

A Device with a Controllable Internal Atmosphere, Independent from the Heating Furnace, for Sintering Metal Particles

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A device with a controllable atmosphere has been designed and fabricated to sinter metal particles in the manufacturing processes of metallic components (porous or non-porous parts). The device avoids the employment of an expensive controlled atmosphere furnace (a furnace in which it is possible to produce a vacuum or through which a constant flow of highly pure inert gas is maintained to protect the specimen). Metallic powders of pure metals or alloys with sintering temperatures (T_s) up to 1300°C and different forms and particle sizes can be used. The device consists of two chambers (an upper chamber for the atmosphere control and a lower chamber for the sintering processes) which are coupled by a sealed system composed of an O-ring and a clamp. The device is designed to be utilized with any conventional vertical electrical furnace (a furnace without the possibility of producing a vacuum inside or maintaining a constant flow of inert gas inside) and can be operated in three different ways: i) with a vacuum inside, ii) with a static atmosphere of inert gas inside, and iii) with a dynamic atmosphere of inert gas inside.

Keywords: *Controllable atmosphere device, Sintering process, Cellular metals.*

1. Introduction

The technology of powder metallurgy (PM) has been highlighted as a source in the development of new materials and light parts, providing a reduction of production costs, principally in the automobile industry. Currently, PM has proven to be competitive in casting, forging, and machine processes; particularly those employed in the manufacture of complex metallic parts, which can be made using the PM processes. During the production processes, a PM process is used to generate a green metallic pre-form and a sintering process is applied to form a stiff metal with high mechanical strength. The metallic particles utilized in the manufacture of metallic components may be of different shapes, e.g., one-dimensional (acicular and irregular bars), two-dimensional (dendrites and ships), and three-dimensional (spherical, round, irregular, angular, and porous particles)¹. In the sintering process, the green metallic pre-form is heated at temperature values between 70 and 90% of the melting temperature (T_m) of metal powder (sintering temperature, T_s) within a controlled atmosphere furnace (a furnace in which it is possible to produce a vacuum or through which a constant flow of highly pure inert gas is maintained) to avoid the fast oxidation of the compact. Generally, the PM has been used to manufacture steel tools, tungsten carbides, cermets, magnetic materials, metallic filters, porous metals,

biomaterials, automotive parts (around 70%), and several components utilized in industrial, aerospace, and nuclear applications, among others purposes¹. In the manufacture of new materials, the PM has been of great interest because i) it enables us to obtain components with dimensions close to the final dimensions of the part, ii) porous metals (cellular metals) may be manufactured with a controllable porosity, iii) the manufacturing of complex components that is not achievable using melting and casting methods is possible using PM processes, and iv) it allows for the production of parts with specific mechanical properties.

In the last two decades, a significant number of information concerning metal components manufactured by PM have been published, mainly about large components made with high-purity metals or composites of different materials, e.g., Pt-Rh², Al-Al₂O₃³, Mg-Al-Zn⁴ and Co-Cr-Mo⁵ among many others. Additionally, porous metals have been produced with different metal particles such as metal and alloy powders using PM processes, e.g. Fe, Cr and, Al foams⁶, stainless steel 310SC, Fe, Fe-Cr, Fe-Cr-Ni, Fe-Cr-Zn, Fe-Cr-Al, Fe-Cr-Mg, Fe-Cr-Pb, Fe-Al, Fe-Zn and Fe-Ni foams⁷, Al foams⁸, Al-Cu and Al-Mg-Si-Cu-Mn foams⁹, Ni-Ti-Nb foams^{10,11}, Ti and its alloys foams¹², Al-Si foams¹³, stainless steel 316L foams¹⁴, Al, Cu-Au, Fe, Mg, Ni, Pt, Ag, Cu-Sn, Cu-Zn, Cu-Sn-Zn and Ni-Al foams¹⁵, stainless steel JIS G4303, SUS 310S foams and steel tools JIS G4404, SKD

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61 foams¹⁶, Cu–Al–Mn foams¹⁷, Cu foams¹⁸ and Al foams¹⁹ as well as others. In all cases, the green pre-forms were sintered.

