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# Investigation and Comparison of Aluminium Foams Manufactured by Different Techniques

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## 1. Introduction

Cellular metals offer a large potential for industrial application. Nevertheless, besides the costs, there are a lot of technical improvements necessary in order to gain more widespread use. The reasons for the lack of applications, since now, are multiple and they depend from the physical properties of foams that are still not good enough and not completely experienced, and from the insufficient spreading of research results to designers.

To fulfill parts of these requirements, this work investigates in detail three foam production processes, studies the effect of modifications to standard manufacturing methods, clarifies the influence of process parameters on the structure of foams, characterises relevant properties, and finally discusses the difference between powder or melt routes. During experimentation hundreds of samples were realised to reach these objectives. Moreover mechanical, physical, and microstructural properties of the produced foams have been studied using various techniques including compression testing, scanning electron microscopy, visual inspection, and software measurement tool. The three methods for manufacturing aluminium foams, applied in this investigation, are named TiH<sub>2</sub>, SDP and MGI. Two of them (TiH<sub>2</sub> and SDP) start from powdered aluminium as raw material, while MGI starts from melt aluminium. Moreover TiH<sub>2</sub> and MGI methods let the production of closed cells foam while SDP of open cells foam named, more correctly, metal sponge.

The term “foam” was firstly reserved for a dispersion of gas bubbles in a liquid. The morphology of this type of foams can be preserved by letting the liquid solidify, thus obtaining what is called a “solid foam”. When speaking of “metallic foams” one generally means a solid foam. The liquid metallic foam is a stage that occurs during the fabrication of the material. For metallic systems it is possible to define the following classification according with Babcsan et al. (2003):

- cellular metals are materials with a high volume fraction of voids made up of an interconnected network of walls and membranes;
- porous metals have isolated spherical pores and a porosity level of usually less than about 70%;
- metal foams are a subgroup of cellular metals usually having a polyhedral cells with closed or open cells (even if there are no membranes across the faces and the voids are interconnected, the better definition is metal sponges).

## 2. Manufacturing methods

Considerable progress has been made recently in the production of metallic and in particular aluminium foam. Scale up has progressed so far that widespread commercial use has now become a reality. Methods to produce metal foams are already known since the fifties but their use has not spread so far since now for the difficulties to control the process parameters and high costs. Due to the progress of the last decade with the respect to the production techniques gave new birth to the foam (Surace & De Filippis, 2010).

There are different ways to manufacture cellular metallic materials but some of them methods will always be restricted to specialised applications where cost is not a parameter of paramount importance. The various methods can be classified according to the state the metal is processed in. This defines four group of processes each one corresponding to one state of matter (Banhart, 2001):

- liquid metal;
- solid metal (usually in powdered form);
- metal vapour;
- metal ion solution.

The three methods studied in this work will be now briefly presented.

The "TiH<sub>2</sub>" method consists of mixing aluminium or aluminium alloys powders with an appropriate blowing agent, usually titanium hydride (TiH<sub>2</sub>) for aluminium and its alloys: in particular AlSi7 e AlSi12 show an excellent foamability due to their low melting point. The mixture is then compacted to a dense product called "foamable precursors materials" with different methodologies ensuring the embedding of blowing agent in the matrix without residual porosity: in IFAM process (Fraunhofer Institute in Bremen - IFAM) by uniaxial compression, CIP, powder rolling or extrusion, the Mepura process uses a continuous extrusion technology (Baumgartner & Gers, 1999). The following step is the heat treatment up to the melting point of the matrix and above the decomposition temperature of the blowing agent that release hydrogen gas. The gas leads to an expansion of the material resulting in a porous structure with closed cells. By cooling under the melting point the foaming is stopped. Fig. 1 shows a sketch of the process. The time needed for full expansion depends on temperature and on size of the precursor, and ranges from a few seconds to several minutes. Fig. 2 shows the expansion of an AlSi12/TiH<sub>2</sub> powder compact: the pictures were taken interrupting the foaming by quenching (ex situ measurements of foam expansion). By varying the time span from start to quenching, a series of samples was obtained reflecting various stages of foam evolution. The expanding foam is shown as a function of the furnace holding time; foaming started after about 10 min and was completed after 11 min and 15 s (in this way the best structure was obtained). Beyond this time the foam collapses for two reason: drainage (i.e. the flow of the molten metal, driven by gravity) and coalescence (i.e. merging to form a larger cells).

For attaining by this route near net shape foam parts, it is necessary to insert the precursor material into a hollow mould but also hollow profiles (es. tubes or columns) that can be filled with foam (Fig. 3).

The second studied method is the Sintering and Dissolution Process (SDP). Generally, foams can be obtained by using a leachable material (e.g. salt) together with metal. Following this principle there are two different techniques starting from melt metal or powders. The first method, called Replication Technique, consists of three basic steps: by packing a soluble salt in a mould to have a pattern, casting melt metal around these granules and finally removing

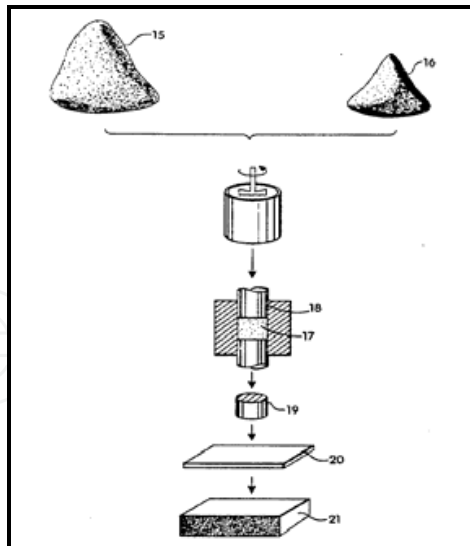


Fig. 1. Sintering of metal powders (US Patent 5,151,246)

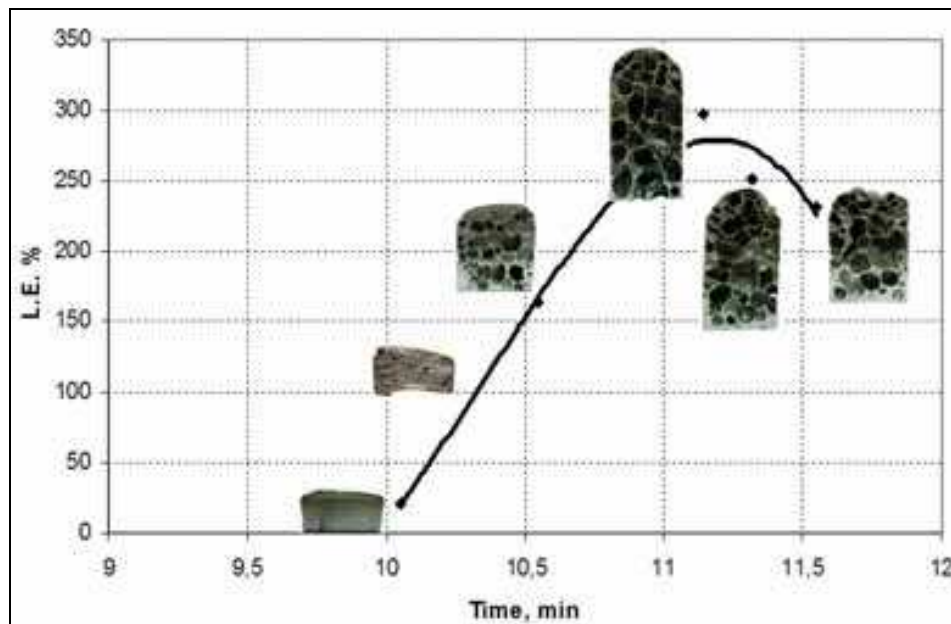


Fig. 2. Foam samples presenting different expansions at different foaming stages



Fig. 3. Hollow tube filled with foam (courtesy of Valerio Mussi - MUSP)

