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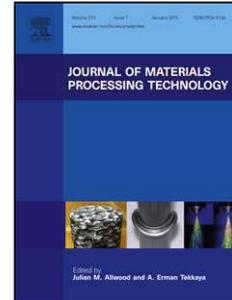


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A replication-casting device for manufacturing open-cell Mg foams

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ABSTRACT

The development of a replication casting device with the capability of manufacturing open-cell pure Mg and Mg alloys foams, with melting points lower than 950 °C is described. The device consists of three basic parts: a cylindrical reaction chamber, a valve system for controlling the vacuum and the gas injection, and a heating system. The purpose of the present design was to improve the existing laboratory-scale devices, making them simpler than those reported in the

literature, as well as to optimize the parameters (atmosphere, temperature, injection pressure, etc.) of the replication casting process. The proposed device has shown significant improvement over other devices reported, especially in terms of easiness of operation, adjustability and low cost of maintenance. The design of the device provides the ability to produce large foams free from defects such as large volumetric shrinkage and large concavities. The latter is minimized by the implementation of a perforated nozzle for gas impulsion. Examples of manufactured open-cell pure Mg and Mg-10 wt.% Al alloy foams are also presented. The design is shown in a conceptual format; however it could be modified to produce larger or smaller samples.

Keywords: Foams, Pure Mg, Replication casting, Device, Manufacture

1. Introduction

Porous metal structures, also known as metallic foams, have shown a combination of physical and mechanical properties such as high strength-to-weight ratio, high energy absorption capacity, large specific surface, high gas and liquid permeability and low thermal conductivity as has been reported by Baumgartner and Gers, 1999. As mentioned Banhart (2001), metallic foams with open-cells are mainly used in functional applications (heat exchangers, filters, catalysis supports), while the metallic foams with partially open and closed cells are used in structural applications (bio-medical implants, silencers, bearings, sound, energy absorbers, etc.).

As pointed by Asbhy et al. (2001), pure aluminum (Al) and Al based alloys foams have been widely manufactured by several techniques. In contrast, little attention has been paid to developing pure Mg and Mg alloys foams, due to the high production costs and the difficulty of processing them. Mg foams have been mainly studied for their functional properties such sound

and energy absorption capacity, excellent vibration reduction capacity. Wen et al. (2001) found that Mg foams were promising biomaterial for bone implants due to its open-cellular structure promote the in-growths of the new-bone tissues and the transport of the body fluids.

Some methods have been used to manufacture closed-cellular pure Mg and Mg alloys foams. Körner et al. (2004) proposed a method where combined high pressure casting using a thixomoulding machine in a cavity to add and disperse MgH_2 particles as foaming agent. Renger et al. (2005) obtained Mg foams using a technique called vacuum foaming method. Later, Dong-Hui et al. (2008) obtained a Mg foam by melt-foaming method, also known as blowing agent method, using $CaCO_2$ powders as foaming agent and a mixed of CO_2 and SF_6 atmosphere to protect Mg alloy from being ignited or oxidized during the process. On the other hand, the manufacture of the open-cell pure Mg and Mg alloys foams has been limited to the powder metallurgical method and replication casting process. Hao et al. (2009) obtained pure Mg foams by four stages powder metallurgy manufacturing process including mixing, compacting, dissolution and sintering. The space holding filler used was commercially carbamide particles with a rounded shape. Later, Aghion et al. (2014) produced MRI 201S Mg alloy foams using Ammonium Hydrogen Carbonate salt particles as a space holding filler. Both works used high purity argon atmosphere as a protective gas. In contrast, as (San Marchi and Mortensen, 2002) mentioned, the replication casting process offers the possibility to produce open-cell foams with high control of the topology. However, the manufacture of open-cell pure Mg foams by the replication casting method is still limited.

