Aluminum Metallic Foams Made by Carbonate Foaming Agents

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Abstract
Recent developments in metal foams, especially aluminum, have produced a new class of lightweight materials at the side of the traditional ones such as polymers, ceramics or glass. The combination of a metallic character together with a cellular structure gives an interesting potential for a wide application of this material, particularly for high volume markets such as the automotive industry.

Increased demands concerning cost economy, passenger safety in automobiles and materials recycling all bring constructors now to use metal foams. Hereby it provides the additional environmental benefits from a potentially improved fuel economy and lower CO\textsubscript{2} emissions. Then, short review of metallic foam was done in the present paper.

The possibility of carbonate and hydroxide as foaming agent for Al-Si-Cu alloy by powder metallurgy route is studied, after preparation processes of metallic foams were briefly reviewed in the present paper.

It was done by measuring thermal decomposition behavior of foaming agents and evaluating cell structure of those aluminum foams. To obtain fine and homogenous cell structure in powder metallurgy route by using safer carbonate as foaming agent, it has made clear that importance of selecting foaming agent starting decomposition after melting of matrix. It is clearly different from TiH\textsubscript{2}-foam to grow coarse-rounded cell structure. From this point of view, MgCO\textsubscript{3} and CaMg(CO\textsubscript{3})\textsubscript{2} is suitable for matrix of Al-Si-Cu alloy. CaMg(CO\textsubscript{3})\textsubscript{2}-foam could expand to 1.19 in specific gravity, and keep homogeneous, fine and spherical cell structure.

Key words: Metallic foams, Carbonate, Aluminum alloy, Light materials

1. Introduction
Evolution of mankind is going hand in hand with a request for new constructional and tool materials. Until recently, the principal evolutionary forces were those relating to improved performance and functionality. Although polymers, ceramics, or composites have already been employed in various industrial applications the demand for stronger, stiffer and lighter materials is still growing. However, the production, disposal, and use of materials in products have environmental impacts throughout the whole product life cycle, and this fact can no longer be ignored.

Strong and stiff materials can be found also in nature but there they usually do not induce any pollution problems and recycling requirements. Therefore they can be a very good guide for the prospective development of new materials. The difference between strong natural and synthetic materials has very well been characterized by Ashby\textsuperscript{1}: “When modern man builds large load-bearing structures, he uses dense solids: steel, concrete, glass. When Nature does the same, she generally uses cellular materials: wood, bone, coral.” Really, natural materials are strong enough to withstand loads in the bones of running elephant or to carry the weight of a 100 m high redwood tree. Cellular structure of these materials...
provides the tool for the optimal combination of properties, e.g., realization of highest stiffness at minimum weight.

Many of today’s vehicles incorporate deformable energy absorbing elements within the vehicle structure\(^2\text{3}\). These elements, which represent the crushable zone, manage the collision energy for protection of the rigid passenger cell. Metallic foams have been researched recently because of their unique properties, like low density, energy-absorption, low thermal-conductivity.

Due to low density and novel physical, mechanical, thermal, electrical and acoustic properties metal foams already have a number of established and profitable market niches apart from car industry. Metal foams are used as heat exchangers, support structures for aerospace applications, electrodes for batteries, gas and fluid filters, acoustic absorbers, electrical applications etc.

The viability of metallic foam in a given application depends on the balance between its performance and its cost. At present all metal foams are produced in small quantities using time and labor-intensive methods, and all, relative to the solid materials from which they derive, are expensive. But it is not the present-day cost which is relevant; it is the cost which would be obtained where the process to be scaled and automated to meet the increased.

In the present paper, the possibility of carbonate and hydroxide as foaming agent for Al-Si-Cu alloy by PM route is studied with a brief review on metallic foam.

### 2. Various processes for preparation of metallic foams

Solid metallic foams are cellular materials that are made up from a framework of solid material enclosing and surrounding gas-filled voids (bubbles). In order to make a metallic foam it is firstly necessary to create a gaseous phase within the metal, then to rearrange the two-phase medium into a foam and finally to cool it beneath the melting point of the respective metal and obtain a solid cellular structure. A variety of different methods for the production of metallic foams are available and can be classified in accordance with the initial state of the metals to be foamed—Liquid, Powder, Ionized.

Only the most widespread methods will be described in detail. The main processing techniques with their basic materials specification are listed in Table 1 and 2.