the pattern. Reference technique was first applied in 1966 (Kuchek, 1966) and similar processes have been developed in recent years (San Marchi et al., 2000; Li et al., 2003; Gaillard et al., 2004). A more advanced version of this process uses a hot-wall pressure infiltration process: the salt preform is held under vacuum while a block of aluminium is melted over it and an inert gas at high pressure is applied during the subsequent infiltration step. In the second method, called Sintering and Dissolution Process (SDP), the powders have been used to produce a dense two-phase precursor where one phase is water soluble. The powders (usually Al and NaCl) are mixed and compacted, forming double-connected structures of both phases. After furnace sintering (Fig 4 a-b), by dissolving the leachable phase, a foam of the other phase is produced (Fig. 4 c). This process, studied at Liverpool University (Zhao et al., 2004; Sun & Zhao, 2002; Zhao & Sun, 2001; Sun & Zhao, 2005), belongs to the processes defined "space holder techniques" giving structure of a great uniformity (Ashby et al, 2000). A recent study compares the sintering of Al-NaCl compact by traditional electric furnace sintering and by spark plasma sintering that allows the increase of plateau stress (Wen et al, 2003). Moreover Zhao developed Lost Carbonate Sintering process (LCS) to manufacture copper foam using potassium carbonate ( $K_2CO_3$ ) as leachable salt with the same reference technique of SDP (Zhao et al., 2005). Fig. 5 shows a cross section of a sample.

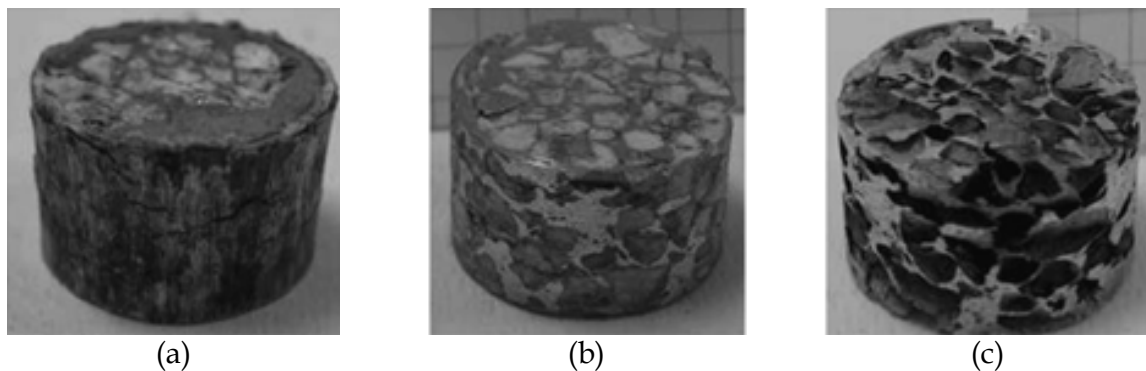


Fig. 4. Sample after sintering (a), machining (b) and leaching (c) (40 wt% Al, 3 h, 650 MPa)

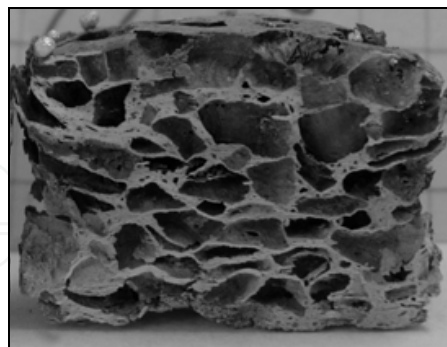


Fig. 5. SDP sample cross section

By far the cheapest type of process is melt-route processing (the Alcan/Norsk Hydro process) called Melt Gas Injection - MGI (Wood, 1997 and Asholt, 1999). According to this process, silicon carbide, aluminium oxide or magnesium oxide particles are used to enhance the viscosity of the melt metal; their volume fraction ranges from 10% to 20% and the mean size from 5 to 20 $\mu$ m. The ceramic particles trap gas bubbles owing to the favorable interface energy and serve as stabilizer of the cell walls and delay their coalescence. They also reduce the velocity of the rising bubbles by increasing the viscosity of the melt (Ip et al., 1999;



Kaptay, 2003; Hur et al., 2003). Therefore, the first step requires the preparation of a melt bath containing these substances. A variety of aluminium alloys is used, e.g. the casting alloy AlSi10 Mg (A359) or wrought alloys such as 1060, 3003, 6016, or 6061. The liquid Metal Matrix Composites (MMC) is then foamed in a second step by injecting gas (air, nitrogen or argon) into it, using specially designed rotating propellers or vibrating nozzles as depicted in schematic form in Fig. 6. The function of the propellers or nozzles is to create very fine gas bubbles in the melt and distribute them uniformly. This is an important requirement because only if sufficiently fine bubbles are created, a foam of a satisfactory quality can be obtained. The floating foam is then continuously pulled off from the surface of the melt with different techniques (for example by means of conveyor belt to obtain sheets).

The MGI for processing closed-cell metal foams is very attractive since this approach allows economic handling of large quantities of material. Melt route processes are also well suited to the use of scrap as feedstock. Although cheap and relatively simple, consistently obtaining a cell structure with a reasonably high quality is difficult, which has given rise to various adaptations to control the cells more closely. Fig. 6 shows a sample of foam obtained by MGI process.

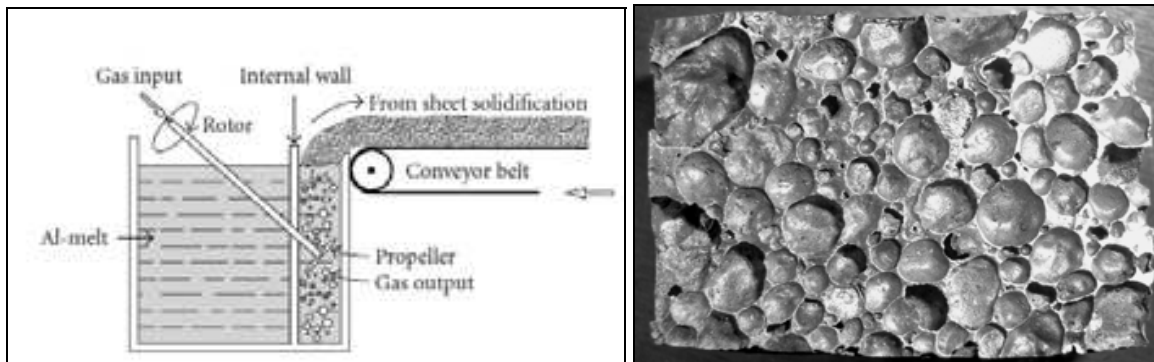


Fig. 6. Apparatus for MGI and example of foam obtained by MGI

Moreover, with this method, it is possible to produce 3D-shaped parts with complicated shape or configuration, with a modification of original process (Fig. 7) (Surace et al., 2009). These parts are expected to be utilized as filling material and for encasing in components without machining. Casting aluminum around a foam can create components where a low density foam core is completely surrounded by a massive outer shell. Potential applications for this kind of foam core castings are space frame nodes, knuckles, control arms, cross-members, and stiffness providing structural components.