Porous metals have also been manufactured using a controlled atmosphere furnace with a vacuum inside or with a constant flow of inert gas inside, e. g. Fe containing Cr and Al foams⁶, stainless steel 310SC, Fe, Fe–Cr, Fe–Cr–Ni, Fe–Cr–Zn, Fe–Cr–Al, Fe–Cr–Mg, Fe–Cr–Pb, Fe–Al, Fe–Zn and Fe–Ni foams⁷, Ni–Ti–Nb foams¹¹, Mo, Mo alloys, W and W alloys²⁰, Mg foams²¹, Ti and Mg foams²², Al foams²³ and Ti–Al–V foams²⁴, etc. On the other hand, to obtain open-pore porous metals, techniques such as powder metallurgy are recommended. These materials can be used in the manufacture of filters, heat exchangers, and catalytic converters²⁵. Further, the traditional sintering–dissolution method has been modified for Li et al.²⁶ to produce open-cell copper foams with completely interconnected sphere pores. The modified method uses a preformed calcium chloride template skeleton as a space holder. Compared with the traditional method of foaming process, the open-cell copper foam obtained by this method has fewer impurities and better surface morphology. The employment of controlled atmosphere furnaces implies obtaining components at higher costs since a controlled atmosphere furnace is more expensive than a conventional electric furnace, however, it can't control its internal atmosphere through a constant flow of an inert gas or vacuum. In this study, a new device with a controllable atmosphere of inert gas is developed which is used in the sintering step of components manufactured with compacted metal powders (non-porous or with variable porosity). The device is placed into a conventional vertical electric furnace and operated with a vacuum inside. The device may be employed for the sintering of porous or non-porous green metallic pre-forms produced with metal particles

whose T_s is less or equal to 1300 °C. The device allows for the control of its atmosphere independent from the heating furnace, avoiding the acquisition of an expensive controlled atmosphere furnace and reducing the production costs of metal components. This allows students, teachers, and researchers in the laboratory to have an easily acquired tool at a low cost to study the sintering processes. Furthermore, the device has some advantages compared to a controlled atmosphere furnace such as it is lightweight, easy to carry and move, it is affordable, it has lower inert gas consumption, lower energy consumption, reduced operation time, and the possible use of any conventional electrical furnace opened in its upper side.

2. Description of the New Controlled Atmosphere Device

The device mainly consists of two chambers, an upper chamber for the atmosphere control and a lower sintering chamber. The specimen is placed within the crucible and the crucible is placed within the sintering chamber. Both chambers are connected by an O-ring, a centering ring, and a clamp. The sintering chamber is placed into a conventional vertical electrical furnace and the upper chamber along with the sealed system is outside of the furnace. To guarantee an oxygen-free environment in the device, the air is evacuated from the upper chamber using a vacuum pump and replaced by inert gas (mainly Ar and He, or any other inert gas if it is necessary). The device is used to sinter porous or non-porous components made of metal particles (pure metals and metal alloys with low sintering temperatures).

Figure 1 shows a schematic diagram of the upper chamber for the atmosphere control (1). The upper side of

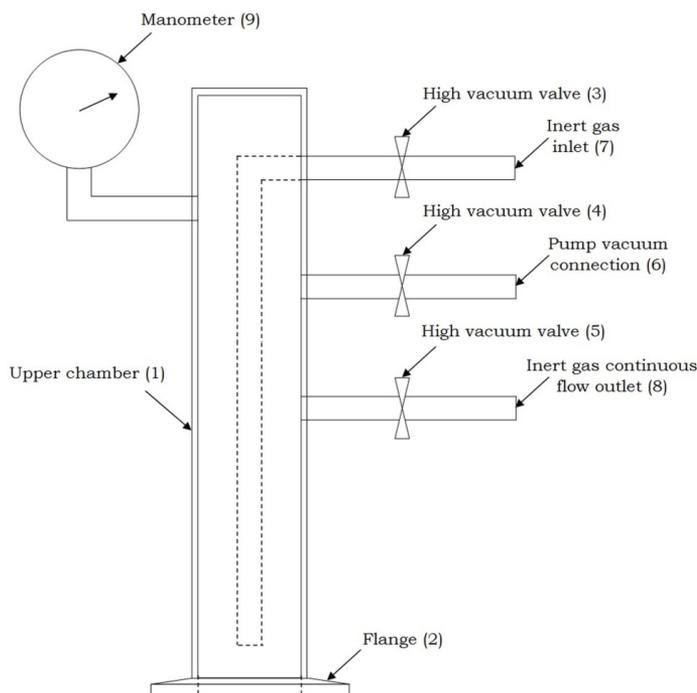


Figure 1. Diagram of the upper chamber for the atmosphere control.

the chamber is closed, and its lower side is open and has a flange (2). There are three high vacuum valves (3, 4, and 5), a pump vacuum connection (6), an inert gas inlet (7), an inert gas continuous flow outlet (8), and a manometer (9) for positive and negative pressure control in the chamber for atmosphere control. Figure 2 shows a schematic diagram of the lower sintering chamber (10). The lower side of the chamber is closed, and its upper side is open and has a flange (11). The crucible (12) containing the specimen (13) is placed within the sintering chamber.

