates of aluminum particles are still problematic for sintering aluminium metal foams [34]. A possible solution to this problem is varying the sintering temperature and atmosphere [35], as well as various additives used to dissolve the metal oxide layer [36].

2.3.1 Current manufacturing of open-cell metal foams by sponge replication technique. The manufacturing of open-cell metal foams by sponge replication technique has been successfully applied for titanium [37–39], Ti6Al4V [40] and austenitic steel [41]. Open-cell aluminum foams made by sponge replication technique are attractive for catalytic application and for heat exchange systems. In ref. [42], the feasibility of the aluminum foam preparation by the sponge replication technique in air at 620 °C for 4 h and 7 h was demonstrated. The aluminum foams were stable and had a porosity between 94.4 % and 95.5 %. This work was more focused on determining the optimal composition of the metal suspension and demonstrating applicability of this manufacturing method for aluminium starting powder.

Investigations carried out by our research group [32] have shown that the shape and size of aluminum powders used for the manufacturing of foams influence the thermal consolidation process of the foam resulting in a variation of structure features. It was found that the foams obtained from spheroidal aluminium particles have a lower strut porosity, a lower amount of residual alumina, and a higher compressive strength in comparison with foams manufactured from flaky, irregularly shaped aluminum powder. Figure 5 shows cross sections of struts of the foams made from the flaky, irregularly shaped Aldrich powder and spheroidal-shaped Ecka powder after heat treatment at 750 C for 3 h [32].
Figure 5. SEM images of cut struts of the foams made from the (a,b) flaky, irregularly shaped Al powder and (c,d) spheroidal-shaped Al powder after heat treatment at 750 °C for 3 h [32].

The manufacturing of the open-cell aluminum foams from the spheroidal aluminum powder by a sponge replication technique was performed in ref. [33]. The thermal processing of aluminum foams at temperatures up to 800 °C in Ar reduced the strut porosity in comparison with the strut porosity of foams thermally processed in air. In addition, after thermal processing in Ar the Al metal foams were characterized by a lower amount of aluminum oxide, a higher thermal conductivity, and a higher compression strength due to a comparatively lower amount of aluminum oxide after thermal processing. In figure 6 Al foams are shown after thermal processing at different temperatures [33]. However, despite of the successful consolidation, the foams have porous struts resulting from incomplete thermal processing. This may be explained by a thin oxide shell around the starting aluminum particles and a steady disruption of the alumina shells during thermal processing. Therefore, for the further research it is necessary to focus on reduction of the alumina/oxygen content in the aluminum starting powders.

In general, the reticulated aluminum foams are stable, had high porosity and satisfying strength. Consequently, processing open-cell aluminum metal foams is possible to produce highly porous foams with a relatively large surface area by the sponge replication technique.

Figure 6. Al foams after thermal processing for 3 h in (a) air and Ar at 750 °C and (b) Ar at 750–900 °C [33].
Open-cell copper foams are attractive for advanced electronics, energy-related devices such as rechargeable batteries, electrochemical catalysts, heat exchangers, heat pumps, fuel cells, and super capacitors because of the combination of high thermal/electric conductivity and high surface area [10, 43, 44]. Thus, based on the results with aluminum open-cell foams, manufacturing open-cell metal foams by sponge replication technique may be applicable for processing copper foams. According to the above, this work aims to demonstrate the feasibility of copper foam manufacturing with the sponge replica technique with an aqueous copper-containing slurry.

3 Manufacturing of open cell copper foams

3.1 Specimen preparation

For the manufacturing of copper foams, copper powder was used, supplied by Ecka Granules (ECKA Kupfer CH UF 10, Ranshofen, Austria) with a purity of 99.81 wt.%. The average particle size was <10 µm. For the preparation of a coating slurry the copper powder was mixed with a 10.7 wt.% solution of a polyvinyl alcohol binder (1.2 wt%, Optapix PA 4G, Zschimmer and Schwarz Chemie GmbH, Lahnstein, Germany) in distilled water. The solid content of copper in the slurry was 82.8 wt.%. A planetary centrifugal mixer (THINKY Mixer ARE-250, THINKY Corp. Tokyo, Japan) was used for mixing the copper slurry at 2000 rpm for 6 min. As a PU template, an open-cell PU foam with a linear cell count of 20 ppi with a geometric size of 15 mm × 15 mm × 20 mm (Koepp Schaum GmbH, Oestrich Winkel, Germany) was used. A schematic illustration of the open-cell copper foam preparation by the sponge replication technique is shown in figure 7.