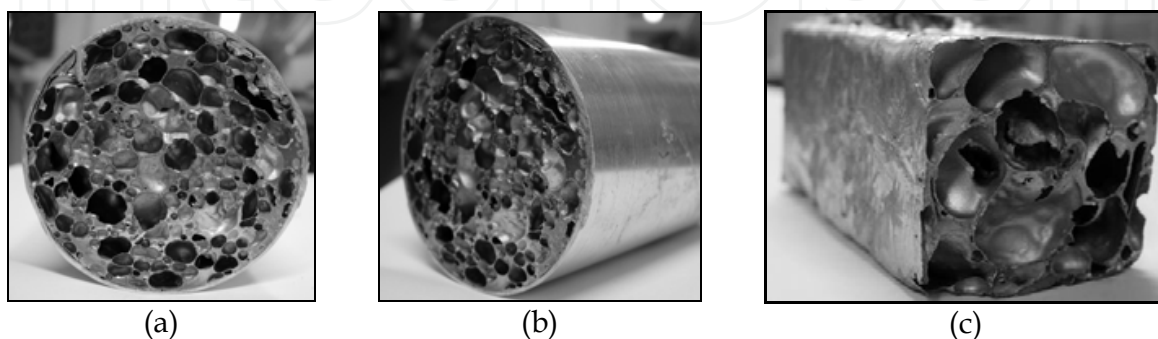


Fig. 7. MGI foam cores attached by mechanical bonding (front and lateral view (a-b) and foam part with parallelepiped shape (c))

### 3. Processes comparison

In this section will be discussed the advantages and disadvantages of each of three presented processes. The first advantage of  $TiH_2$  process is that complex-shaped metal foam parts can be easily manufactured by expanding the foam inside a confined mould. The light weight solution based on aluminium foam can replace traditional cast aluminium parts saving 30% weight because composite structures of aluminium foam and bulk metal parts can be made without using adhesives. For making such composites the foamable precursors material is first bonded to the solid section or sheet by co-extrusion or roll-cladding, after which the foamable core layer is expanded by heat treatment. Other advantages of the powder-compact route are listed in Table 1 reported from Banhart & Baumgartner, 2002. Besides the first two features already mentioned, the flexibility arising from the preparation of the precursor from powders is important because different alloys composition can be made simply by mixing elementary powders. No ceramic additives are needed to stabilize the foam, in contrast to some of the melt-route foaming processes in which silicon carbide has to be added. However, if required, ceramic powders or fibers can be added for a special applications such as for reinforcement or to increase wear resistance.

Naturally, there are also some disadvantages inherent to the process. Metal powders are more expensive than bulk metal and they require efforts for compaction. This rules out applications that require very cheap materials. Moreover, the size of aluminum foam parts that can be manufactured is limited by the size of the backing processes.

Differently, the liquid-metal route allows for making panel 15 m in length (Cymat Corporation, 2002) and 100 cm thickness (Miyoshi et al., 1999). However, as reported from Banhart & Baumgartner (2002), these processes cannot be used for near-net-shape production and only permit very simple geometries but Surace et al., 2009 have demonstrated the possibility of production of shaped parts with MGI-mould process.

In  $TiH_2$  process, it is of critical importance to reach the right compaction of the precursors so that blowing agent results tightly embedded into the metal matrix. Foam formation, indeed, happens during the heating process when the metal matrix is near the melting point and sufficient liquid phase is formed so that the gas released by the foaming agent can bubble in it. The more the two phenomena, liquid metal formation and gas development, will overlap, as easier will be the foam formation (Matijašević-Lux, 2006; Speed, 1976). Decomposition of  $TiH_2$  begins at a temperature around 350 °C at atmospheric pressure, and continues up to 600 °C. However for the hydride embedded in the metal matrix, the gas release is shifted to higher temperatures because of the increase in partial hydrogen pressure that counteracts further  $TiH_2$  decomposition. In principle, the compaction can be done by any technique that ensures that the blowing agent is embedded into the metal matrix without any notable residual open porosity. From this point of view, a higher compaction helps to match the main hydride decomposition with the metal melting temperature. Examples of compaction methods are hot uniaxial or isostatic compression, rod extrusion or powder rolling. However, extrusion seems to be the most economical method at the moment and is therefore the most used way (Banhart, 2001).

In this investigation powder compaction was performed by cold compaction by uniaxial pressing. This is a fair simplification of the process respect to other methods. Even if cold uniaxial compaction can not reach densification values like that obtained by hot processing and residual porosity in the green is higher, using appropriate process parameters combination it is possible to foam aluminium with a cell structure comparable with the

foam made by the usual method (Fig. 8). The middle column of Table 1 lists some of the problems that are still encountered when foaming aluminium with the powder-compact method.



Fig. 8. TiH<sub>2</sub> foam sample obtained by cold uniaxial pressure

ADVANTAGE	PROBLEM	DISADVANTAGE
Net-shape foaming possible	Uniformity of pore structure still not satisfactory	Cost of powders
Composites can be manufactured	Process control must be improved	Very large volume parts difficult to make
Parts are covered by metal skin	Permeable (holes)	Coating process requires sealing
Graded porosity can be achieved	Difficult to control	
Flexibility in alloy choice		
No stabilising particles have to be added		
Ceramics and fibers can be added		

Table 1. Characteristic of powder compact foaming method

Advantages of SDP process comprise considerable control of the pore size, and thus density, the capacity for near-net shape component production, and the potential for production of components that are part dense and part porous. Also, the open-porosity that is intrinsic to metal sponge can be put to advantage in several applications that require fluid flow through the porous metal, such as filters and heat-exchangers.

The structure or architecture of metal sponge produced by replication is flexible and determined by the pattern: porosity as high as 98% (Wagner et al., 2000) and as low as 55% (Banhart, 2000) for different pattern materials have been reported and pore sizes as small as 10  $\mu\text{m}$  have been achieved (San Marchi et al., 2000). In addition, metal sponge can be produced from virtually any alloy that can be cast or powdered including aluminium alloys, magnesium alloys, and iron and nickel alloys. Sodium chloride (NaCl) has been employed



most extensively as a leachable pattern, because it is inexpensive and easy to handle. Salt patterns are limited by their melting point; NaCl, for example, is limited to aluminum and lower-melting alloys, while NaF could potentially be used at temperatures greater than 900°C. Additionally, highly concentrated saline solutions are generated during dissolution and can cause significant corrosion in some alloys.

The main disadvantage of the powder metallurgy techniques are their relatively high cost, due mainly to the handle fine metal powders. But differently from each powder technique, SDP does not require a powder foaming agent but only a culinary salt with saving of money and precautions. Material chosen as space holder for the production of SDP foams must fulfill a main requirements: it must be chemically compatible with the foam material. For the production of foams with materials that are processed at temperature around 650-750 °C, sodium chloride is a well suited space holder. It is inexpensive, chemically inert in contact with molten aluminium and many other materials, leaches easily since it is highly soluble in water, and creates essentially no environmental or health hazards (Conde et al., 2006).

In some cases, leachable patterns can also be machined to complex geometries prior to leaching and without damaging the architecture of the sponge. In the case of salt, removal of the pattern is by immersion in water causing dissolution of the salt. Dissolution is primarily a diffusive process, as the dissolved salt ions diffuse from their place-holding position into the water bath through the narrow channels in the sponge and the rate and total time of dissolution depend strongly on the size of the specimen. Salt is more difficult to remove from small pores, particularly if gas is entrapped in the pores; this is the so-called "gas-lock" phenomenon, which can block the penetration of fluid into the porous network. Corrosion of metal sponge during leaching of the pattern can be a problem due to the large surface area of the porous network, thus the salt concentration and the immersion must be minimised (San Marchi & Mortensen, 2002). Summarizing the advantages of this process are:

- Low cost: space holders are cheap.
- Environmentally friendly: removal of space holders does not lead to harmful emissions. However, if polymers are used, care must be taken to condense and collect the extracted polymers.
- Recyclable: used components can be recycled in the same way as normal cast components.
- Composite structures: sponge and non-porous metal parts can be bonded during casting.
- Near-net-shape manufacturing of components is possible.