Figure 3 shows the schematic diagram of the sealed system, which consists of a centering ring (14), an O-ring (15), and a metal clamp (16).

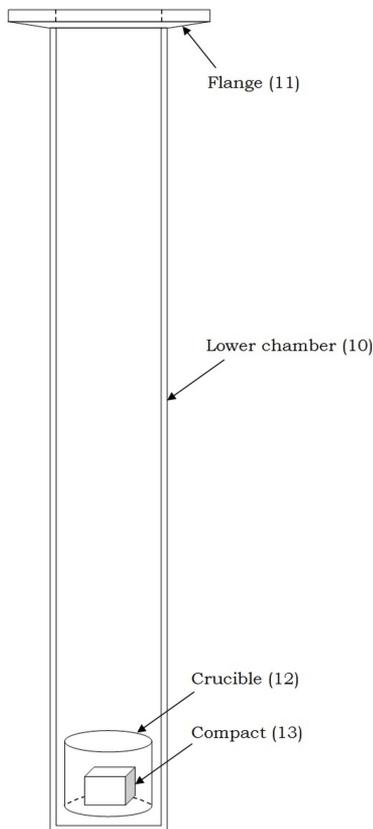


Figure 2. Diagram of the lower chamber for the sintering processes.

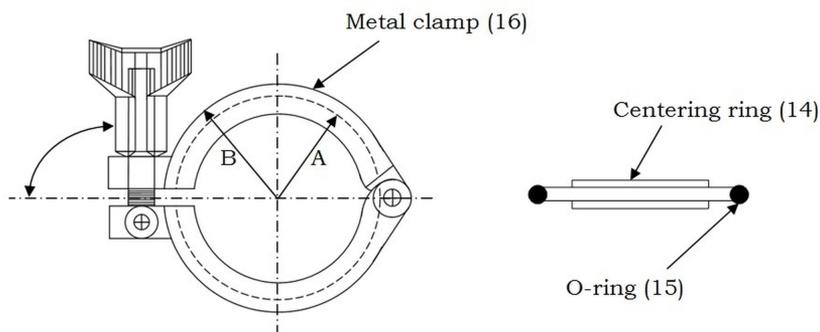


Figure 3. Diagram of the sealed system.

2.1. Materials used in the fabrication of the new device

Figure 4a shows the device built in our laboratory and Figure 4b shows the configuration of the device/furnace. This is used by placing the device into the conventional vertical electrical furnace; this was the method employed in this study.

The material used in the manufacturing of the chamber for atmosphere control shown in Figure 1 was borosilicate. The length, internal diameter, and wall thickness of the chamber were 200 mm, 29 mm, and 1.5 mm, respectively. The internal diameter and wall thickness of the manometer connection, vacuum pump connection, inert gas inlet, and inert gas continuous flow outlet were 6 mm and 1.5 mm, respectively. The distance between the vacuum pump connection, inert gas inlet, and inert gas continuous flow outlet was 50 mm. The manometer shown in Figure 1 is for general use Metron trademark, model 63100 with a carbon steel box, and internal connections of bronze and brass, the measuring range is from -76 cm of Hg to 1 kg/cm² with a dual scale from 30 inches of Hg to 14 lb/plg² and an external thread connection of 6.35 mm NPT. The diameter and thickness of the flange were 50 mm and 4 mm, respectively.

The sintering chamber shown in Figure 2 was made of quartz. The length, internal diameter, and wall thickness of the chamber were 350 mm, 29 mm, and 1.5 mm, respectively. The diameter and thickness of the flange were 50 mm and 4 mm, respectively. The diameter and height of the crucible were 20 mm. The crucible may be made of quartz, alumina, or other refractory materials. The sealed system shown in Figure 3 consists of an aluminum centered ring, an O-ring Parker trademark reference 2-222 with W=3.53 mm, ID=37.69 mm, and ED=44.75 mm, and a metal clamp with an internal diameter A=44.4 mm and a medium diameter B=53.6 mm. The inert gas used can be argon or helium.

3. New Device Validation and Experimental Techniques

To validate the device proposed, Al foams were made and sintered employing argon as an inert gas. The Al foams were manufactured using the sintering dissolution process (SDP). The metal powders used in the current study were Al powders with 98.0% purity and a particle size of ~74 μm .