The replication casting process was first proposed by Polonsky et al. (1961). According to Polonsky this method is able to produce an interconnected open-cell structure by infiltrating the molten metal under pressure through an open pore preform (or negative of the foam) made from granular particles. This step is followed by the dissolution of the particles using an adequate

solvent. However, it was until 2001 when (San Marchi and Mortensen, 2001) retook this method to produce open-cell aluminum foam. The main parameters for the replication casting process are: 1) the infiltration pressure, 2) the melting temperature of the metal charge and 3) the design of the preform (material, size, solubility, etc.). The possibility of controlling these parameters with different mechanisms, the physic-chemical properties of the metal foams and the design of the preforms are factors that have led to develop a variety of devices.

The infiltration can be made by a mechanical mechanism or using gas pressure. Fabrizio et al. (2011) obtained an Al-Si-Mg foam using a hydraulic cylinder to generate the infiltration pressure. On the other hand, San Marchi and Mortensen, 2001 used a hot-wall gas-pressure infiltration apparatus to produce the same type of foams.

In relation to the preforms, these are commonly manufactured with sodium chloride (NaCl) due to their heat resistance and water solubility. As Gaillard et al. (2004) mentioned, in the foams that are produced by the replication process, the initial shape of the leachable powder that was used to produce the infiltrated preform, predominantly controls the pore shape. As a consequence, the powder shape influences significantly the strain at which the foam stress–strain curve deviates from power-law behaviour. However, most of the devices used for replication casting process are limited to produce small foams constituted by not-reactive metals such as Al and Al alloys, Zn and Zn alloys, among others. The replication casting technique has not been extensively used for manufacturing pure Mg foams, due its reactivity with oxygen in liquid state and with the material of the preforms (NaCl) during leaching stage.

The above mentioned limitations open up the possibility of designing new practical devices for the replication casting process with optimal control of the parameters (atmosphere, injection pressure, temperature, etc.) that allow the manufacture of pure Mg and Mg alloys foams and

other metallic foams. Therefore, the objective of this project is to manufacture a practical replication casting device for obtaining pure Mg foams, Mg alloys foams and other metallic foams with melting points lower than 950 °C, with optimum control of the process parameters. The design of the device allows producing samples with sizes up to 8.8 cm in diameter and 14 cm height, with the possibility of increasing or decreasing this geometry. With this replication-casting device, pure Mg foams and Mg alloys foams free from cavities and volumetric shrinkage defects can be obtained.

2. Description of the infiltration device

Fig. 1 shows the complete replication-casting device. The replication-casting device is conformed by three basic parts: First, a reaction chamber (a), followed by a valve system (b) for controlling the chamber pressure (i.e. vacuum or partial pressure) and gas injection for infiltration, and finally, a heating system (c). The heating chamber consists in an electrical resistance furnace with vertical chamber; this could be part of the device or could be external. In the following sections the design of the parts and operation of the replication-casting device will be described in detail.

2.1. Reaction chamber

In the reaction chamber the casting and infiltration process takes place. As can be observed in Fig. 2a, the chamber is composed by a hollow cylinder (1) and two covers (2-3). These parts are made of stainless steel 316. The stainless steel 316 is considered as the second most common austenite stainless steel, preferred for use in marine environments for its high resistance to pitting corrosion. This steel contains nickel and molybdenum elements, which increase the resistance to chloride-induced stress corrosion cracking (suitable for using preforms NaCl basis). Besides, these elements stabilize the austenite phase, (from cryogenic temperatures up to its melting

point). In addition, this stainless steel has high strength at elevated temperatures and is sometimes used for structural and pressure-containing applications at temperatures above 500 °C, as has been reported by Davis (1994). Therefore, the stainless steel 316 was selected for manufacturing the components of the reaction chamber.

The dimensions of the cylindrical chamber or hollow cylinder are: outer diameter 9.5 cm and inner diameter 9 cm with a thickness 0.5 cm and a height of 25 cm.

