STEEL AND GRAY IRON FOAM BY POWDER METALLURGICAL SYNTHESIS

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STEEL AND GRAY IRON FOAM BY POWDER METALLURGICAL SYNTHESIS

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Resumen
Se elaboraron mezclas con polvos comerciales de hierro, grafito y carbonato de magnesio (MgCO 3) las cuales se compactaron a presiones de 900 y 1000 MPa. Las mezclas compactadas se espumaron en estado semisólido a 1300°C en un horno con atmósfera inerte durante 17 minutos, obteniéndose espumas con densidades equivalentes al 50-60% de la densidad del acero. Debido a que las muestras presentaban un alto contenido de cementita se llevo a cabo un tratamiento de decarburización a 1000°C. El comportamiento a compresión de las espumas presento una meseta de deformación característica de esta clase de materiales la cual les confirió una alta capacidad de absorción de energía

Palabras clave: Espumas metálicas, Espumas de acero e hierro gris, Metalurgia de polvos

Abstract
Foam synthesis, from several blends of pure iron, commercial graphite and gray iron powders plus the foaming agent (MgCO₃), were carried out. These powders, in determined proportions and commercial graphite with up to 2.5Wt%, were blended in a small tumbler-type ball mill for 1½ hours. Then, they were compacted by means of a 20 mm diameter die, to obtain 30 mm. to 35 mm. height samples having an average density near to 85% the iron's density. It was done by applying pressures between 900 MPa and 1000 MPa. These compacts were sintered at 1300°C during 17 minutes, under a nitrogen atmosphere. The furnace was previously preheated. The foam density was about 50% to 60% steel's. Because of sintered samples contained too much cementite, a heat decarburizing treatment was carried out, after lathe machining samples surface. Samples were protected against excessive surface oxidations by using small pieces of coke.

Keywords: Metal foams, Steel and Gray Iron foams, Powder metallurgy

1. INTRODUCTION
Metallic foams are a new kind of material. Their structure is modified to obtain a closed porosity morphology they offer a lot of functional and structural applications. The steel foams have their main applications as structural elements, light constructions, core of beams and sandwich panels where, light weight and high specific rigidity, are required [1]. Moreover, their capacity to absorb energy makes them ideal for applications when impact absorption is needed.
Steel foams can reach, at low cost, very high strength as compared with other materials [1]. This is why, a raw material apparently useless as well as economic like grey iron turnings, could be used to produce ferrous foam by the powder metallurgical process. Several mixtures of iron (Fe), graphite (C) and foaming agent (MgCO₃) as well as mixtures with a part of iron substituted by gray iron debris have been evaluated to produce steel foams.

2. EXPERIMENTAL TECHNIQUE
Iron (Fe) and graphite (C) commercial powders were used, as well as, Magnesium carbonate (MgCO₃), and calcium carbonate (CaCO₃) as foaming agents. Also gray iron powder obtained from scrap milling was used. The mixtures weight percentage composition was defined according to common ones reported in the literature [1,3,5]. Then, metallurgical powder process steps were accomplished: mixing, compacting and foaming at temperatures up to material's melting point.

The Percentage of magnesium carbonate was increased from 0.2 to 1%, with the aim to generate a greater amount of pores, and reduces the foam material density. The more blowing agent added
the more empty spaces expected. And the higher graphite content the lesser foaming temperature.

Table 1 exhibits the powder mixtures used in this work.

Table 1. Sample composition used, in this work, to obtain steel foams

<table>
<thead>
<tr>
<th>Sample</th>
<th>% wt Fe</th>
<th>% wt C</th>
<th>% wt MgCO₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>M2</td>
<td>97.25</td>
<td>2.5</td>
<td>0.25</td>
</tr>
<tr>
<td>M3</td>
<td>97.00</td>
<td>2.5</td>
<td>0.5</td>
</tr>
<tr>
<td>M4</td>
<td>96.75</td>
<td>2.5</td>
<td>0.75</td>
</tr>
<tr>
<td>M5</td>
<td>96.25</td>
<td>2.75</td>
<td>1</td>
</tr>
<tr>
<td>M6</td>
<td>97.05</td>
<td>2.75</td>
<td>0.2</td>
</tr>
<tr>
<td>M7</td>
<td>96.8</td>
<td>3</td>
<td>0.2</td>
</tr>
<tr>
<td>M8</td>
<td>96.8</td>
<td>3</td>
<td>0.2</td>
</tr>
<tr>
<td>M9</td>
<td>43.6% Fe + 56.15% HG*</td>
<td>0</td>
<td>0.25</td>
</tr>
</tbody>
</table>

*gray iron powder

2.1 Mixing
Mixing was done in the device shown in figure 1. The mixing time was 90 minutes at 58 RPM. The powders and balls volume, occupied up to 42% that of container. The tumbler speed was 58 rpm when the so named, "balls cascading fall", was taken place, a necessary condition for mixture homogenization.