Many processes of manufacturing metallic foam have been proposed. Most popular process uses titanium hydride (TiH\(_2\)) as foaming agent. The processes using TiH\(_2\) have two routes, melt processing and powder metallurgy (PM) routes. Typical melt processing route is “Alporas” method by Sinko Wire, melt foams with calcium as the viscosity-enhancing additive and TiH\(_2\) as foaming agent. In other side, typical PM route is “Fraunhofer” method by IFAM, aluminum matrix powder and TiH\(_2\) is hot-extruded, it is called precursor, then precursor is heated in closed die to foam for near-net shaping. In comparison with Alporas and Fraunhofer method, Alporas method is lower cost, but Fraunhofer method is better at controlling cell structure and near-net shaping.

### Table 1 Processing techniques for production of metallic foam and porous structures, together with associated basic material specifications

| Processing technique       | Solid phase composition | Porosity (%) | Cell size and type | Tradename or type          |
|----------------------------|-------------------------|--------------|--------------------|---------------------------|
| Melt-based routes          |                         |              |                    |                           |
| Gas injection              | Al alloys + ceramic particles | 80–98       | 3–25 mm closed     | ALCAN (also CYMAT and HYDRO) |
| In situ prior oxidation of the melt | Al+ (oxide particles) | 89–93        | 4.8 mm (mean) closed | ALPORAS®               |
| Delayed release gas generation | Al alloys + ceramic particles | 50–96    | 0.8–3 mm closed    | FORMGRIP®               |
| Gas-eutectic reaction      | Ni, Cu, Mg, Al and others | 5–75        | 5 m–10 mm closed/open | GASAR, Lotus       |
| Infiltration and replication | 6101 and A356           | 88–92        | 0.5–4.3 mm open    | DUOCEL®                  |
Early attempts concentrated on vaporization different foaming agents in liquid metal with the gas serving as a blowing agent. These ideas were developed and applied and foamed aluminum was firstly produced in 1951. According to this process, metal hydrides such as TiH₂ or ZrH₂ were put into the melt. Next, the hydrides were decomposed under heating and the evolved gases caused the molten metal to foam. After foaming the resultant body was cooled forming a cellular solid structure.

The techniques mentioned above meet some problems as follows:

1. a non-uniform cellular structure arises, because the foaming process is rather difficult to control;
2. a relatively short time interval between adding a foaming agent to the molten metal and foam formation which rendered metal casting difficult;

These problems have been treated as follows:

- Particles of the foaming agent were rapidly dispersed through the molten metal mass by high speed mixing, which bubbles growth as was prevented by increasing the melt viscosity utilizing alloys with a wide difference between the alloy solidus and liquidus temperatures or by introducing viscosity-increasing agents.

Numerous attempts have been also made to overcome the structural weakness of foamed metals. All these methods improved the foaming but could not sufficiently optimize yield foams of a satisfactory quality and cost.

Currently there are two ways for direct foaming of metallic melts.

Perhaps the simplest method of melt foaming is to inject gas into a melt and then arrange for it solidification in such a way that the bubbles become entrapped in the material matrix. This approach was developed by Alcan International Ltd., Canada, Hydro Aluminum, Norway and licensed to Cymat Aluminum Corp., Ontario. The process scheme is shown in Figure 1. The starting material is aluminum containing solid substances (silicon carbide, aluminum oxide, etc.) to increase the melt viscosity and stabilize gas bubbles. The liquid melt is then foamed by blowing gas, usually air, using rotating impeller. The bubble size can be controlled by adjusting the gas flow rate, the impeller design and the speed of impeller rotation. The bubbles float up to the surface of the liquid where foam can be pulled off e.g. by conveyor belt. The collected foam is cut into the required shape after cooling. Porosity ranges from 80 to 97%. The advantage of the process is its ability of producing large volumes at rather low cost compared to other metallic foams. It is not also necessary to add an expensive gas-generating compound or to conduct the foaming in a restricted melt temperature range and processing time.

While the process is simple and cheap, the large and variable cell sizes constitutes a significant problem for many potential applications. The possible disadvantage is the high content of ceramic particles that cause difficulties during foam ma-

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**Table 2** Processing techniques for production of metallic foam and porous structures, together with associated basic material specifications

| Processing technique                  | Solid phase composition      | Porosity (%) | Cell size and type | Tradename or type   |
|---------------------------------------|------------------------------|--------------|--------------------|---------------------|
| Baking of powder-blended consolidated precursor | Al alloys + residual oxide particles | 63–89        | 1–8 mm closed      | ALULIGHT FOAMINAL (IFAM) ALUFOAM |
| Baking of entrapped gas precursor     | Ti–6Al–4V                    | 20–40        | 10–100 mm closed   | “LDC sandwich”      |
| Sintering                             | Steel, Ti–, and Ni–based hollow spheres | 65–87        | 0.5–6 mm closed    | Hollow sphere structures |

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**Fig. 1** A schematic depiction of the Alcan process
chining. Also the problem of wetting the particles by the melt and their nonuniform distribution within the melt should be solved. But the refractory particles play a critical role. The objective is to ensure that they adhere to the gas-liquid interface within the foam, so as to stabilize the bubbles and inhibit their movement and further coalescence in the melt.