The reaction chamber is closed tightly with two removable circular covers of 16.5 cm in diameter and 1.25 cm in thickness. In the upper cover a stainless steel 316 tube is attached through which the injection of gas for the infiltration process is carried out and the fumes produced during casting are taken away. The dimensions of the tube are 2.5 cm in diameter and 100 cm in length. The tube length must be long enough to hold the valve system isolated from overheating. On the top of the tube a NW type connection is attached, this kind of connection is the most appropriate for joining the valve system. Inside the upper cover, a perforated nozzle for gas impulsion was assembled; this was also manufactured with the same stainless steel (Fig. 2b). The function of this nozzle is to evenly distribute the gas pressure throughout the whole sample. This mechanism prevents the formation of concave recesses on the top of the samples (in the liquid state) when high gas pressure is used (1.9-2.9 bar).

Four removable bolts of stainless steel 316 of 1.25 cm in diameter and 30 cm long are used to hold the covers to the cylindrical chamber. It is essential to place graphite seals between the covers and the cylindrical chamber in order to achieve a high vacuum and prevent the spill of molten metal during the infiltration process. The design of the device allows the change in its

dimensions i.e. increasing or decreasing the size or total geometry of the reaction chamber proposed in this work.

2.2 Valve system

In Fig. 3 the valve system suggested for gas and vacuum control is shown. As it was already mentioned, the valve system is assembled to the upper part of the tube for gas flow. This system has three important functions: i) controlling the argon (Ar) injection, ii) withdrawing gases during the infiltration process, and iii) allowing the connection of the reaction chamber to the vacuum pump. The valve system is completely detachable and consists of the following components: (1) a valve for controlling the reaction chamber, (2) a valve for controlling (Ar, He) gas injection, (3) a valve for controlling the vacuum, (4) cross-connector with terminal NW, (5) gauge and/or vacuum sensor (Pirani) (6) regulator for the (Ar, He) gas and (7) flexible hose for the connection to vacuum pump.

For practical purposes, for the operation and maintenance, it is recommended to use valves with NW terminals (sizes 10 and / or 25) and clamp connections of quick release with O-rings for high temperature (Fluorocarbon Type A). For the vacuum step, it is necessary to implement a digital vacuum sensor (Pirani), in order to obtain a more accurate measurement of vacuum in the reaction chamber. During the casting and infiltration steps, the digital vacuum sensor must be substituted by an analogue vacuum gauge. This gauge allows measuring pressures from -1 to 5 torr. Most models require an adapter to assemble elements with terminals NW. A rotary vane pump should be used to generate vacuum inside the chamber.

2.3. Heating system

An electric resistance furnace of vertical box with covers in the upper part can be used in the heating process. The design of this kind of furnaces allows that, the valve system can be located outside of the heating chamber and also facilitates the handling of the reaction chamber during the various stages of the process. Its capacity depends on the size of the reaction chamber to be used. An electric resistance furnace with the following dimensions: 50.8 cm side x 50.8 cm side x 50.8 cm height, was employed in this work.

3. The preform (Space holder)

In the replication-casting process the preform is essential for the final properties of the metallic foam obtained. The final structural and mechanical properties depend on the structural properties of the preform (porosity and sizes of pores). The particles used in the fabrication of the preform must achieve some requirements (i.e. high solubility in water to dissolve the preform from solid material, good adhesion among particles to preserve the integrity of the preform during the infiltration process). In this sense, as has been reported by Goodall and Mortenssen, 2007 the particles of NaCl (melting point= 801 °C) and NaCl-based particles of different size and geometry are appropriate to make preforms for the manufacture of pure Mg and Mg alloys foams and other metals such as Al, Zn, Sn, and their alloys.

The preform is manufactured inside the reaction chamber. At this stage, the reaction chamber must be pre-assembly with the bottom cover, including the graphite seal. It is advisable to apply a thin coat of boron nitride on the inner walls of the reaction chamber in order to further protect it from the molten metal. The size of the cylindrical preform must have a diameter equal to the

internal diameter of the reaction chamber; in this case 9 cm in diameter and a height of 14 cm were used. The maximum height of the preform is limited to half-height of the reaction chamber, since it requires a similar space to place the metal charge.