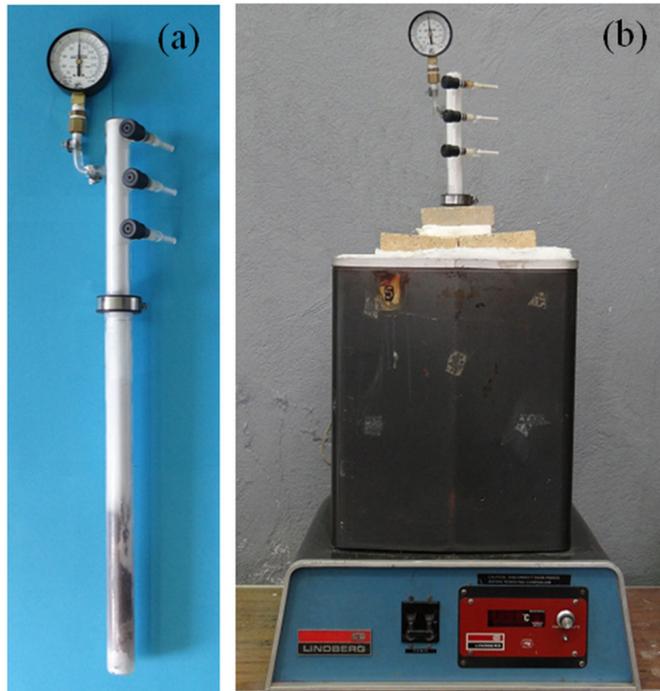


Figure 4. a) Image of the new device with controllable atmosphere of inert gas fabricated in our laboratory, and b) Device/furnace configuration used for the sintering processes in the current study.

Spherical carbamide (CON_2H_4) particles with particle sizes in the range of 1–2 mm in diameter were used as space holder particles (SHP). The form and size of carbamide particles were characterized using a Sony 3D digital camera. To determine the metallic particles' form and the quality of the metallurgical bond among the metallic powders, the Al powders, and sintered metal powders were characterized by scanning electron microscopy SEM (JEOL JSM 6610 HLV), operated at 20 keV. The mechanical properties of the specimens were measured through the compression tests, employing an MTS–810 materials testing machine with a crosshead speed of 0.1 mm/min. For each specimen, the weights of the resultant metal foams after the sintering processes were measured using an Ohaus balance with a maximum capacity of 310 g and readability of 0.001 g. Using these data, the relative density of foams was determined through $\rho_f = \rho_f / \rho_{\text{Al}}$ equation²⁶ where ρ_f is the foam density and ρ_{Al} is the Al density. The ρ_f was determined through the sample weight and its dimensions, using the conventional equation $\rho = m/V$ (m =mass, V =volume).

3.1. New device operation employing a vacuum inside

The green pre-form is placed inside the crucible and then the crucible containing the green pre-form is placed into the sintering chamber. The chamber for the atmosphere control and the sintering chamber are coupled using the sealed system. Finally, the sintering chamber is placed into a conventional vertical electrical furnace. The chamber for the atmosphere control with the sealed system should be outside the electrical furnace. When the device is operated with a vacuum inside the operation route is as follows:

1. The vacuum pump is connected to the device in (6).
2. The inert gas cylinder is connected to the device in (7).
3. The outlet (8) is connected to the fume hood.
4. The vacuum pump and the fume hood are turned on.
5. The air is removed with the vacuum pump by opening the valve (4). The valves (3) and (5) should be closed. The negative pressure (-25 inch of Hg) is registered by the manometer (9).
6. The valve (4) is closed, and the valve (3) is opened so that the device is filled with inert gas until atmospheric pressure is reached. The valves (4) and (5) should be closed. Zero pressure is registered by the manometer (9).
7. The valve (3) is closed.
8. Steps 5 to 7 are repeated three to five times to guarantee an atmosphere with the lowest possible concentration of oxygen.
9. The valve (4) is opened to produce a vacuum in the device. The valves (3) and (5) should be closed. The negative pressure (-25 inches of Hg) is registered by the manometer (9).
10. With the device now in a vacuum, the sintering process of green pre-form (13) is made by heating the furnace from 25 °C to T_s of metal particles at a heating rate of 10 °C/min.
11. The furnace temperature is maintained at T_s during the required time to produce a stiff structure with high strength.
12. The furnace temperature is decreased from T_s to 25 °C at a cooling rate of 10 °C/min.