The melt route MGI for processing closed-cell metal foams is very attractive since this approach allows economic handling of large quantities of material. Melt route processes are also well suited to the use of scrap as feedstock. For the production of homogeneous foams some prerequisites have to be fulfilled. One important step is to increase the viscosity of the melt to prevent gas escape with particles added that increase the melt viscosity and stabilise the cell walls. But if ceramic particles are used for stabilising the liquid foam, the excessive wear on the machine tools has to be taken in account during machining, especially if SiC particles are present. It should also be noted that the preparation of the composite material requires relatively long time steering processes to achieve the proper homogeneous distribution of the particles in the melt. The production facility setup by Cymat is capable of casting foam panel in continuous length at an average rate of 900 kg/h up to 1.5 m wide

with a thickness range of 25-150 mm (Korner & Singer, 2002). This shows that the process is relatively straightforward and economical.

The foam, obtained by MGI, usually presents a gradient in density and pores elongation as a natural consequence of the gravitationally induced drainage and the shearing forces of the conveyor belt that lead to distorted cells in the final product. This obviously has a pronounced effect on the mechanical properties which become anisotropic (Beals & Thompson, 1997). The situation could be improved by pulling off the foam vertically (Sang et al., 1994). Moreover, obtaining shaped parts by this process is very difficult for the ceramic particles that make difficult the cutting. Attempts for making shaped parts have been undertaken by casting the semiliquid foam into moulds or by shaping the emerging foam with rolls, thus trying to eliminate this disadvantage (Kenny & Thomas, 1994, Kleinheyer & Bilz, 1995; Nichol, 2006). Surace et al. (2009) proposed a new method, called MGI-mould process, that makes possible to produce 3D-shaped parts with complicated shape or configuration using some moulds obtained by traditional investment casting process. The MGI-mould process was capable of producing the near net shape aluminum foam parts with length of about 200 mm and different shapes with skin surface and good internal quality especially for the core of automotive articles.

Foam parts prepared by expansion in a mould show a closed surface skin with a thickness comparable to the cell-wall thickness of about 200  $\mu\text{m}$  (Fig. 9). Casting aluminum around a foam can create components where a low density foam core is completely surrounded by a massive outer shell. The shell can be designed in such a way that additional functions besides its load carrying can be fulfilled. As only one processing step is required to produce such a foam core component, production is expected to be very economical (Degischer & Kottar, 1999). A further difficulty results from the fact that methods have to be developed for fixing the cores. A prerequisite for encasing by casting is a dense foam surface.

A suitable core attachment system has to perform two functions: first it has to keep the core in place in the die when the die is open and during the movement of die closing. The more important second function is to maintain the desired distance between the foam core and the die wall during the casting process because this distance determines the wall thickness of the castings. One method to realise a suitable attachment of the core is by creating elongated wedge-shaped spacers during the foaming process of the cores (Fig. 9). These spacers have to be long enough to transfer the acting forces to the foam core without damage. It is very important to place the spacers corresponding to the melt flow to avoid flow barriers resulting in "dead zones".

Generally, no bonding develops between the core and the shell during castings because of the continuous aluminium oxide layer that prevents the core surface from reaction with the molten metal (Simancik & Schoerghuber, 1998). There are two possible ways to improve the bonding:



Fig. 9. Shaped MGI part with dense skin



Fig. 10. MGI foam core insert with spacers

- mechanical bonding by flow of liquid metal into the outer foam structure supported by intentional weakening of the surface skin, e.g., by sand-blasting. Disadvantages are that the weight of the casting increases and the bonding occurs only locally and is difficult to control (Fig. 11).
- Metallurgical bonding by coating the cores with various agents supporting diffusion through the aluminium oxide layer. With a suitable metallic coating a metallurgical bonding can be achieved. On this point, however, further research is necessary.

Stress bonding, which is caused by shrinkage of the casting during solidification or the fixing of the core due to its geometry, leads to a solid joining of the core insert in the casting (Kretz & Wolfsgruber, 2003).



Fig. 11. MGI foam cores attached by mechanical bonding (front and lateral view)

The recyclability of metal is a benefit, enabling ecologically sustainable product life cycles. Cellular metals can be shredded too and treated as normal scrap. It might be of interest to recycle the MMC matrix of the foam without significant reduction of the particle content. Any reuse of MMC saves the effort necessary to bond the two components together during processing. Originally, this type of foam was a spin-off of particulate-reinforced aluminum processing, consequently it has been proposed to reuse particulate-reinforced aluminum alloys in the production of aluminum foam (Gergely et al., 2000). For SDP could be difficult the recycling due to trapped salt that can contaminate the melt. Recycling of  $TiH_2$  seems to be the most simple.

#### 4. Experimental

The aim of the experimental part is to evaluate the properties and to optimize the process parameters of the three mentioned techniques by means of a statistical approach. During the

experimental work many samples have been made following the principles of the design of experiments (DOE) and the final evaluation of the most important features (morphology and compression strength) was carried out.

#### 4.1 TiH<sub>2</sub> process

In this process the selection of the aluminium powders was a very difficult task. In fact, during the initial screening, four types of Al powders from different suppliers were tested. The problems were associated with the size of the powders: too small particles are not suitable to obtain foam. The content of the blowing agent (TiH<sub>2</sub>) was in the range of 0.5–2 wt%. This wide range (different from that reported by Duarte & Banhart, 2000) was chosen due to the mentioned problems with fine Al powder (causing hydrogen loss during precursors heating). Moreover, in order to improve the stability of metal foams, usually ceramic particles were added. Literature data report that ceramic particles inhibit melt flow, after increasing the viscosity, thus decreasing the rate of cell coarsening and drainage of liquid through the structures (Ip et al., 1999). The tested amount of SiC was in the range of 0–10 wt%.

The mixing was the second process step carried out through mixing of the metal powders with the blowing agent in glass mixer to ensure the homogenization of components. After the powder compaction may be performed using different techniques (hot pressing, cold isostatic pressing, extrusion) (Baumgartner & Gers, 1999). In our experiments, powder compaction was performed adding about 15 g of the mixture (depending on the composition) in a 32 mm diameter steel die, followed by cold compaction by uniaxial pressing. This is a fair simplification of the process in comparison to others methods. Compaction in the range of 100–700 MPa was obtained with a MATEST E157 hydraulic motorized press. Finally foaming was carried out in a pre-heated electric furnace at different temperatures in the range of 620–950°C. Foaming operation took place in normal atmosphere and the mould was opened only at the top. In the furnace, the mould was placed on a ceramic plate, excluding temperature gradient in vertical direction as suggested from Arnold et al., 2003. Careful control of heating conditions during foaming is essential to obtain quality foams. The difficulty is that the liquid foam is thermodynamically unstable and conditions change constantly during foaming (Baumgartner et al., 2000). Due to that, when the mould is filled with foam, it must be suddenly cooled down below its melting point to stabilize the structure. Air quenching leads to foam collapse, therefore water was preferred for cooling. Moreover, in air-cooled samples the foam structure shows pronounced cracking of cell walls that is not the case by water quenching (Lehmus & Banhart, 2003). In the present work, foaming was stopped at a moment, which depended mainly on the furnace temperature.

Variables, influencing the foaming process, are the TiH<sub>2</sub> and SiC content, mixing time, compaction tool, pressure, sintering temperature and time, mould features and the cooling procedure. Each variable influences the output foam quality. In the initial screening, a large number of samples was tested by changing parameters in wide ranges. As a result it was found that although many control factors influence the process, the output quality depends mainly on three primary control factors: SiC fraction, compaction pressure and sintering temperature. Influence of these reference factors on the cells area, cell wall thickness, linear expansion, relative density and plateau stress was investigated. A standard approach of DOE is to use the full factorial method that requires a total of 27 experimental runs if there



are 3 factors to be investigated and each consists of 3 different levels (high, medium and low). Table 2 shows the levels of factors in this systematic investigation. For each combination of the parameters, three replications have been carried out. The quantity of Al was fixed at 15 g and TiH<sub>2</sub> at 1 wt%. After screening, different samples were made in the second experimental step, following the principles of DOE to study mechanical properties and the evolution of the foam in time.