4. Assemble of the device parts

4.1. Assemble of the reaction chamber for the infiltration stage

Once the formation of the preform has been completed, the metal charge is placed into the reaction chamber, above the preform, in a volume ratio similar to that of the preform. In the infiltration stage, the use of graphite seals and the adjustment of the reaction chamber with bolts are necessary to reach the required vacuum and prevent leakage of molten metal during the infiltration process.

4.2. Installation of the valve system

Once the reaction chamber has been sealed, the valve system has to be connected to the NW terminal located in the upper part of the gas flow tube. For the first stage, the valve system shall be equipped with a digital vacuum sensor. It is necessary to check out that all the O-rings located between the clamps are in good condition (no cracks) and lubricated with vacuum grease.

When the valve system has been assembled, two thermocouples should be placed outside of the reaction chamber; one located at the bottom of the reaction chamber and the other in the area where the metal charge is located. The purpose of employing such thermocouples is to have a more accurate measurement of the temperature in the chamber and also monitoring the flow of liquid metal during infiltration (i.e. when the temperature decreases at the bottom of the reaction chamber indicates that the liquid metal has infiltrated the preform completely). The number of thermocouples and the type of data acquisition card are optional. Thereafter, the inner gas system

(Ar) and the vacuum pump must be connected to the valve system through the appropriate hoses. Finally, the complete replication-casting device is introduced into the furnace, ensuring that the valve system remains outside the furnace, with at least 50 cm, to avoid overheating. It is suggested to use a refractory thermal insulation ceramic fiber cloth between the furnace door and the stainless steel connecting tube.

5. OPERATION OF THE REPLICATION CASTING DEVICE

The replication casting process is carried out in five steps: A) atmosphere conditioning, B) melting of the metal charge, C) infiltration of the molten metal charge by gas injection, D) cooling stage, E) disassembled, salt leaching and preparation of specimens.

5.1. Conditioning of atmosphere

Due to the magnesium and its alloys have high reactivity with oxygen in the air; ignition or combustion occurs rapidly when they are in contact with oxygen in the molten state. For this reason is necessary to carried out the process in a controlled atmosphere.

The conditioning of atmosphere involves removing the air from the reaction chamber and replacing it with an inert atmosphere, typically of Ar. The procedure is as follows: based on the valve system shown in figure 3, the valves (1) and (3) should be opened and the vacuum pump and the vacuum digital sensor switched on. The proposed replication-casting device can achieve a vacuum up to 3.5×10^{-2} Torr approximately, within a period of time of ~ 0.2 h. When the required vacuum has been reached, the valve (3) is closed and then the gas (Ar) valve (2) is opened, using a pressure of 0.9 bar to fill up the reaction chamber. This purging procedure should be repeated at least three times in order to minimize the residual amount of oxygen in the chamber.

5.2. Melting stage

Due to the sensitivity of the digital vacuum sensor to high temperatures, it must be replaced by an analog vacuum gauge before starting the melting of the metal charge. In this step, valve (1) (that controls the inlet and outlet of the gases from the reaction chamber) should be closed to keep the inert atmosphere.

With the vacuum gauge already installed, the (Ar) gas pressure is adjusted at 0.49 bar and then the valve (1) is again open. This valve is kept opened during the melting process. During this step, the metallic charge positioned above the preform is gradually melted. Therefore, the melting temperature should be high enough to drop the viscosity of the liquid metal and then, be able to completely infiltrate the preform through its pores. It is suggested to maintain the heating for 1 hour so that the metallic charge, as mentioned above, can be completely melted and the liquid metal has an adequate fluidity. After this thermalization period, the infiltration process is the next step.

5.3. Infiltration process

The infiltration process is carried out at a gas pressure greater than that used in the melting process. In order to start the infiltration process, the valve (1) is closed and the (Ar) argon pressure is increased, using the regulator of the argon (Ar) cylinder. According to the valve system and the reaction chamber that are being proposed, it is suggested to use a maximum pressure of 2.95 bar to prevent damages to any parts of the system. When the injection pressure has been adjusted, the valve (1) must be opened slowly and stay open for the necessary period of time so that the molten metal completely fills the pores of the preform. In the present work, 10 minutes for the infiltration process were enough.