13. The valve (3) is opened to fill the device with inert gas until atmospheric pressure is reached. Finally, the valve (3) is closed.
14. The device is removed from the furnace, and it is opened to obtain a sintered component.

3.2. New device operation employing static atmosphere of inert gas inside

The sintering process of green compact (13) may also be performed with the device operated with a static atmosphere of inert gas inside (e.g., Ar or He, or any other inert gas if it is necessary). In this case, after performing steps 1 to 8, the valve (4) is closed, and valve (3) is opened so that the device is filled with inert gas until atmospheric pressure is reached. After having filled the device, valve (3) is closed and valves (4) and (5) should be closed. Finally, with a static atmosphere of inert gas inside the device, steps 10 to 14 are performed. If during the sintering process, the internal pressure of the device exceeds 5.0 psi, valve (5) should be opened to allow the depressurization of the chamber to diminish the internal pressure at 0.0 psi. Afterwards, valve (5) should be closed.

3.3. New device operation employing dynamic atmosphere of inert gas inside

The sintering process of green compact (13) may be performed with a continuous flow of inert gas into the device, also (dynamic atmosphere). In this case, after performing steps 1 to 8, valves (3) and (5) are opened to let the continuous flow of inert gas into the device, and finally, steps 10 to 12 and 14 are performed.

4. Results and Discussion

Figure 5a shows the Al powders with a 98.0% purity and $\sim 74 \mu\text{m}$ particle size, and Figure 5b shows the carbamide with a particle size in the range of 1–2 mm.

To determine the optimum sintering temperature T_s for the Al particles, specimens with 1.6 mm in diameter and 1.3 mm in length, containing 100%vol. of Al powders compacted at 400 MPa, were sintered for 90 minutes with a vacuum in the device. For each experiment temperature increments of 30°C were employed, starting from 530°C until Al particle union was reached. Following this procedure,

Aluminum powder T_s results were 620°C . Figure 6a–d show the SEM images of the Al specimen's thermal treatment at temperatures of 530°C (Figure 6a), 560°C (Figure 6b), 590°C (Figure 6c), and 620°C (Figure 6d). At lower temperatures, poor particle bonding is observed (530 and 560°C), whilst at 590 and 620°C , good particle bonding is observed. In consequence, the selected temperature for all subsequent sintering treatments of Al foams was 620°C because at this temperature a higher sintering was reached.

After establishing the T_s for Al powders, foams series containing from 20 to 100%vol. of metallic powders (with increases of 5%vol.) were manufactured employing the method known as the sintering dissolution process (SDP); carbamide (CON_2H_4) was used as space holder particles (SHP). The mixing of the powders was performed in an agate mortar for 15 minutes. To ensure good adhesion of Al powders, 1.0%vol. of ethanol was sprayed on the carbamide particles before the mixing stage to obtain a sticky surface. Following this, the mixture was put into a steel mold and uniaxially pressed to produce cylindrical compacts with ~ 16 mm diameter and ~ 13 mm length. The pressure used for the compaction stage of the Al and $\text{CH}_4\text{N}_2\text{O}$ mixture was 400 MPa for all samples.

The carbamide fraction of the compact formed at this stage was dissolved by double immersion in a water bath at 25°C for two hours. In all cases, the dissolution stage of the process was carried out before the sintering stage. The sintering processes of the green Al-foams were carried out in two steps: a) The samples were put into the device with a vacuum inside. The temperature was increased from 25 to 250°C at a heating rate of $10^\circ\text{C}/\text{min}$; the samples were held at 250°C for 60 min to eliminate the remaining carbamide in the green sample. In the second step the temperature was increased from 250 to 620°C at a heating rate of $10^\circ\text{C}/\text{min}$ and the samples were held at this temperature for 120 min to form a metallurgical bond between the Al powder particles.

Figure 7 shows the Al foams series after the sintering process, containing from 20 to 100%vol. of Al powders and formed using spherical carbamide particles in the range of 1–2 mm. Macroscopically, the samples presented a regular pore distribution, which results in an Al foam with a regular framework, as it is required for reliable measurements of mechanical properties in this kind of material. In the image,