Factors	Levels		
SiC content, wt%	0	2.5	5
Pressure, MPa	310	370	430
Temperature, °C	750	800	850

Table 2. Experimental factors and levels for TiH<sub>2</sub> process

#### 4.2 SDP process

Two types of Al atomised elemental powders from different suppliers (Baker and Riedel-de-Haen) have been tested and final experimentation Riedel-de-Haen aluminium powder has been used. The NaCl particles have been obtained by commonly culinary salt particles with different sizes. In order to control the cell sizes of the resulting foam, the commercial salt has been sieved into three groups with different particle diameters: <3, 3–4, and >4 mm. Large particles (70 wt%) have been mixed with medium size particles (30 wt%) in order to increasing porosity and to create an interconnected structures and because Li et al., 2003, found that the stiffness and the strength of foams increase with multi size cells with identical relative density. Before mixing, NaCl powder was dried in two steps: at 110 °C for 1 h in a heater and at 400 °C for 45 min in an electrical furnace as suggested in the literature (Sun & Zhao, 2005).

Magnesium addition in the mixture is due to oxidation problems. In fact the sintering of aluminium has always been considered problematic due to the oxide film present on the surface of powder particles. Trace additions of magnesium react with the oxide to form spinel. This breaks up the oxide, which facilitates sintering (Shaffer et al., 2001). The tested amount of Mg is 0.15 wt% of Al–NaCl compact.

The mixing is the second process step carried out: firstly through mixing of the Al with Mg powders to ensure the homogenization of metal components, and secondly with NaCl particles. Because of the need to ensure a continuous network of Al, an upper limit to NaCl fraction is necessary in the compact. Similarly there should be a lowest obtainable porosity to avoid trapped NaCl in the final structure which could lead to corrosion of the foams. A quantification of the content of NaCl has been carried out in the range 30–70 wt%. The powder compaction may be performed using different techniques (i.e. hot pressing, cold isostatic pressing). In these experiments powder compaction has been performed adding about 15 g of mixture (depending on composition) in a 25 or 32 mm diameter steel die followed by uniaxial cold pressing. A compaction in the range 100–600 MPa has been realised with a hydraulic motorised press. This wide range (different from literature data) is due to the mentioned problems associated with Al oxide films that have to be disrupted by mechanical deformation.

The sintering has been carried out in a pre-heated electric furnace at different temperatures in the range 650–700 °C (Al melting point is 660 °C). The sintering takes place in liquid state so the metal viscosity is enough to fill the spaces between NaCl particles. If the sintering



temperature is too low, the sintering can take too long time causing severe oxidation. If it is excessively high, some molten Al often oozes out from the surface of the compact to form globules. The same considerations involve the sintering time. The experiments have been carried out in the range 2–20 h. Moreover NaCl has a much lower thermal conductivity than Al, so the sintering time should be increased in presence of great salt amount. The sintering takes place at normal atmosphere and both ends of the mould are sealed in order to protect the compact from air effects. In the furnace the mould is positioned on a ceramic plate guaranteeing no temperature gradient along vertical direction as suggested by Arnold et al., 2003. Careful control of heating conditions during foaming is essential to obtain “quality” foams. After required time the samples have been cooled by air.

The salt dissolution takes place into two steps. Firstly, the sintered specimens have been placed into a running hot water bath to leach out the most of embedded NaCl particles. Secondly, samples have been immersed in an ultrasonic washer to ensure the complete removing of salt. The dissolution of the NaCl is a significant rate-limiting step: in large samples, the tortuosity of the foam structure means that it takes a long time to dissolve all the salt. Finally the samples have been washed by ethanol and dried. After screening the experimental full factorial plan reported in Table 3 have been carried out.

Factors	Levels		
Al fraction, wt%	30	40	50
Pressure, MPa	550	600	650
Time, h	2	3	4

Table 3. Experimental factors and levels for SDP process

### 4.3 MGI process

The aluminium liquid foam has been produced by melting aluminium ingots in a graphite crucible by an induction furnace. The applied foaming equipment has been specifically designed for these experiments. It is formed by a rotating system that allows to distribute the bubbles in the melt aluminium. The experimental apparatus is constituted by a drill, a shaft, a propeller, a panel of stainless steel, and a pipe for the gas injection. This system is designed, in a proper way that allows the gas coming out from the pipe, and diffusing homogeneously in the bath by the propeller (connected to the drill by the shaft). The presence of the stainless steel panel is of paramount importance for the success of the experiment reducing turbulences and vortex formations in the bath (Cingi, 1992). The process control for aluminium foam is regarded to be difficult due to its multivariability and invisibility. There is a close similarity of behaviour of bubbles in water and in melt aluminium, and such similarity is very useful. The bubbles generation and their rise to surface can be investigated much more easily in water and, then, the results can be applied to metal. In fact Reynolds number for both water and aluminium, within the turbulent regime, are close enough so that very similar properties of bubbles should be expected in these liquids. Therefore, to simulate bubble generation in foamed aluminium, a screening has been carried out also by means of this physical modelling (Oak et al., 2002).

Duralcan metal matrix composite (A356/SiC/20p) with a particle size of approximately 12  $\mu\text{m}$  have been used. Aluminium 356 has the following composition: Al 90.1–93.3 wt%, Si 6.5–7.5 wt%, Mg 0.2–0.45 wt%, Cu max 0.25%, Fe max 0.6%, Mn max 0.35%. Nitrogen is injected as foaming medium and temperature usually has been kept around 700 °C. After a

screening test using different combinations of parameters, the selected factors for deep observations have been: flow rate ( $Q$ ), spin speed of the propeller ( $N$ ), and silicon carbide particles content, because they shown the high influence on foam properties (mechanical and morphological).

The authors assumed these three reference main factors and verified their effects on cells area, cells wall thickness, relative density and plateau stress. The adopted standard approach for Design Of Experiments (DOE) has been the use of a full factorial method. Moreover, two replications of each combination were realised. Table 4 shows the levels of factors in this systematic investigation.

Factors	Levels		
	Gas flow rate, l/min	2	4
Spin speed, RPM	300	700	900
SiC content, wt%	10	15	20

Table 4. Experimental factors and levels for MGI process

## 5. Results discussion and comparisons

### 5.1 Morphological characterization

In the following section the results about morphological characterization will be compared and discussed. In this investigation the foams have been cut by a diamond saw ( $TiH_2$  and SDP) a by a metallographic saw (MGI). The major problems have been encountered with MGI foams due to the high level of SiC particles fraction; in fact, for 5 parameters combinations of experimental plan, were not possible to cut specimens and some of them were damaged during cutting. For  $TiH_2$  and SDP this problem was not so critical. The morphological parameters have been measured in 2D cross section by an images software tool and cell walls have been observed by a Scanning Electron Microscope Philips XL 20.

The most important parameters that influence the structure-sensitive properties of cellular metals are (order by their importance):

1. intrinsic properties (properties of cell wall material);
2. relative density;
3. type of cellular structure (open or closed cells) ;
4. in a closed-cell foam, the fraction of the solid contained in the cell nodes, edges of the cell faces;
5. irregularity or gradients in mass distribution;
6. the cell size and size distribution (including exceptional sizes) ;
7. shape of the cells and the anisotropy of cells (including exceptional shapes) ;
8. connectivity of cell edges;
9. defects, by which we mean buckled or broken cell walls.

Table 5 (Kriszt et al, 2002) defines a list of 20 structural parameters for describing the geometrical structure and microstructure of cellular materials. They were studied and measured for each foam types obtained with the  $TiH_2$ , SDP and MGI methods and Table 6 gives here a summary to compare the results.