Figure 1. Small Ball Mill used to even out the powder mixtures.

2.2 Compaction
A pressure of 900 MPa was used to compact the samples M2 to M6 and 1000 MPa with samples M7 to M9. Such pressures were established experimentally by compacting the samples until a free residual porosity material was obtained.

2.3 Foaming temperature
The compact material was placed inside an electric furnace at 1250°C under the presence of an inert atmosphere of nitrogen during approx. 17 minutes, when the melting temperature was reached. Then the samples were taken out of the furnace. This temperature depends on sample compositions.

2.4 Decarbonizing treatment
The high graphite content affects the material's ductility, because of the formation of cementite. For this reason, a decarbonizing heat treatment was required, foams were heated up to 1000 ºC and maintenance time of 2 to 6 hours (see table 2). The samples were partially protected against oxidation by immersing them into a container full of small coke fragments. It ensures that oxygen decarbonizes the sample, without producing an excessive amount of rust. Some samples were machined prior the heat treatment, and others after it, this procedure conducted, in the case of the former one to attain a better volume decarbonization.

Table 2. Heat treatment times at 1000°C.

<table>
<thead>
<tr>
<th>Sample</th>
<th>M2**</th>
<th>M3**</th>
<th>M4*</th>
<th>M6**</th>
<th>M7**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time(h)</td>
<td>2</td>
<td>4</td>
<td>2</td>
<td>6</td>
<td>6</td>
</tr>
</tbody>
</table>

*Heat treatment before lathe work
**Heat treatment after lathe work.

3. RESULTS AND DISCUSSION

3.1 Mixtures based on gray iron.
This alternative was initially thought in order to reduce the process’s temperature. Gray iron presents higher amounts of carbon, approximately 3.2%, which should lower the foam's melting temperature.

It was hoped that, when gray iron was heated above the eutectic point (1154°C), a higher concentration of liquid phase, at areas in which pores could be nucleated, was formed. Pores nucleate at sites where liquid phase occurs and the generated gas diffuses through the partially molten material [2]. Figure 3 shows the results obtained from iron plus iron gray based mixtures.

The top sample had nucleated the pores but they did not grow enough, possibly because lack of time and temperature. The bottom ones presented big pores possibly attributed to the absence of graphite in the mixture, in accordance to the literature, amounts of carbon smaller than 0.77 % produces a material with a single centre pore [3].
Steel and gray iron foam by powder metallurgical synthesis

3.2 Foams base on iron/graphite/MgCO$_3$ mixtures.

According to figure 4 the material grows irregularly shaped, primarily in the opposite orientation to the direction of compacting as according to literature [4].

3.3 Samples characterization

3.3.1 Density measurements

The implemented procedure reached the following densities (see Table 4):

<table>
<thead>
<tr>
<th>Sample</th>
<th>M2</th>
<th>M4</th>
<th>M6</th>
<th>M7</th>
<th>M9*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm$^3$)</td>
<td>4.71</td>
<td>4.35</td>
<td>4.86</td>
<td>4.86</td>
<td>4.57</td>
</tr>
</tbody>
</table>

*The density was found through Archimedes method as the sample was after foam.

The density measurements were made after samples were machined, the values were 55-60% that of steel density (7.86 g/cm$^3$).

3.3.2 Result Analysis: The blowing agent effect

It is expected that adding blowing agent over the conventional percentage of 0.25% MgCO$_3$ will increase porosity [3,5]. However, higher values showed a hollow structure in place of a porous material. This behavior is observed in figure 6 shown samples M3 and M5.