Addition of a foaming agent (TiH₂, ZrH₂) into the melt instead of blowing gas is the second way for foaming melts. A novel technique (tradename ALPORAS) (Fig. 2), developed by Shinko Wire Company Ltd., Japan differs from the previous one by using calcium additives and stirring the mixture in an ambient atmosphere. These steps increase melt viscosity, prevent the occurrence of bubble flotation and ensure a relatively uniform distribution. Foam slabs are obtained after cooling and can be cut into sheets of the required thickness. Typical foam porosity is in the range of about 84–93%, with a mean cell diameter of about 4.8 mm.

The concept of foaming agent (TiH₂) pre-treatment was developed by Gergely and Clyne. The FORMGRIP process provides relatively easy control over the kinetics of hydrogen evolution by manipulating parameters such as the foaming agent pre-treatment, thermal histories during baking and composite melt viscosities (ceramic particle content and size).

Porous materials (metal, alloy, or ceramic), termed the GASAR (an acronym from the Russian for “gas reinforced”) are produced by utilizing a eutectic reaction in melts which are already supersaturated with hydrogen. Such a melt is cooled below the eutectic point, resulting in concurrent formation of both a gaseous and a solid phase from the liquid. Nakajima et. al also successfully produced lotus-structured porous metals with elongated pores.

The advantage of this method is the possibility of making metal foams with medium and high melting point (copper, nickel, iron) and with elongated pores. The maximum porosity achieved is 75%.

2.2 Powder-based routes

The second group includes methods to make highly porous metallic structures using metal powders instead of the liquid metal. As a starting material in some of these processes the powders are processed into a compact precursor material prior to the actual foaming step (Fraunhofer and “Alulight” processes, Gas entrapment technique). Early methods used powders or slurry, polymer mixtures and filler materials directly.

According to IFAM process, commercial aluminum powder is mixed with foaming agent powder. The mixture then compacted to a semi-finished product and worked into sheets, profiles etc. in order to improve the flow conditions during its eventual foaming inside moulds.

Heat treatment at temperatures near the melting point of the matrix metal causes the foaming agent to decompose and precursor material to expand yielding a highly porous structure. This process can be used to manufacture different products such as near-net shaped parts, sandwich panels, and tubes. The density of aluminum foam typically ranges from 0.5 to 1 g/cm³. Distribution of cell sizes and shapes is at random.

“Alulight” is another novel melt processing technique, combining melt and powder routes in metallic foam production. At first a pre-treated powder (TiH₂) is distributed into an aluminum alloy. Allowing this melt to solidify produces a precursor material, having a relatively low porosity level (20–25%). At the second stage, the precursor material is heated into a semi-liquid state, when progressive evolution of hydrogen converts it into a cellular structure. To obtain complex 3D-shaped foam the precursor can be injected in a controlled manner into the cavity with the desired shape.

Foamed metal can also be made by compressing powders to a precursor material and allowing gas to be entrapped in the metal structure. Heating the precursor leads to an expansion of the metal due to the internal pressure created by the entrapped gas.

TiH₂ is most popular foaming agent because its decomposition temperature is closed to melting temperature of aluminum alloys. But TiH₂ has a problem which is high cost and dangerous of hydride. So, lower cost and safer foaming agent is expected.

One of the authors reported the method to use calcium carbonate (CaCO₃) as foaming agent by melt processing route. CaCO₃ is low cost and...
very stable. But PM route used carbonate as foaming agent has been less studied.

On PM route, precursor is heated in metal die, so lower foaming temperature is preferred because of low input energy and heat resistivity of die material. In aluminum alloy, Al-Si-Cu alloy has lower melting temperature than other alloys because of Al-Si and Al-Si-Cu eutectic.

3. Experimental

Air-atomized powder of Al-10.8Si-2.4Cu (mass%), assigned here as AlSiCu, was used to make precursor. Powder is sieved under 150 μm.