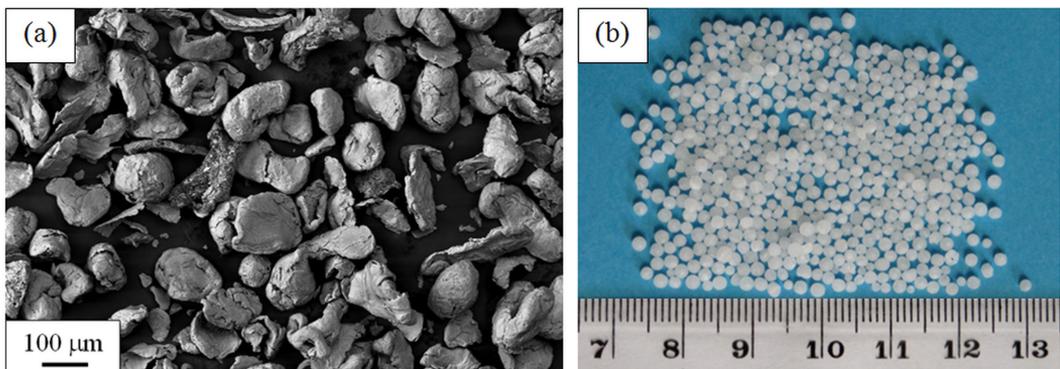


Figure 5. a) SEM secondary electron images of Al powders with a size of $\sim 74 \mu\text{m}$, and b) optical image of spherical carbamide granulates with a size range from 1 to 2 mm.

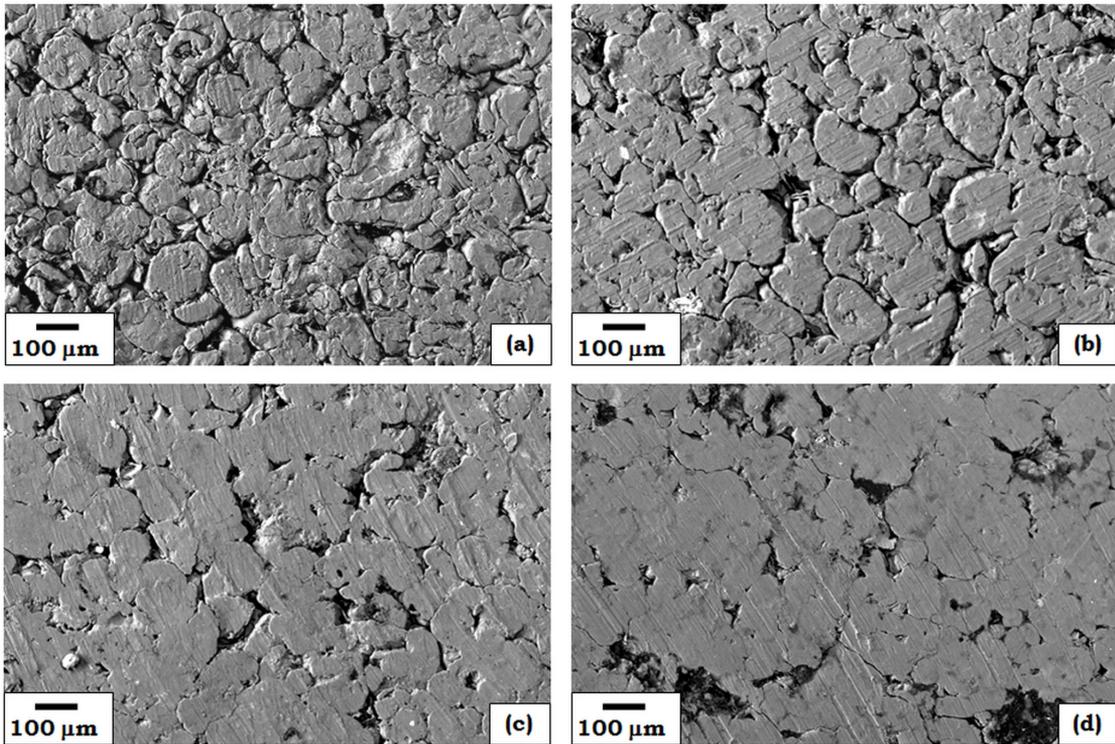


Figure 6. SEM secondary electron images showing the superficial bonding of compacted aluminum particles sintered at temperatures of: a) 530 °C, b) 560 °C, c) 590 °C and d) 620 °C.