These samples show the formation of pores extremely large and heterogeneous, which is undesirable because it generates unsatisfactory mechanical properties. For these reason, it was decided to use quantities of MgCO$_3$, close to conventional (0.25%). The porosity obtained from this composition can be seen in the sample M2, figure 7.
M2 sample shows a uniform pores and a diminishing size tendency, nevertheless, the sample still presents a high percentage of opened and interconnected pores. From this fact, the amount of foaming was reduced to 0.2% in samples M6 and M7. The results were satisfactory as asserted by the plateau presented in the compression test (see figure 7).

3.3.3 Analysis and results of carbon content.
Initially, it was used 2.5% carbon, as reported literature [1,3,5]. There were subsequently experiments with equal percentages and higher than this value. This variation in the percentage of carbon is an important contribution from this work to the development of foam steel.

Foam's temperature were, on M2, M3 and M4 at 1330°C, M5 and M6 at 1300°C, for M7 and M8 at 1280°C.

3.3.4 Result Analysis: heat treatment effect
The heat treatment or annealing decarburization was done with the machined foams and non machined ones. Machining discovers pores and allows oxygen penetration throughout the foam volume to produce a generalized decarburization.

3.3.5 Metallographic Analysis
The foam microstructure obtained before making the decarburizing heat treatment is shown in figure 8. A thick cementite network (white) around perlite grains (dark) can be seen.

After decarburizing treatment is made, a microstructure with a significant carbide (cementite) reduction appears. That cementite has had a transformation phase to form ferrite and perlite. This can be observed in figure 9.
It can be seen that in M2 surface, the cementite network has disappeared completely, forming ferrite lamellas (white) with perlite grains (black). The ferrite phase was verified through microhardness testing. While in M4 surface there is still a cementite network even though small. This is because the M2 sample was machined before annealing, exposing the pores and allowing oxygen to penetrate and achieve better decarburization.

The microphotography of M2 and M4 samples taken at the inside, reveal the presence of cementite network, although the M2 sample, has a size reduction because, as mentioned above, this was machined before annealing.

Figure 10. M6 sample. Surface (upper) and intern (bottom) region microphotos.

Figure 10 shows the M6 sample's microphotography taken at its surface and internal regions. This sample was heat-treated for 6 hours. The carbon content was initially about 2.7%.

It was seen in M6 surface, a total decarburization, disappearing cementite network and forming a matrix of ferrite lamellas (white) with some grains of perlite (black). On the inside of M6, there is a network of very thin cementite, located in a small region of sample centre.

3.3.6 Result Analysis: compression test

The objective of this test is to establish the effort-deformation curve on compression and to determine the most relevant properties that foam steels have like energy absorption, the fluency point, stiffness and deformity of densification.

According to the curve of figure 11, foam deforms elastically at first and then plastically until crushed, with the exception of the sample M4 which failed prematurely and was so fragile. A cementite network thicker than in other foams appears to be the reason for this behavior. This sample also showed an interesting behavior, it was the only one that presented a well defined plateau or area of constant stress. Possibly, this is due to the amount of built in foaming agent (0.75% MgCO3), which create greater pores concentration.

M6 and M7 foams deform at a low stress level in contrast to the M2 foam that has a higher resistance and higher values of stress. This behavior can be attributed to the decarburizing time. Therefore, It can be expected that M6 and M7 samples were more ductile than M2 sample.

The behavior of all samples with the exception of M4 foam can be divided into 3 zones, namely 1.Elastic, 2.Slow increasing stress as the material deforms plastically and 3. swift increasing stress and material densification. M6 and M7 foams were made with mixtures of carbon 2.75% and 3%C but the response to compression is characteristic of steel. This is due to the decarburizing heat treatment.

Figure 11. Compression curves for M2, M4, M6 and M7 foams

4. CONCLUSIONS

Through the process of powder metallurgy, steel foams were obtained. This steel foams had densities between 55-60% of steel density, and low
meltng points, even below conventional points. Moreover, the foams have a response to compression test similar to those reported by international literature.

With graphite additions, it was achieved a significant reduction of foaming temperature. Higher additions of foaming agent (MgCO3) did not yield to the best results. So, the best alternative is to have amounts already used conventionally, like (0.25%).

With heat treatment, it was obtained a reduced network of cementite. The longer the decarburizing time is, the thinner the cementite network. As a result material's ductility is improved.

Gray iron turnings are a virtually useless raw material.

5. ACKNOWLEDGMENTS
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6. REFERENCES