TiH₂, magnesium hydroxide (Mg(OH)₂), magnesium carbonate hydroxide (4MgCO₃·Mg(OH)₂·5H₂O), magnesium carbonate (MgCO₃), dolomite (CaMg(CO₃)₂), calcium carbonate (CaCO₃) were investigated as foaming agent. Particle size and chemical reaction are given in Table 3. TiH₂ releases Hydrogen (H₂), MgOH₂ releases water vapor (H₂O), 4MgCO₃·Mg(OH)₂·5H₂O releases H₂O and carbon dioxide (CO₂), MgCO₃, CaMg(CO₃)₂ and CaCO₃ release CO₂.

Dolomite (CaMg(CO₃)₂) is mineral made of CaCO₃ and MgCO₃, so composition and impurity depend on area of mine. CaMg(CO₃)₂ were obtained from Murakashi Lime Industry Co., Ltd. (ML), Yoshizawa Lime Industry Co., Ltd. (YL) and JFE MINERAL COMPANY, LTD. (JFE). Table 4 gives catalog data and measured composition ratio.

TG-DTA was used to examine melting of AlSiCu and thermal decomposition of foaming agents in argon flow. Alumina pans were used, and heating rate was 10 K/min.

Precursors were made by hot-extrusion using mixed powder of AlSiCu and foaming agents. Extrusion temperature was 623 K, extrusion ratio was 18, and cross section size was 5 × 15 mm. Precursors were cut 30 mm long, measured by K type thermal couple, heated to certain temperatures in furnace. Aluminum foams were cooled in air, measured specific gravity by Archimedes method, and observed cell structure by optical microscope.

4. Results and Discussion

4.1 Decomposition of foaming agent

A melting of AlSiCu alloy starts from 793 K and completes 853 K. Foaming agent is necessary to decompose between 793 and 853 K for using AlSiCu as matrix.

TG-DTA curves of several carbonate foaming agents have been measured in this study.

Table 3: Candidates of foaming agents in Carbonate and Hydroxide

| Foaming agent | Average particle size, d/μm | Gas | Chemical reaction |
|---------------|-----------------------------|-----|------------------|
| Titanium hydride | TiH₂ | 26 | H₂ | TiH₂ = Ti + H₂ |
| Magnesium hydroxide | Mg(OH)₂ | 7.4 | H₂O | Mg(OH)₂ = MgO + H₂O |
| Magnesium carbonate hydroxide | 4MgCO₃·Mg(OH)₂·5H₂O | 11 | H₂O, CO₂ | 4MgCO₃ = 4MgO + 4CO₂ |
| Magnesium carbonate | MgCO₃ | 11 | CO₂ | MgCO₃ = MgO + CO₂ |
| Dolomite | CaMg(CO₃)₂ | — | CO₂ | CaMg(CO₃)₂ = CaCO₃ + MgO + CO₂ |
| Calcium carbonate | CaCO₃ | 13.5 | CO₂ | CaCO₃ = CaO + CO₂ |

Table 4: Dolomite minerals used in the experiments

| Supplier | Size | Composition (catalog) | Composition (measured, mass%) |
|----------|------|-----------------------|--------------------------------|
| Murakashi Lime Industry | 3.5 μm (average) | Ca>20%, Mg>10% | CaO: 70.8, MgO: 28.1 |
| Yoshizawa Lime Industry | Coarse | — | CaO: 73.7, MgO: 23.3 |
| JFE Mineral | <2.36 mm | CaO = 34.5%, MgO = 18.3% | CaO: 73.8, MgO: 25.0 |
MgCO$_3$ decomposes from 793 K to 993 K. It is close to melting temperature of AlSiCu. So it is suitable for foaming agent of AlSiCu. CaCO$_3$ decomposes over 993 K, hence it is not appropriate.

TG-DTA curves of CaMg(CO$_3$)$_2$. YL-CaMg(CO$_3$)$_2$ are shown in Fig. 4. There are two-stage of decomposition. First stage starts from 713 K, and second stage starts from 1013 K. On the other hand, ML and JFE-CaMg(CO$_3$)$_2$ decompose slowly from 773 K, then decompose drastically from 1013 K. YL-CaMg(CO$_3$)$_2$ was a little wet, so first stage was assumed releasing H$_2$O or decomposition of MgCO$_3$. The decomposition-reaction of CaMg(CO$_3$)$_2$ has two stages as shown in Table 1. But it is reported that it is differed by atmosphere$^{21}$, so it could be single-continuous decomposition obtained in argon flow. ML-CaMg(CO$_3$)$_2$ was selected because particle is fine and homogenous, and decomposition temperature is closed to melting temperature of AlSiCu.

