# Experimental analysis of cold sprayed precursors for closed-cells aluminum foams

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Abstract. Metal foams are a relatively new class of materials with many interesting combinations of physical and mechanical properties. Among them, the closed-cells aluminum foams are the most interesting for structural applications in the aerospace industry. There exist different methods to produce metal foams. An innovative manufacturing technique was developed in this study, proving the possibility to produce closed-cells aluminum foams through the cold spray additive manufacturing technology (CS). In particular, two different sets of samples were produced by varying the cold spray process parameters aiming at: i) studying the advantages and the issues of cold spray for manufacturing precursors for metal foams; ii) analyzing the correlation among the CS printing strategy and parameters, the foaming process and the physical and the morphological characteristics of the resulting foams; iii) finally, finding appropriate conditions to form closed-cells aluminum foams via CS.

## Introduction

It is a matter of fact that, in the aerospace industry, the main trend at the design stage is currently directed toward the wider employment of lightweight materials, due to obvious environmental and economic reasons [1]. In this scenario, the closed-cells metal foams are rapidly emerging as a new class of advanced materials with very low specific weight and novel physical, mechanical, thermal, electrical and acoustic properties due to their cellular structure consisting of solid metal containing a large volume fraction of gas-filled pores [2].

Metal foams of different metals are available, such as: aluminum, nickel, magnesium, lead, zinc, copper, bronze, titanium, steel and even gold [3]. The more used in structural applications are aluminum foams that are characterized by low density coupled with high energy absorption capacity, high specific stiffness and reduced thermal and electrical conductivity; these materials can be used as core for sandwich structures, internal anti-buckling reinforcement for thin walled structures and so on [4,5].

The closed-cells aluminum foams can be manufactured through several methods [6]. The gasreleasing particle decomposition in semi-solids process, which is known as powder compact melting technique (PCMT), starts from a compacted powders mixture of aluminum alloy and a blowing agent in form of fine particles, obtaining the so-called foamable solid precursor [7]. Heating the precursor to a little above the melting temperature of the alloy leads the compacted metal matrix in a semi-liquid viscous state; the foaming agent decomposes and releases hydrogen,

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which blows bubbles in the soft alloy. Under these conditions, the released gas forces the melting precursor material to expand, modelling a highly porous structure.

In order to ensure satisfactory foaming results, appropriately starting powders have to be chosen [8]. The most suitable materials for foaming are pure aluminum or cast alloys (e.g. AlSi10Mg and AlSi12) coupled with titanium hydride (TiH<sub>2</sub>), which is the foaming agent. Several other foaming parameters, which have to be suitably controlled, influence the final foam quality. The most important are powder mixing composition, particles dimensions and shape, distribution of blowing agent within the metal matrix, compaction pressure, time and temperature of foaming and heating rates [9].

Based on the mechanisms ruling PCMT, an innovative manufacturing technology was developed by the authors in this study, proving the possibility to produce closed-cells aluminum foams through the cold spray additive manufacturing technology. The problems encountering when using the traditional PCMT-based manufacturing processes for foams can be overcome through cold spray [10].

CS is a significantly greener, low-temperature, non-combustion, and scalable alternative process with an extraordinary potential. The bonding concept in CS is based on kinetic rather than thermal energy. During CS, feedstock powder particles (5-50  $\mu$ m) accelerated to supersonic velocities (300-1200 m/s) by preheated compressed gas passing through a convergent-divergent nozzle, undergo severe plastic deformation upon impact and bond to the substrate [11]. CS, as a "cold" technique, can be used to process thermally sensitive materials [12,13], like the powder-based solid precursors for metal foams. The key idea is spraying the powder mixture on a metallic substrate and then carry out the foaming process in order to obtain the final foamed component. The shape of the precursor can be ruled by using a complex shaped substrate or by imposing a complex trajectory at the cold spray gun.

In line with this topic and with the scope to explore in details the developed manufacturing process, two different sets of samples were produced by varying the cold spray process parameters aiming at: i) studying the advantages and the issues of cold spray for manufacturing precursors for metal foams; ii) analyzing the correlation among the CS printing strategy and parameters, the foaming process and the physical and the morphological characteristics of the resulting foams; iii) finally, finding appropriate conditions to form closed-cells aluminum foams via CS.

For this scope, AlSi10Mg fine powders were mixed with titanium hydride particles to form the powder mixture precursors. Then, the low-pressure cold spray facility was used for the powder deposition by using different CS printing parameters; hence, the foaming process was carried out. SEM observations of the cross-sections of both the precursors and the resulting foams were carried out, pointing out the effectiveness of the process and the critical issues to be faced when cold spraying precursors for metal foams.