After dipping the PU template foam into the slurry the specimens were dried for 24 h at room temperature in air. The binder and PU burning out were carried out in air at 250 °C for 3 h and at 500 °C for 3 h in a circulating air furnace (KU 40/04/A, THERMCONCEPT Dr. Fischer GmbH, Bremen, Germany). The foams were thermally processed for 6 h in Ar atmosphere with 2% H₂ (99.999% purity) at 900 °C in a conventional tube furnace (alumina tube, HTRH 70-600/1800, Carbolite-Gero GmbH and Co. KG, Neuhausen, Germany).

![Figure 7. Schematic illustration of the open-cell copper foam preparation by the sponge replication technique.](image-url)

3.2 Characterization

The microstructure imaging of the thermally processed foams was carried out by a scanning electron microscope (SEM; FEI ESEM XL30 FEG, Hillsboro, OR/USA). For the determination of the phase composition X-ray diffraction analysis (XRD) conducted with an X’Pert Pro diffractometer (PANalytical GmbH, Kassel, Germany, Co Ka1/2 radiation, 2θ, 40–85°) with Bragg–Brentano geometry was applied. The total porosity was calculated from the geometric foam density and the skeletal density of the strut material ($V_{pores}/V_{foam}$) [45], considering that the density of the pure bulk
copper $\rho_{Cu}$ equals to 8.96 g·cm$^{-3}$. The quantification of the strut porosity ($V_{\text{strut pores}}/V_{\text{struts}}$) of copper foams was conducted by the Archimedes method with water according to the DIN EN 623-2:1993-11 standard procedure [46]. For the measurements of the compression strength a TIRAtest 2825 testing machine was used (TIRA GmbH, Schalkau, Germany) with a loading plate of 150 mm in diameter and the applied load 2 mm·min$^{-1}$.

3.3 Results

After thermal processing in Ar with 2% H$_2$ the copper foams were characterized by a volumetric shrinkage of ~56%. The copper foams possess a total porosity of ~93%, which includes material pores, cavities (hollow strut pores) and cell pores. The total strut porosity consisting of porosity of hollow struts and material pores amounts to ~44 %.

Figure 8 shows SEM images of cross-sections of copper foams thermally processed at 900 °C. It can be seen, that their struts are hollow and represent the shape of the PU template foam, despite the high degree of shrinkage. No molten copper beads outside and inside the foam struts were detected, as it was the case for the of Al foams. The microstructure of the foam struts is characterized by homogeneous (figure 8, top) and inhomogeneous regions with pores in the strut walls, which may result from incomplete thermal processing (figure 8, bottom).

The results of the XRD phase analysis (figure 9) indicated that there is only a pure copper phase present after thermal processing for 6 h at 900 °C in Ar with 2% H$_2$. No copper oxide and/or carbide phases were found.

![Figure 8. SEM images of copper foams thermally processed for 6 h at 900°C.](image1)

![Figure 9. X-ray diffraction patterns of copper foams thermally processed for 6 h at 900°C in Ar with 2% H$_2$.](image2)

Figure 10 shows a typical stress-strain curve of the copper foams after thermal processing. The stress-strain curves on the initial stage had an elastic deformation region with a linear increase of the stress from the strain; in a point where the stress-strain changed its behaviour, the yield strength $\sigma^*$ exceeded the strength of the cell struts, which is the onset of plasticity. Further increase of the stress is characterized by a long plateau region (plastic cell collapse) with a significant strain increasing. When the cells of the foams are compacted, the stress-strain curve increased drastically (densification region). The average yield strength value $\sigma^*$ of 10 stress-strain measurements was $\sigma^* = 0.38 \pm 0.06$ MPa.
Figure 10. Typical stress–strain curves of copper foams after compressive strength test of thermally processed for 6 h at 900 °C in Ar with 2% H₂.

4 Conclusion
In this work, investment casting and space holder techniques for the manufacturing of open-cell metal foams were reviewed and compared to the sponge replication technique. As material examples, aluminum and copper foams were prepared by using polyurethane as foam templates. It was shown, that, despite of oxide impurities in the case of aluminum, or a melting point of copper at 1085 °C, manufacturing of intact foam structures of these metals is possible. Both replica-manufactured foams possess a high porosity, a high specific surface area and hollow struts. While more research is necessary to reduce the oxide share of the aluminum foams, copper foam-processing in an Ar/H₂ gas mixture led to a plain/elemental copper material. In comparison with the aforementioned processes a number of processing steps can be economized, and the hollow struts may be used for further functionalization of these type of cellular materials.

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