5.4. Cooling stage

Before removing the reaction chamber from the furnace, valve (1) that controls the reaction chamber, and then the valve (2) have to be closed. At this stage, the furnace could be turned off and then the hoses that connect the vacuum pump and the Ar cylinder with the valve system should be disassembled. With exception of valve (1), the entire valve system should be removed from the reaction chamber to facilitate its manipulation. Subsequently, the reaction chamber must be taken out from the furnace and place it on a suitable surface (copper plate) for cooling to room temperature. It is important that the solidification of the molten metal begins at the bottom of the samples and finish in the top this in order to avoid defects such as volumetric shrinkage in the body of the foams.

5.5. Disassembled and salt leaching and preparation of specimens

When the reaction chamber is completely cold, the bolts and the covers are removed. Subsequently, the metal-preform sample is extracted from the reaction chamber. The final step in the magnesium foams manufacturing by infiltration method is the salt leaching. For this purpose, it is necessary to consider the reactivity of the pure magnesium with water in order to minimize or prevent the corrosion. Shaw, (2003) reported that according to the Pourbaix diagram for the magnesium-water system, increasing the pH value above 11.5, a stable $\text{Mg}(\text{OH})_2$ layer is formed, being this layer a good protection against further corrosion. According to the calculations, it is necessary to dissolve 2g of NaOH in 500 ml of water to obtain a solution with pH=13.

VI. RESULTS AND DISCUSSION

Fig. 4 shows representative foams of: a) pure Mg and b) Mg-10 wt.% Al alloy obtained by the proposed replication-casting device. The preforms were manufactured with irregular NaCl granules with an average size of 3.3mm. The size of the preforms was 9 cm in diameter and 10 cm in height. The working temperature used for both materials; a) pure Mg and b) Mg-10 wt.% Al alloy was of 720 °C, while the pressure and the injection time of Ar were 1.96 bar and 10 minutes, respectively.

The above-mentioned conditions are optimal to obtain complete foams with equivalent preform size, without defects such as internal voids (several cross section cuts were carried out confirming this statement) and surface concavities. This can be also attributed to the use of the perforated nozzle for gas impulsion lid, which evenly distribute the gas pressure (non-centralized) on the molten metal charge. Fig. 5 shows a sample manufactured without the use of the perforated nozzle device. It can be seen that in the central part of the foam a deep concavity was formed due to centralized gas flow. Therefore, an evenly distribution of gas pressure during the infiltration process and the control of the solidification from the bottom of the reaction chamber to the top could minimize the formation of defects in the foams.

Representative structures of pure Mg and Mg-10 wt.% Al alloy foams are shown in fig. 6a and fig. 6b. It can be seen that both foams are entirely constituted by open-porosity with a high visual degree of openness. Furthermore, pore structures are homogeneous with high degree of interconnection between them, forming interconnected channels (Fig. 6). From the same figure, it

can be observed that the metallic structure of the Mg foams was not oxidized, which means that the protective atmosphere used during the infiltration process as well as the use of a H₂O+NaOH solution with pH=13 for the salt dissolution were effective, protecting the foam from oxidation and ignition.

The relative density of the pure Mg foam was 0.30, while the relative density of the Mg-10 wt.% Al alloy foam was 0.33, with porosities of 70% and 67%, respectively. This morphology of the produced pore structure could be suitable for functional applications such as filters, catalyst supports, and CO₂ captors, among others.

VII. CONCLUSIONS

A replication casting device with the capability of manufacturing open-cell pure Mg and Mg alloys foams and other metals or alloys with melting points lower than 950 °C was developed. In comparison with other devices reported in the literature, the device proposed in this work showed significant advantages, such as easiness operation, feasibility of producing smaller or larger foam samples, safety and low maintenance costs. The experimental results demonstrated the ability of the replication casting device to optimally produce large open-cell pure Mg and Mg-10 wt.% Al alloy foams. The design of the device provides the advantage of producing foams free from defects such as large volumetric shrinkage and large concavities. The latter was drastically minimized by the implementation of a lid with a perforated nozzle for gas impulsion. Due to the

structural properties of the open-cell Mg and Mg-10 wt.% Al alloy foams produced, these foams could be used in functional applications such as filters, scaffolds, CO₂ captors, catalyst supports, etc.