#### **Materials and Methods**

Micron sized powders of aluminum alloy AlSi10Mg and titanium hydride TiH<sub>2</sub>, provided by LPW South Europe and Dymet, respectively, were used in this experimentation. The particle mean size of AlSi10Mg powder is 25  $\mu$ m, while the mean diameter of TiH<sub>2</sub> particles is 10  $\mu$ m.

The powder mixture was obtained through the sound assisted fluidization bed processes; the use of fluidization makes it possible to handle and process large quantities of powders, so obtaining a mixing to the scale of the primary particles [14]. The fluidization and mixing tests of these cohesive fine powders were performed by the experimental apparatus schematically reported in Fig. 1. It consists of a metallic fluidization column equipped with a porous plate gas distributor to maximize the uniformity of the gas flow rate entering the column. The acoustic field is introduced inside the column through a sound wave guide located at the top of the column. Pure dry nitrogen from a compressed tank is used as fluidizing gas. All the tests were carried out at room temperature and ambient pressure conditions. Both the preliminary fluidization/mixing tests and the

Materials Research Proceedings 28 (2023) 49-56

preparation of the final AlSi10Mg-TiH<sub>2</sub> mixtures to be used to produce the metal foam precursors were performed in this experimental apparatus.



Fig. 1. Sound assisted fluidization bed process apparatus.

A mixture with a 2.5 wt.% of TiH<sub>2</sub> was prepared. The following fluidization process parameters were used based on preliminary fluidization/mixing tests: i) gas flow rate: 70 l/min; ii) acoustic field: 80 dB-150 Hz; fluidization time: 30 min. The SEM picture (Fig. 2) shows the effective mixing achieved with the blowing agent particles homogeneously dispersed within aluminum alloy powder.



Fig. 2. SEM picture showing AlSi10Mg-TiH<sub>2</sub> mixture achieved through sound assisted fluidization bed process (2.5 wt.% of TiH<sub>2</sub>, nominally).

The low-pressure cold spray facility was used for spraying the powder mixture constituting the foamable precursors. The spraying parameters such as fluid temperature and fluid pressure, stand-off distance (the distance between the substrate and the nozzle) and horizontal speed nozzle across the substrate were chosen after a preliminary experimental campaign that is not discussed in this paper for the sake of brevity. Two different sets of samples were produced by varying the cold

spray process parameters, in agreement to what reported in Table 1. The first raw of Table 1 reports the CS process parameters  $(CS_L)$  used to obtain less compact precursors characterized by internal microporosities that should promote the bubbles growth and the formation of lightweight materials. The second raw reports the CS process parameters  $(CS_H)$  used to manufacture more compacted precursors.

	Gas used	Gas temperature [°C]	Gas pressure [bar]	Stand-off distance (SoD) [mm]	Speed nozzle [mm/s]
$CS_L$	Air	400	6	30	1.0
CS <sub>H</sub>	Air	600	7	5	1.0

Table 1. Deposition process main parameters.

The mixture was sprayed on a C40 thin steel plate producing a 30x40 nominal plane shape. One single pass was imposed to the spray gun, in agreement with the strategy shown in Fig. 3a. Macrographs of the foamable solid precursors manufactured via cold spray under  $CS_L$  and  $CS_H$  process conditions are shown in Fig. 3b and Fig. 3c, respectively.



Fig. 3. a) Cold spray deposition strategy; b) macrograph of foamable solid precursor produced with  $CS_L$  process parameters; c) macrograph of foamable solid precursor produced with  $CS_H$  process parameters.

All the precursors made via cold spray were inserted within a preheated furnace set at a temperature of 660°C for the foaming process. The time needed for expansion was estimated in the range of 4-6 minutes; afterwards each sample has been taken out from the furnace and cooled at room temperature.

The cold sprayed precursors (obtained via  $CS_L$  and  $CS_H$  process parameters) before foaming as well as those resulting from the foaming process were cut for the cross-section observations. Hence, all the specimens were mounted and prepared according to the international ASTM standards for metallographic analyses through SEM microscopy. The blowing agent distribution after the cold spray deposition was analyzed, the shape of the cell and the expansion of the foam

were examined for the foamed samples. EDS inspections were also carried out with the scope to analyze the phenomena resulting from the foaming process, highlighting the critical issues of the manufacturing technology, for leading optimized process conditions.