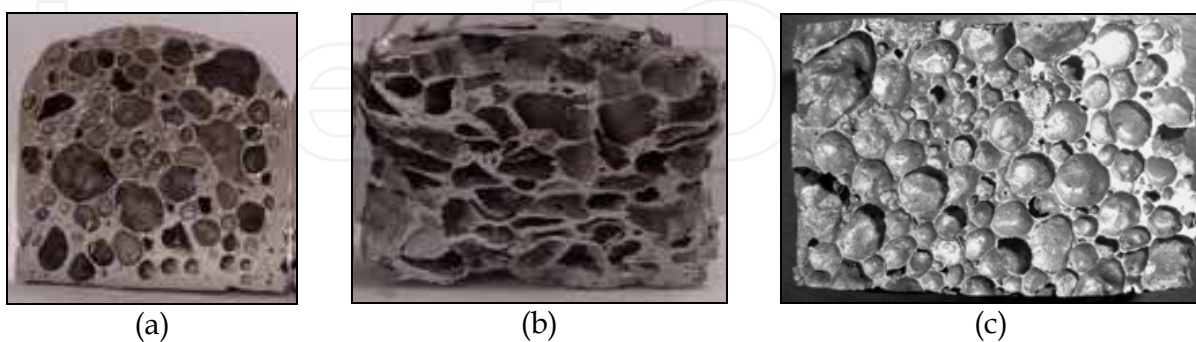
The significant differences in the structures of the  $TiH_2$ , SDP and MGI foams are function of the manufacturing process. The  $TiH_2$  foams realised in this investigation have a variety of little defects including: cracks, voids, shared cells and the pore area range is wide (Fig. 1a).

<b>Geometric structure</b>	<b>Pores</b>	<b>Cell skeleton</b>	<b>Microstructure</b>
open or close cells	Volume fraction	Thickness and length of cell walls	Dendritic structure
Arrangement of cells	Shape factor	Number/area of nodes	Grains
Neighborhood relation	Aspect ratio	Curvature/corrugation of cell walls	Chemical inhomogeneity
Stochastical models	Orientation		Micropores
Chemical composition	Size		Inclusions
			Precipitates
			Dislocations

Table 5. List of parameters for describing structure of metallic foam

<b>Property</b>	<b>TiH<sub>2</sub></b>	<b>SDP</b>	<b>MGI</b>
Alloy	Al, AlSi12	Al	A356/SiC/20p (AlSi7)
Density range, g/cm <sup>3</sup>	0.63-1.07 (0.77)	0.69-1.51 (0.99)	0.18-0.91 (0.51)
Equivalent diameter, mm	0.53-6.40 (1.85)	0.53-4.85 (1.54)	1.65-6.19 (2.81)
Pore area range, mm <sup>2</sup>	0.22-32 (3.77)	2-35 (4.92)	2.13-30.13 (6.89)
Cell wall thickness, mm	0.10-1.82 (0.63)	0.05-0.4 (0.21)	0.10-1.3 (0.40)
Circularity parameter	0.33-0.91 (0.62)	0.13-0.85 (0.46)	0.35-0.98 (0.81)

Table 6. Obtained properties of the three families of investigated aluminium foams

Fig. 13. Foam samples: a) TiH<sub>2</sub> b) SDP and c) MGI

In particular oversized pores as well mass concentrations can be observed and they can cause degradation if specific properties. Presence of microvoids has been observed also in open cell SDP foams but no cell edge curvature (Fig. 1b). Microstructural observation of the MGI foams has identified a number of irregularities. Many of the cell walls have some initial

curvature as well as small voids within them. There are local regions of higher density where the cell walls are thicker at the nodes. The shape and orientation of the cells in the lower density MGI foams vary throughout the thickness. In particular there is a density gradient throughout the thickness of the lower density MGI foams realised with moulds due to drainage of the liquid before solidification (Fig. 1c). Observations of cells of MGI foams indicate that they are slightly oriented due to the direction of rotation of the impeller during foaming. Also SDP foam has been found with elongated cells due to the compaction pressure. Only TiH<sub>2</sub> foams have roughly equiaxed cells. The cells area of the three types of foam is comparable. The foams, obtained by the processes TiH<sub>2</sub> and SDP, have a solid skin on the outer surfaces, which was removed before testing. Instead traditionally MGI foams does not have the skin; the implemented version, realised by mould, shows the presence of external skin originated by the contact with the ceramic moulds.

Obviously the structural performance of the three foams can be improved by reducing the occurrence of defects in the cell walls (curvature, porous inclusions, corrugations). Cell wall defects can cause drastic decreases in the mechanical properties of cellular materials. Cell wall curvature and porous inclusions within cell walls can be prevented by reducing the size distribution of cells and controlling better the foaming conditions in order to minimize convection etc. The face wrinkling in the cell walls of the MGI foams can be avoided by reducing the stresses applied to the foam immediately after solidification and by reducing the rate of cooling.

The major encountered problem has been the lack of standard test method: in particular architectural quality criteria should be better developed and nondestructive test method has to be well established and standardized. Moreover the determination of pore size, area, cell wall thickness and of all architectural features request intensive labor and it is time consuming. This can be a problem for future quality control in industrial environment.

Fig. 14 shows a diagram taken from Wadley (2002) reporting the range of cell size against relative density for different metal foam manufacturing methods. Red, orange and green lines represent the mean values of foams obtained in this investigation. The diagram does not show reference about SDP foam. TiH<sub>2</sub> foams enter in the expected range while MGI foams show better behaviour because for equal density they present lower cell size figures. Table 7 reports numerical figures showed in the diagram.

## 5.2 Mechanical characterization

After checking foam morphology, mechanical tests were carried out on the samples. Compressive strength has been chosen as evaluation criterion because it is relevant in the context of energy absorption of foam.. The tests were carried out using a machine model 5869 from Instron in quasi static condition, following the statements of the italian standard UNI 558 and of the standard test method for compressive properties of metal foam (Cymat Corporation, 2002) prepared by Cymat Company taking into account ASTM and DIN

Process	Average relative density	Average cell area (mm <sup>2</sup> )	Average cell size (mm)
TiH <sub>2</sub>	0.286	3.77	2.19
SDP	0.368	4.92	2.50
MGI	0.165	6.89	2.96