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Fig. 1. Replication casting device: (a) steel reaction chamber sealed with two covers, (b) valve system and (c) heating system (furnace).

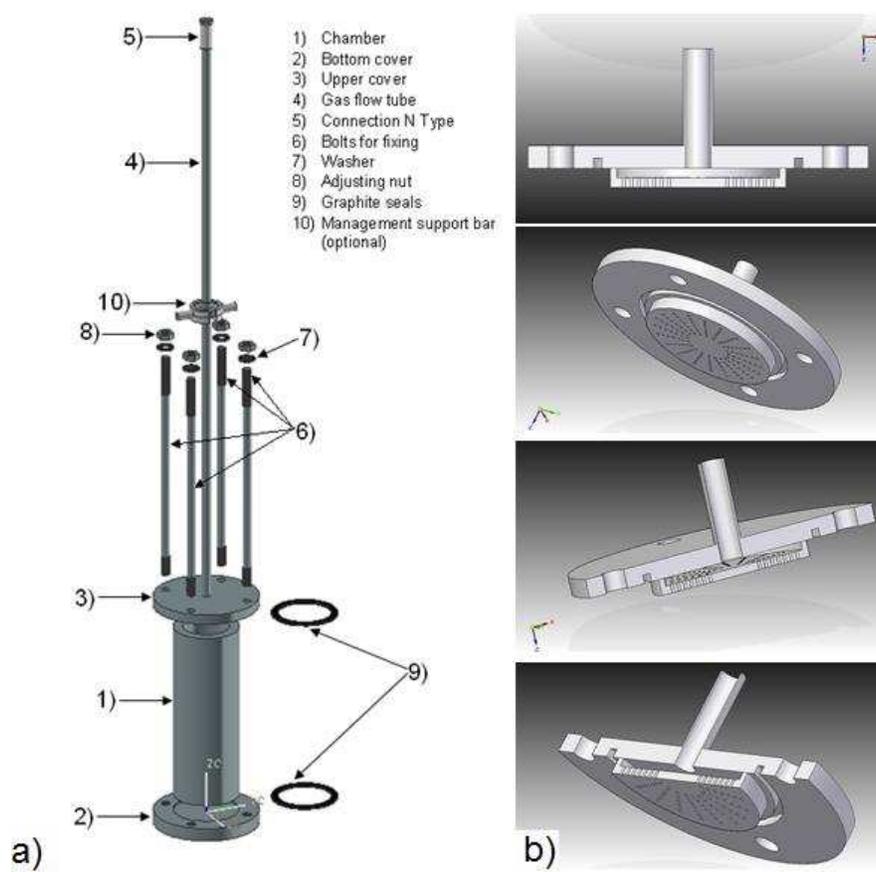


Fig. 2. a) Components of the reaction chamber, b) perforated nozzle for gas impulsion.