#### **Results and Discussion**

The results from the SEM observations of the cross-sections of the cold sprayed precursors before foaming obtained by using  $CS_L$  and  $CS_H$  process parameters are reported in Fig. 4 and Fig. 5, respectively.



Fig. 4. SEM observations of the cross-sections of the cold sprayed precursors before foaming obtained by using CS<sub>L</sub> process parameters at a) 150x, b) 500x.



Fig. 5. SEM observations of the cross-sections of the cold sprayed precursors before foaming obtained by using CS<sub>H</sub> process parameters at a) 150x, b) 500x.

From Fig. 4a the thickness of the specimen was estimated to be equal to about 800  $\mu$ m. Moreover, voids and microporosities are well visible with the particles that are not completely deformed during the impact. This is clearer by looking the picture in Fig. 4b at 500x magnification. The white particles are the fragile TiH<sub>2</sub> powders that keep entrapped among aluminum particles, forming cluster and agglomerations homogeneously dispersed within the metal powders.

The cold sprayed precursors obtained by using  $CS_H$  process parameters are denser and more compact, as expected from literature [15], the particles deformed and flattened, and the coating thickness reduced at about 500  $\mu$ m, due to particles compactness (Fig. 5a). The higher magnification picture in Fig. 5b shows a precursor appearing quite dense and free of porosity, with

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Materials Research Proceedings 28 (2023) 49-56	https://doi.org/10.21741/9781644902479-6

the foaming agent particles (the lighter zones in the figure) homogeneously dispersed within the aluminum-silicon matrix. In fact, as proved in literature [16,17], the cold spray technology can create metal matrix composite depositions by mixing fragile materials with ductile material particles that deform and promote the coating formation and grow-up. Moreover, a stratification of TiH<sub>2</sub> particles, related to technology employed, can be identified.

Anyway, in both the examined cases, EDS analyses confirmed a weight percentage of  $TiH_2$  within the coatings equal to  $2.5\pm0.5$  wt. %., proving the capability of CS to deposit the AlSi10Mg-TiH<sub>2</sub> mixture of precursor.

Fig. 6 shows the micrographs of the cross-sections of the aluminum foams obtained starting from the above-described precursors.



Fig. 6. Micrographs of the cross-sections of aluminum foams produced starting from precursors obtained by using: a) CS<sub>L</sub> process parameters; b) CS<sub>H</sub> process parameters.

It can be seen from Fig. 6a that no foaming occurs when the precursor is not quite compacted, and the CS process parameters are not properly set. In this case, the foaming agent was not capable of expanding the thick and so-already existing porous structure. The possible reason is that the oxygen entering within the porous material oxides completely the metal matrix that cannot expand and form the bubbles under the force of hydrogen released by  $TiH_2$  dissociation mechanism. On the contrary, it can be seen from Fig. 6b that the material expands when the precursor is denser and well compacted, leading to a foam relative density [3] equal to 0.35, with the foam percentage porosity of 65%. Under these conditions, the aluminum matrix melts, and the foaming agents blows to form the closed-cells aluminum foams.

#### **Summary**

Based on the experimental results presented and discussed in the previous sections, the following conclusions could be drawn:

- The cold spray technology seems to be an effective technique to produce precursors for closed-cells aluminum foams.
- The sound assisted fluidization bad processes can be used to mix effectively micron size AlSi10Mg and TiH<sub>2</sub> particles forming the precursor, under the following process parameters: i) gas flow rate: 70 l/min; ii) acoustic field: 80 dB-150 Hz; fluidization time: 30 min.
- An existing porous structure for the precursor does not enhance the foaming process and the bubble formation.

• The CS process parameters need to be properly set (appropriate process parameters were found here: gas temperature: 600°C, gas pressure: 7 bar, SoD: 5 mm) to produce dense, compact and free of porosity metallic precursors, which are the characteristics to be ensured for a successful foaming.