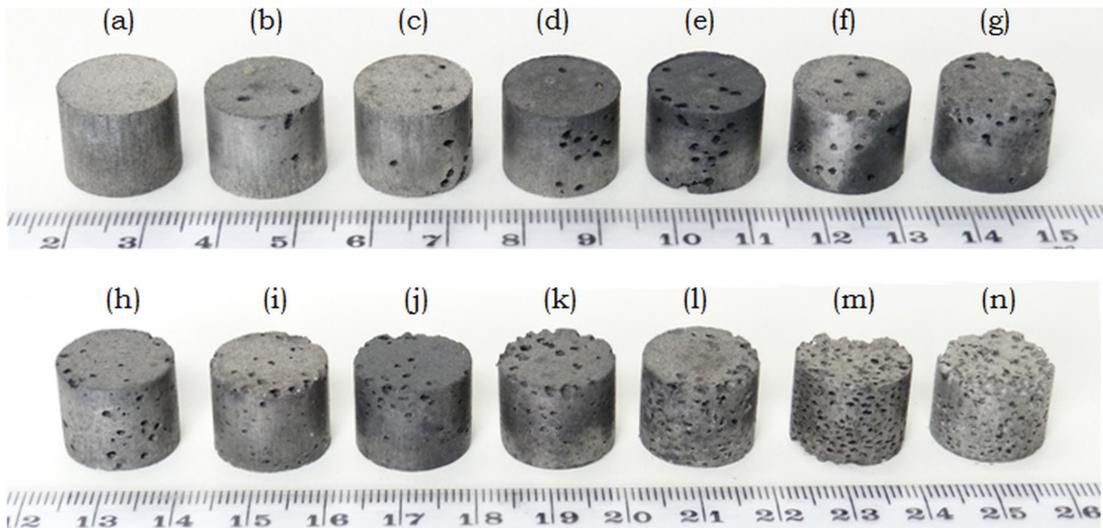


Figure 7. Image of an aluminum foams series after the sintering process, manufactured by the SDP and employing in the sintering step the controllable atmosphere device here proposed: a) 100, b) 80, c) 75, d) 70, e) 65, f) 60, g) 55, h) 50, i) 45, j) 40, k) 35, l) 30, m) 25 and n) 20%vol. of Al powders.

an increment in the number of pores is observed as the Al powder content decreases. The effects of the relative density of the foams on the mechanical properties of the processed foams were assessed through compression tests. Figure 8 shows

the plot of maximum strength σ_{\max} , vs relative density ρ_r , for the foams containing from 20 to 100%vol. of Al powders.

The specimen with 100%vol. of Al powders showed a maximum deformation strength value of $\sigma_{\max} = 47.77$ MPa.

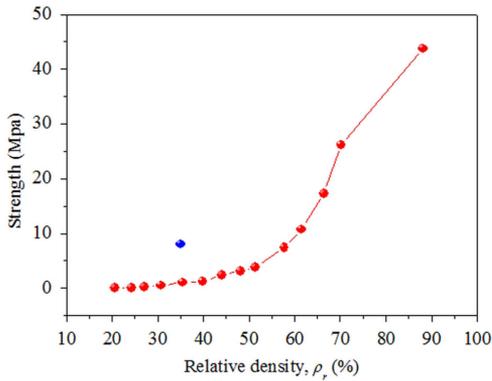


Figure 8. Strength vs relative density for the foam's series containing from 20 to 100%vol. of Al powders.

This value is close to the reported 55 MPa value for the pure Al obtained by the casting process²⁷. The σ_{\max} value obtained from the 100%vol. of Al powders compacted sample indicates a good metallurgical bond among particles caused by the uniaxial compression step and sintering at the correct T_s , previously determined. In this plot, the strength increases as the Al content increases. The value of $\sigma_{\max}=0.04$ MPa for the specimen with 20%vol. of Al powders increases to $\sigma_{\max}=47.77$ MPa for the specimen with 100%vol. of Al powders (when the Al carbamide content diminishes from 80 to 0.0%vol.). Similarly, to previously seen trends in other foams²⁸ this increment in σ is attributed to the diminishing of foam porosity, which implies a higher material amount and consequently a higher area supporting the load. Note that the σ_{\max} value for the sample without space holder particles (100%vol. of Al powders) also supports this trend, since the value of 47.77 MPa is lower than the expected σ_{\max} value of 55 MPa for the pure Al obtained by the casting process. This is due to a significant amount of porosity caused by the inclusions of void spaces among the metal particles (approximately 13%), which implies a lower area supporting the load, compared to the pure Al obtained by the casting process, and consequently the reduction in σ_{\max} value for the 100%vol. of metal powders sample is observed. The foams containing 35 and 40%vol. of Al powders presented σ_{\max} values of 0.47 y 0.85 MPa, respectively (in this case for a 37.5%vol. of Al powders content, a $\sigma_{\max} \sim 0.66$ average value, is obtained). The $\sigma_{\max} \sim 0.66$ value results to be lower than the one reported by Hassani et al.²⁹. They reported, for foams containing 45%wt. of $\text{CH}_4\text{N}_2\text{O}$ (62.60%vol. of $\text{CH}_4\text{N}_2\text{O}$ or what is the same 37.40%vol. of Al powders) compacted to 330 MPa and sintered at 640 °C, an ~ 8 MPa value for σ_{\max} (see the blue point in Figure 8). Although, the employment of multi-size SHP increases the stiffness and the strength of foams as it was previously mentioned by Li et al.³⁰. The higher value in σ_{\max} obtained by Hassani et al.²⁹ is caused mainly by three actions: i) the use of small amounts of additives such as Mg and Sn, which promote the liquid phase formation during the sintering stage. However, in the current study, these kinds of additives were not used; ii) the employment of lower particle size of Al powders, between 2 and 45 μm , which creates a higher superficial area and consequently a