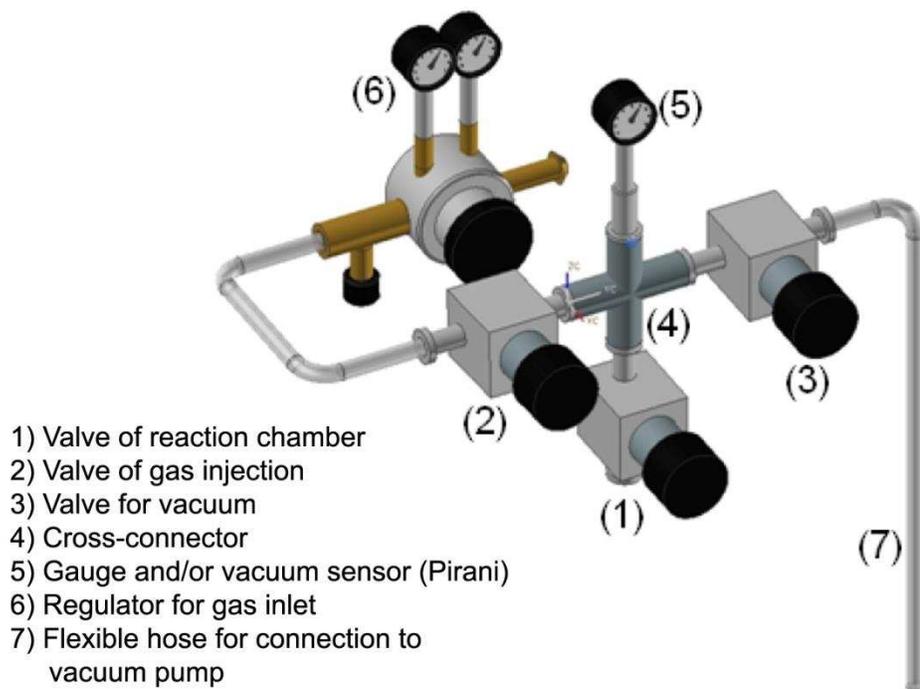


Fig. 3. Scheme of the valve system components.

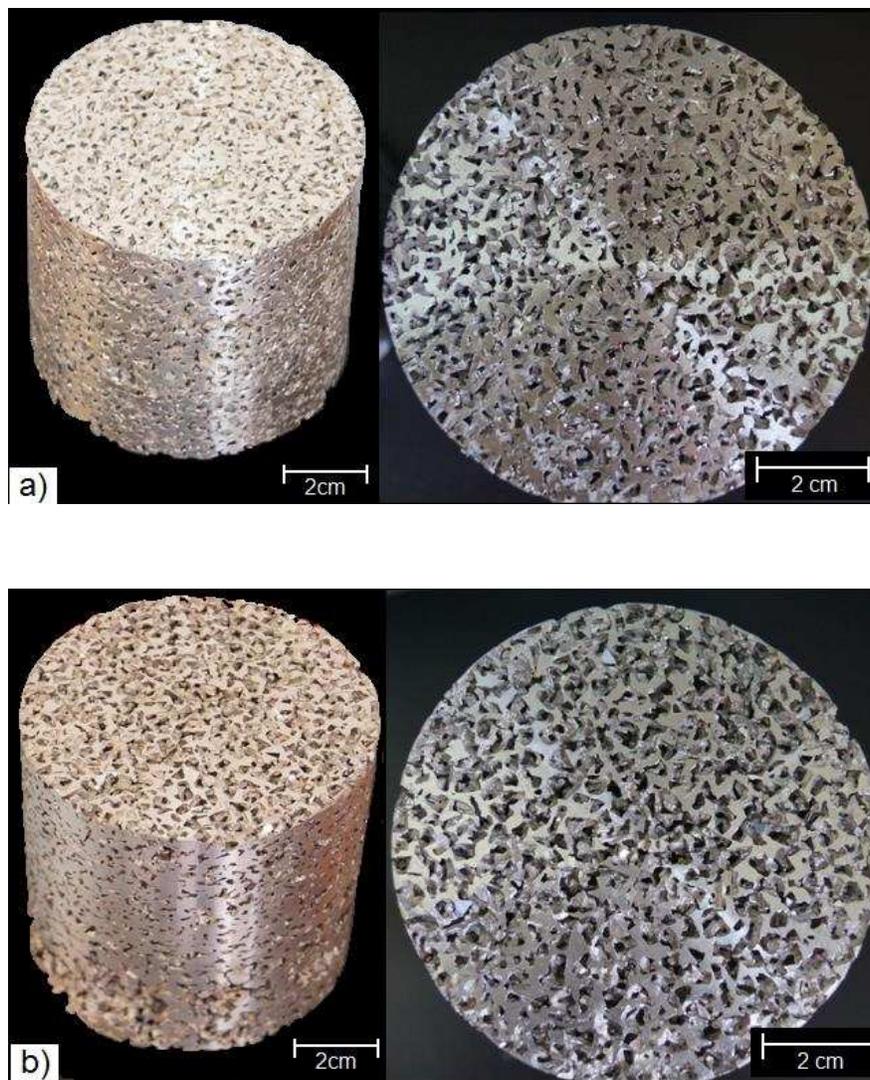


Fig. 4. Digital images of: a) pure Mg foam, b) Mg-10 wt.% Al alloy foam, manufactured by the replication casting method proposed in this work.