4.2 Evaluation of foaming ability

Precursors were made by using 4 kind of foaming agent. MgCO$_3$ and CaMg(CO$_3$)$_2$ is suitable for AlSiCu, for comparison, 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O decomposing lower temperature and TiH$_2$ were chosen.

Distribution of foaming agents in precursor will be influence on cell structure of foam, so it would be important for industrial fabrication. Fig. 3 shows cross sectional views of precursors. Particles of TiH$_2$ and 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O are coarse and agglomerate, on the other hand MgCO$_3$ and CaMg(CO$_3$)$_2$ are fine and not agglomerate. Dispersions of TiH$_2$ and 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O in precursor are heterogeneous, observed both coarse and fine particles. MgCO$_3$ and CaMg(CO$_3$)$_2$ disperse homogeneously.

Fig. 4 shows aluminum foams made by 4 kinds of precursor. Aluminum foams were cooled when expansion started at 843 K, and expanded to 1.0~1.2 in specific gravity.

TiH$_2$-foam has fine cell structure first at 843 K, then grows coarse-rounded cell structure at 856 K. On the other hand, 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O foam expanded under 1.2 in specific gravity at 863 K like TiH$_2$. But cell structure is torn along with extruded direction not like TiH$_2$.

MgCO$_3$ and CaMg(CO$_3$)$_2$ show homogenous cell structure. MgCO$_3$ expanded under 1.2 in specific gravity at lower temperature than CaMg(CO$_3$)$_2$. But MgCO$_3$ has coarsened cell partly, CaMg(CO$_3$)$_2$ has better homogeneity.

4.3 Influence of foaming agent on cell structure

Different cell structures had obtained from each foaming agent as shown in Fig. 5. The factors to determine cell structure would be the type of re-
leasing gas and decomposition temperature.

Fig. 6 shows melting rate of AlSiCu and decomposition rate of foaming agent. The decomposition rate of gas was calculated from mass reduction of TG-DTA. TiH$_2$ and 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O have low starting decomposition temperature. They have released over half of gas before melting of AlSiCu. Released gas have torn boundary of aluminum powder extended as fibrous by extrusion, because solid phase has higher strength than solid-liquid phase, and torn cells combine and grow along with extrusion direction. While temperature rises, TiH$_2$-foam grows coarse-rounded cell structure because the cell expanded by H$_2$ of reaction gas is easily combined and deformed to decrease surface energy of cell wall.

On the other hand, 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O foam grows as torn cell structure. It releases H$_2$O and CO$_2$ of oxidizing gas, and oxidize cell wall to be stable. That stable cell wall prevented cell to be connected and spherical, hence it leads coarse-torn cell structure. One of the authors reported that carbonate-foam had oxidized and made cell wall stable$^{20}$. MgCO$_3$ and CaMg(CO$_3$)$_2$ have higher decomposition temperature, so rate of decomposition gas is less than 1% when AlSiCu starts melting. Main gas release starts with increasing rate of melt, so it means lower flow stress of AlSiCu. These cells had not torn fibrous aluminum powder boundary, hence they could be flat or spherical. Even after expansion, they kept same shape and homogeneous because of stable cell wall.

To confirm the influence of gas releasing volume on cell structure, the volume and radius of gas released from a particle at 843 K are given in Table 3. Gas volume of 1 mol is calculated by using 0.0224 m$^3$ in standard conditions. 4MgCO$_3$·Mg(OH)$_2$·5H$_2$O have large volume because most of gas have already released. MgCO$_3$ and CaMg(CO$_3$)$_2$ have small volume of gas because most of gas have not released yet.

CaMg(CO$_3$)$_2$ has smaller radius of gas than MgCO$_3$, it depends on not only gas decomposition but also particle size. This smaller radius of oxidizing gas are estimated the reason of fine and spherical cell structure of CaMg(CO$_3$)$_2$-foam.

5. Conclusion

A review of metallic foam was shown to understand role of it in materials science. Metallic foams show a good performance as light materials and use in vehicles, houses and buildings. Especially PM route products have a possibility to increase the market.

To obtain fine and homogenous cell structure in PM route by using safer carbonate as foaming agent, it has made clear that importance of selecting foaming agent starting decomposition after melting of matrix. It is clearly different from TiH$_2$-foam to expand coarse-rounded cell structure.

From this point of view, MgCO$_3$ and CaMg(CO$_3$)$_2$ from Murakashi Lime Industry Co., Ltd. is suitable for matrix of AlSiCu. CaMg(CO$_3$)$_2$-foam could expand to 1.19 in specific gravity, and keep homogeneous, fine and spherical cell structure.
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