Table 7. Cell size and relative density

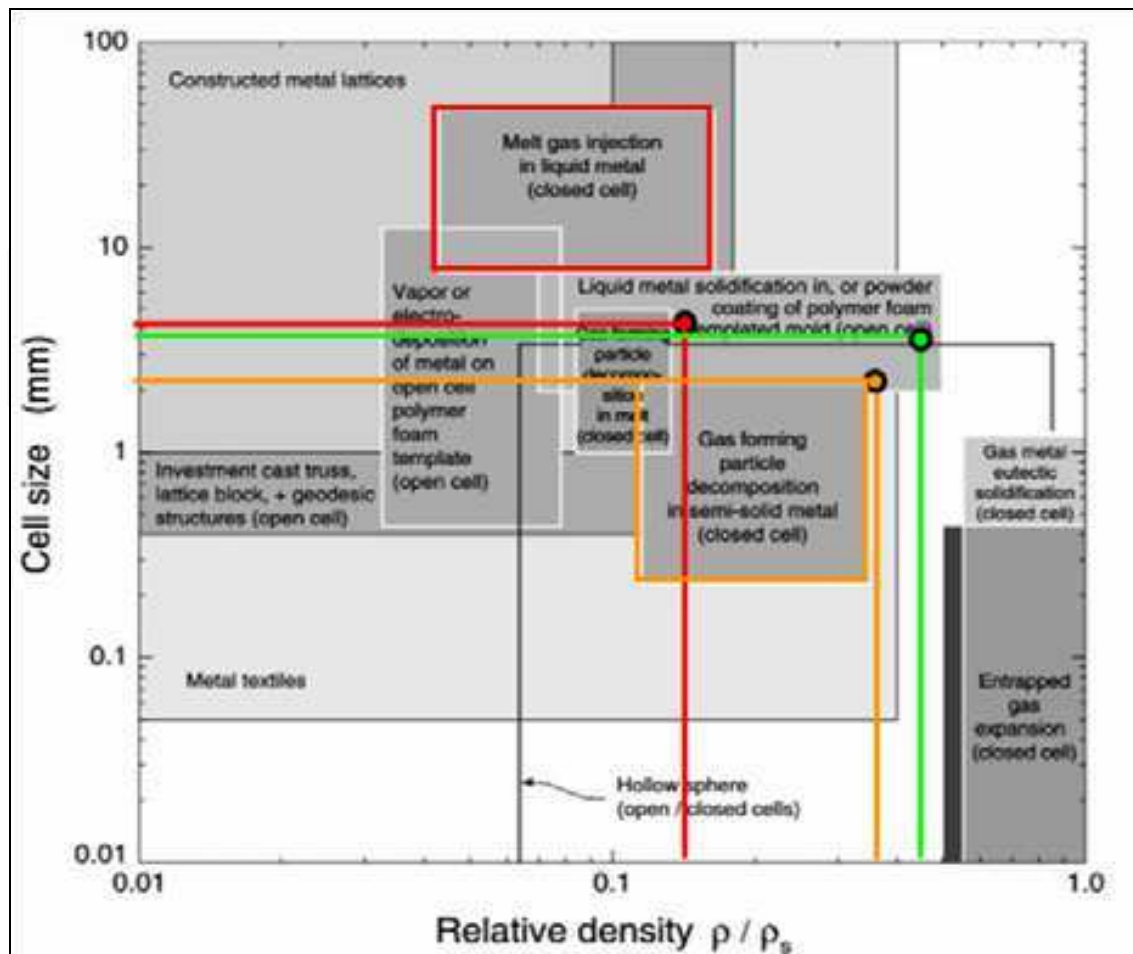


Fig. 14. The range of cell size and relative density for the different metal foam manufacturing methods (Wadley, 2002)

standards. In all mechanical testing, load and displacement were recorded using a National Instruments data acquisition unit and a personal computer. Stress was evaluated as the load per total area of the specimen, including porosity as suggested from Fusheng & Zhengang (1999). Likewise, strain, defined as a nominal value for the foam structure, is not the real strain experienced by the cell walls.

The compressive strength of foams have been measured in a number of studies and the results of several of these studies are summarized in Fig. 15 which plot the strength normalized by that of the solid cell wall material against the foam density normalized by the solid density (Wadley, 2002). In the figure with coloured lines and in Table 8 are reported the numerical figures founded in this investigation. There is an essential difference between open-cell and closed-cell structures. Open-cell foams are represented by a network of connected struts. The main deformation mechanism is bending of the cell edges and, at higher relative foam densities,  $\rho_r > 0.1$ , additional extension and compression of the edges. In closed-cell foams the cell walls between the cell edges and the membrane stresses in the cell walls also play a major role on the deformation mechanisms. Owing to the higher constraints, arising from the existence of cell walls, in closed-cell structures the Young's modulus is theoretically higher by several magnitudes compared with open-cell structures of the same relative density.



Process	Relative density	Relative compressive strength
TiH <sub>2</sub>	0.286	0.148
SDP	0.368	0.119
MGI	0.165	0.026

Table 8. Relative density and relative compressive strength

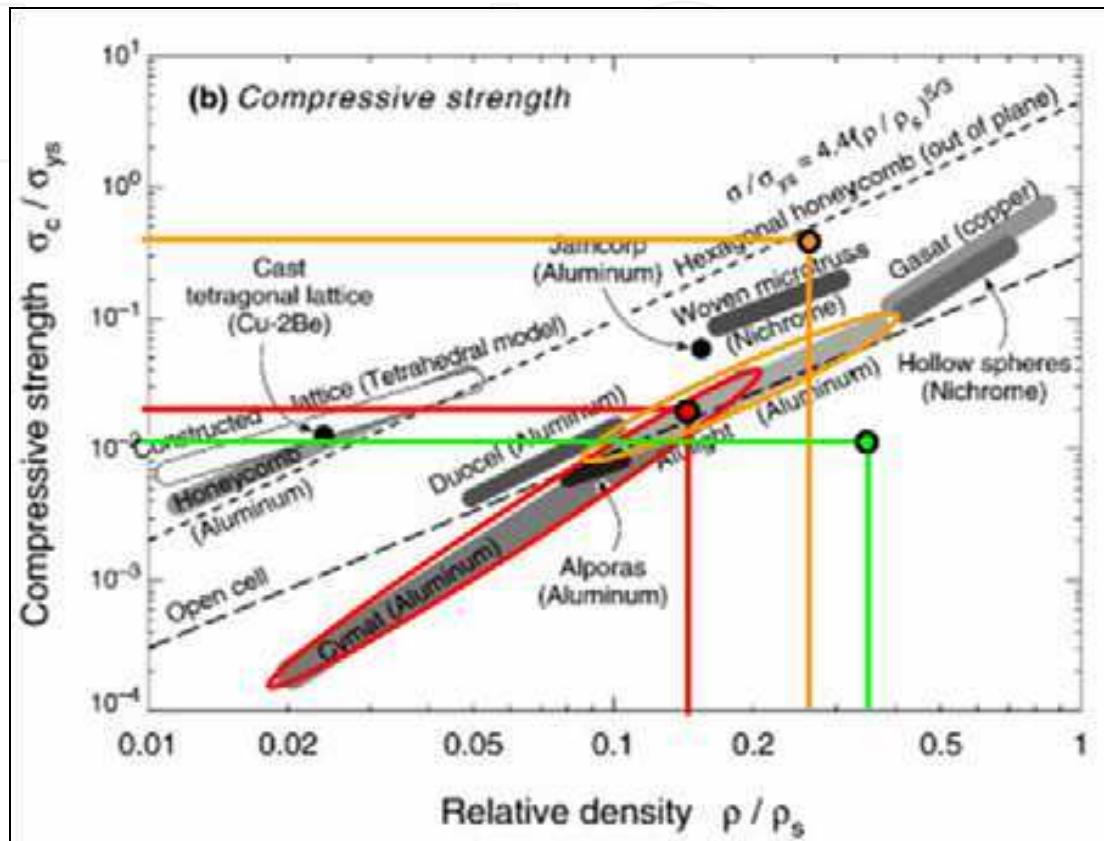


Fig. 15. A comparison of the mechanical properties of cellular metals: relative compressive strength plotted against relative density (Wadley, 2002)

The stress plateau for MGI foam is serrated (red lines Figs. 4-5), as a typical of a brittle foam: the serration correspond to fracture of cell walls. The initial yield point of MGI foam corresponds to debonding between the SiC particles and the aluminium matrix, which is then followed by ductile tearing of the matrix. Once number of cells have yielded, a deformation bands forms and subsequent cell collapse is by membrane tearing and the growth of interfacial cracks. The deformation of MGI foams under compressive loads is not spatially uniform deformation first occurs in the weakest region and propagated in this region until it is completely crushed or becomes fully densified. Then a second weakest band begins to deform until it is densified. This process is repeated in subsequent bands. Final densification occurs at 70% (relative density 0.25 - Fig. 16 a) and at 80% (relative density 0.31 - Fig. 16 b) of the strain.

The Plateau stress for the TiH<sub>2</sub> and SDP are gradually increases with strain until the densification occurs. But the TiH<sub>2</sub> foam shows a gently rising stress-strain curve in compression, and with no evidence of the formation of discrete crush bands (orange lines

Fig. 16). In contrast, the SDP foams crush by the sequential formation of crush bands at random sections of the foam. The foams are ductile in compression: they can undergo much larger strains than the tensile ductility of a fully dense aluminium alloy. This suggests that the cell edges deform mainly in bending, with the bending strains in the cells much less than the macroscopic uniaxial strain. For SDP foams final densification occurs at 40% of the strain while for TiH<sub>2</sub> foams at 50% of the strains.