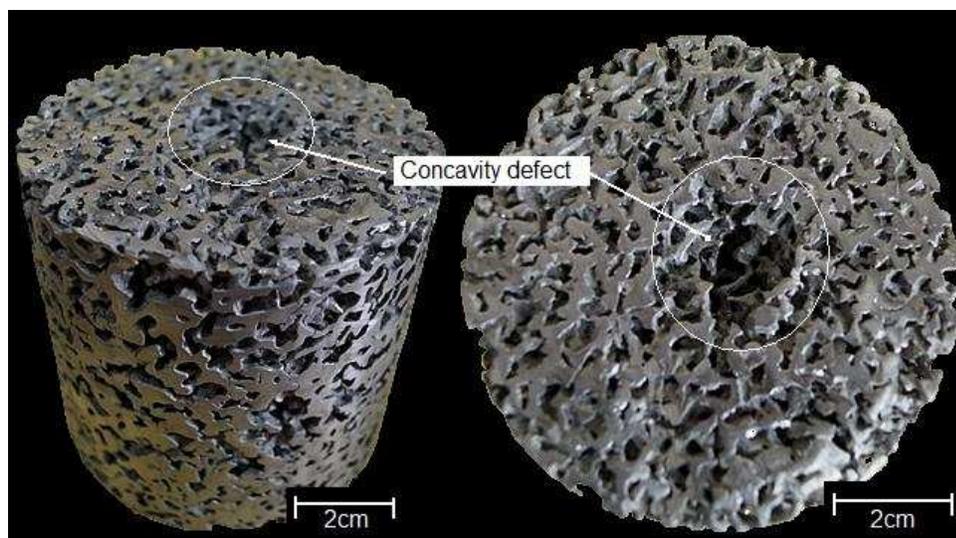


Fig. 5. Representative concavity defect formed on top of the foam due to the centralized gas flow.

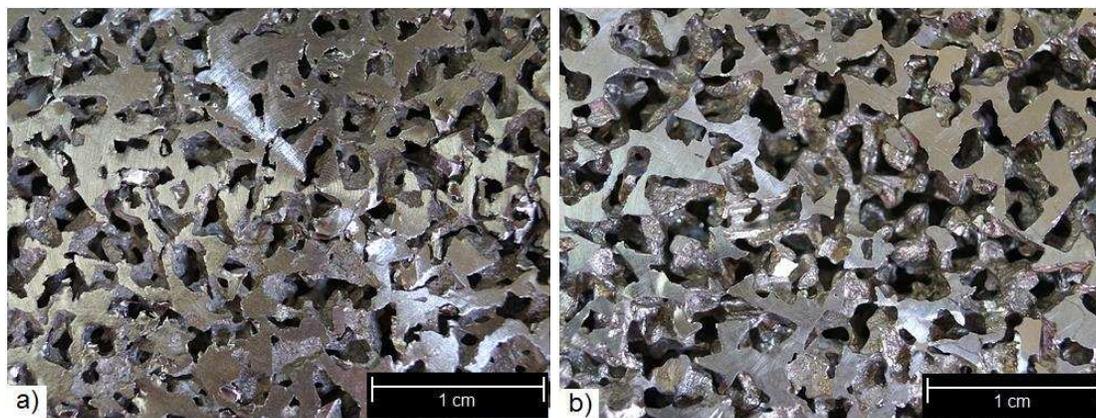


Fig. 6. Cross section of the produced foams showing the open-cell pore morphology of: a) pure Mg and b) Mg-10Al (wt.%) alloy.