higher contact among particles producing a higher sintered material amount. However, in the current study a particles size of ~ 75 μm was used; and iii) The use of $T_s=640$ °C, which produces a higher sintering of the metal powders. However, in the current study a $T_s=620$ °C was employed. Those three actions substantially enhance the foam strength. On the other hand, Amirjan and Bozorg³¹. reported relative density values of 60% for samples with 100% aluminum powders sintered in an argon atmosphere. However, in the current study, a relative density of about 90% was obtained, which may be due to the compaction pressure (400 MPa).

Finally, porous components with controllable porosity or non-porous components, made with pure metal powders or alloy powders with low sintering temperatures ($T_s \leq 1300$ °C) and different sizes and form particles, can be fabricated employing the method proposed here. A good bonding among the metal particles and good mechanical properties with oxide-free samples can be obtained using the device with atmosphere controlled. The results confirm that the device, here proposed, is an excellent tool to be used for teaching and researching of the sintering processes in the laboratory field.

5. Advantages of the Controlled Atmosphere Device

The advantages of the device compared to an atmosphere-controlled furnace are as follows: a) ease of operating, b) it is lightweight and therefore easy to transport, c) it is relative cheap to manufacture, d) it has lower inert gas consumption because the sintering chamber can be built according to the size of the green part to be sintered (the dimensions of the sintering chamber depend on the size of the green part to be sintered), e) there is lower energy consumption when the green parts are small because the furnace can be built according to the size of the sintering chamber. Additionally, there is a lower operating time when the sintering chamber is small, f) the flange connection and sealing system allows the use of the device in a vacuum or at atmospheric pressure or pressure higher than atmospheric pressure; the sealing system allows a 100% hermetic seal, avoiding the air inlet or the inert gas outlet when the device is operated in vacuum or higher than the atmospheric pressure, g) the device can be used for the sintering of components made with metallic powders in a wide range of pure metal or alloys with low fusion temperatures, h) the device can be used in any conventional electric furnace, i) and in the industrial sector, the device can be re-designed according to the green component dimensions or the dimensions of electrical furnace.

6. Conclusions

In conclusion, a new device with a controllable atmosphere of inert gas (which can be operated with a vacuum inside or with a static atmosphere of inert gas, or with a dynamic atmosphere of inert gas inside) consisting of a chamber for the atmosphere control coupled to a sintering chamber through a sealed system, was fabricated. The device may be very useful in the sintering of components manufactured with compacted metallic particles and can be employed as a teaching tool in the laboratory by teachers, students, and

researchers of chemical, metallurgical, and materials science, among other areas. Porous components with controllable porosity or non-porous components made with pure metal powders or alloy powders with low sintering temperatures ($T_s \leq 1300$ °C) and different sizes and form particles, can be fabricated. On the other hand, operating the new device with a vacuum inside, Al foams containing from 20 to 100%vol. of metallic powders were successfully produced by powder metallurgy using the method known as the SDP and utilizing spherical carbamide as space holder particles. Using the new device, a good bonding among the metal particles and oxide-free samples was obtained as it is observed in the SEM images. This indicates that the new device's internal atmosphere was successfully controlled. Furthermore, good mechanical properties were observed in the strength values obtained through compression tests, indicating that the T_s was properly selected. These results confirm that the new device proposed here is an excellent tool to be used in teaching and researching the sintering processes in the laboratory. However, redesigning the device dimensions will allow its use in the industrial sector. Finally, the porous metals obtained in this current study could be used in any of the following applications: filters, heat exchangers, and catalytic converters, as well as others.

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