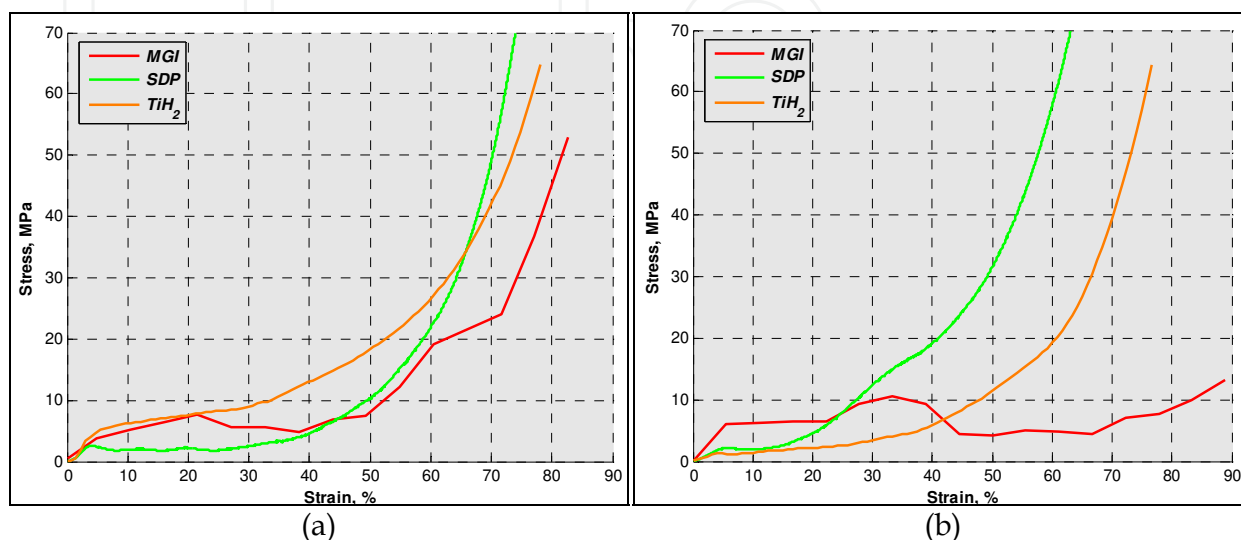


Fig. 16. Stress-strain diagram for 3 samples: a) relative density of 0.25 (TiH<sub>2</sub> 370-750-5, SDP 30-2-550, MGI 300-8-20), b) relative density of 0.31 (TiH<sub>2</sub> 310-850-0, SDP 30-3-600, MGI 300-2-15)

Ideal energy absorbers have a long flat stress-strain curve. The absorbers collapse plastically at a constant at a constant nominal stress, called the Plateau stress up to a limiting nominal strain (50% in this investigation). Energy absorbers for packaging and protection are chosen so that the plateau stress is just below that which will cause damage to the packaged object; the best choice is then the one which has the longest plateau, and therefore absorbs the most energy before reaching  $\epsilon_D$ . The area under the curve, roughly  $\sigma_{pl}\epsilon_D$ , measures the energy the foam can absorb, per unit initial volume, up to the end of the plateau. Foams which have a stress-strain curve like that shown in figure perform well in this function. Fig. 17 shows a plot of absorbed energy plotted against Plateau stress for some available metal foams. Foams retain the advantage that they are isotropic, absorbing energy equally well for any direction of impact. In the plot no reference point is presents for SDP or other similar processes. Mean values of Plateau stress and absorbed energy for TiH<sub>2</sub> and MGI foams show a behaviour slightly worst.

Method	Plateau stress (MPa)	Absorbed energy (MJm <sup>3</sup> )	Absorbed energy (Jg)
TiH <sub>2</sub>	16.0	3.56	4.61
SDP	26.0	5.69	5.72
MGI	5.85	2.16	4.76

Table 9. Plateau stress and absorbed energy

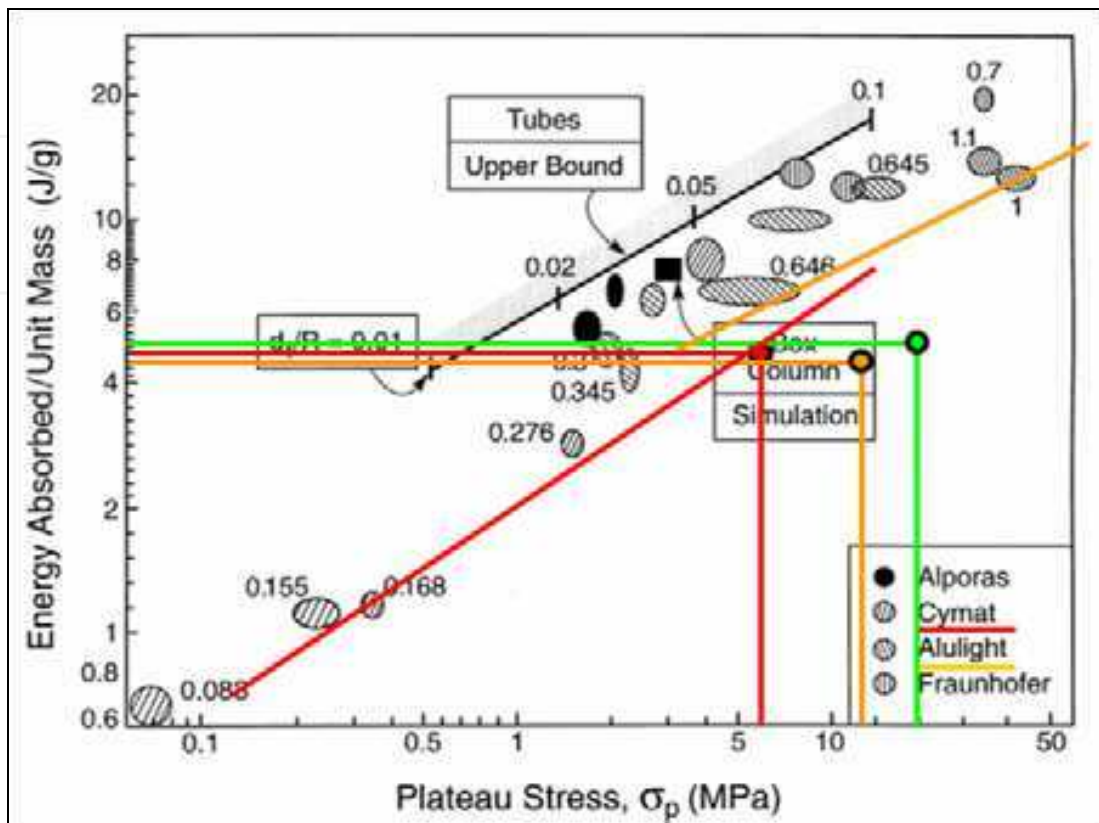


Fig. 17. Energy per unit mass against plateau stress for cellular Al alloys. Also shown is a comparison of the energy absorption per unit mass for Al tubes (Evans et al., 1999)

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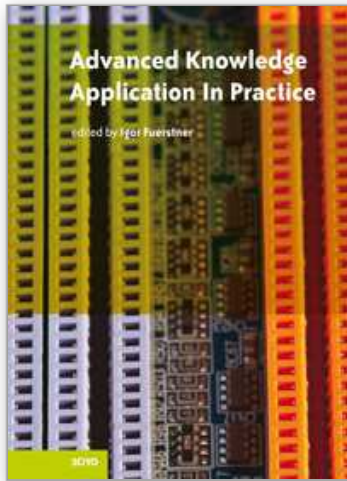


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Edited by Igor Fuerstner

ISBN 978-953-307-141-1

Hard cover, 378 pages

**Publisher** Sciyo

**Published online** 02, November, 2010

**Published in print edition** November, 2010

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