### References

[1] G. Sun, D. Chen, G. Zhu, Q. Li, Lightweight hybrid materials and structures for energy absorption: A state-of-the-art review and outlook, Thin-Walled Struct. 172 (2022) 108760. https://doi.org/10.1016/j.tws.2021.108760

[2] J. Banhart, Metal Foams: Production and Stability, Adv. Eng. Mater. 8 (2006) 781-794. https://doi.org/10.1002/adem.200600071

[3] J. Banhart, Manufacture, characterisation and application of cellular metals and metal foams, Prog. Mater. Sci. 46 (2001) 559–632. https://doi.org/10.1016/S0079-6425(00)00002-5

[4] L. Peroni, M. Avalle, M. Peroni, The mechanical behaviour of aluminium foam structures in different loading conditions, Int. J. Impact Eng. 35 (2008) 644-658. https://doi.org/10.1016/j.ijimpeng.2007.02.007

[5] J. Baumeister, J. Banhart, M. Weber, Aluminium foams for transport industry, Mater. Des. 18 (1997) 217-220. https://doi.org/10.1016/S0261-3069(97)00050-2

[6] J. Kahani Khabushan, S. Bazzaz Bonabi, F. Moghaddasi Aghbagh, A. Kahani Khabushan, A study of fabricating and compressive properties of cellular Al–Si (355.0) foam using TiH2, Mater. Des. 55 (2014) 792–797. https://doi.org/10.1016/j.matdes.2013.10.022

[7] J. Banhart, Aluminium foams for lighter vehicles, Int. J. Veh. Des. 37 (2005) 114-125. https://doi.org/10.1504/IJVD.2005.006640

[8] A. Viscusi, M. Durante, A. Formisano, An Experimental–Numerical Analysis of Innovative Aluminum Foam-Based Sandwich Constructions Under Compression Loads, Lect. Notes Mech. Eng. (2022) 131-150. https://doi.org/10.1007/978-3-030-82627-7\_8

[9] A. Formisano, M. Durante, A. Viscusi, L. Carrino, Mechanical behavior and collapse mechanisms of innovative aluminum foam-based sandwich panels under three-point bending, Int. J. Adv. Manuf. Technol. 112 (2021) 1631-1639. https://doi.org/10.1007/S00170-020-06564-4/FIGURES/8

[10] C. Ensarioglu, A. Bakirci, H. Koluk, M. Cemal Cakir, C. Ensarioglu, A. Bakirci, M.C. Cakir, H. Koluk, Metal Foams and Their Applications in Aerospace Components, Sustainable Aviation. Springer, Cham. (2022) 27-63. https://doi.org/10.1007/978-3-030-91873-6\_2

[11] A.S. Perna, A. Viscusi, R.D. Gatta, A. Astarita, Cold spraying on polymer-based composites: Understanding the single-particle adhesion, Surf. Coatings Technol. 447 (2022) 128837. https://doi.org/10.1016/j.surfcoat.2022.128837

[12] A.S. Perna, A. Viscusi, R.D. Gatta, A. Astarita, Integrating 3D printing of polymer matrix composites and metal additive layer manufacturing: surface metallization of 3D printed composite panels through cold spray deposition of aluminium particles, Int. J. Mater. Form. 15 (2022). https://doi.org/10.1007/s12289-022-01665-9

[13] A. Viscusi, R. Della Gatta, F. Delloro, I. Papa, A.S. Perna, A. Astarita, A novel manufacturing route for integrated 3D-printed composites and cold-sprayed metallic layer, Mater. Manuf. Process. 37 (2022) 568-581. https://doi.org/10.1080/10426914.2021.1942908.

[14] P. Ammendola, R. Chirone, F. Raganati, Fluidization of binary mixtures of nanoparticles under the effect of acoustic fields, Adv. Powder Technol. 22 (2011) 174-183. https://doi.org/10.1016/J.APT.2010.10.002

[15] A. Viscusi, M. Bruno, L. Esposito, G. Testa, An experimental/numerical study of bonding mechanism in cold spray technology for metals, Int. J. Adv. Manuf. Technol. 110 (2020) 2787-2800. https://doi.org/10.1007/s00170-020-06060-9

[16] A.S. Perna, A. Viscusi, A. Astarita, L. Boccarusso, L. Carrino, M. Durante, R. Sansone,

Materials Research Proceedings 28 (2023) 49-56

https://doi.org/10.21741/9781644902479-6

Manufacturing of a Metal Matrix Composite Coating on a Polymer Matrix Composite Through Cold Gas Dynamic Spray Technique, J. Mater. Eng. Perform. (2019). https://doi.org/10.1007/s11665-019-03914-6

[17] A.S. Perna, A. Viscusi, A. Astarita, L. Boccarusso, L. Carrino, M. Durante, R. Sansone, Manufacturing of a Metal Matrix Composite Coating on a Polymer Matrix Composite Through Cold Gas Dynamic Spray Technique, J. Mater. Eng. Perform. 28 (2019) 3211-3219. https://doi.org/10.1007/s11665-019-03914-6