Study of lead foams under microgravity

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This article describes an experiment to investigate the influences of physical properties like drainage and surface tension during the production of a metallic foam according to a powder metallurgical method. Foams were produced under microgravity conditions, so that gravity driven drainage was not present during the process. This lead to significant changes in the resulting structures especially of material containing a low amount of oxide. We found evidence that oxides not only influence the surface tension and thus the foamability of a material, but also act as pinning centres in the liquid metal.

1 Research subject

Metallic foams have become a modern material of high interest. In recent years many production processes have been established and it is possible to produce foams made of different metals with a wide range of customizable properties [1]. This leads to a number of applications not only in automotive industries. But still metallic foams have not been able to compete with classical materials in a way that industrial production of large quantities is of interest. One problem is the amount of energy and production steps needed to produce a proper metallic foam. Another is the inhomogeneous pore structure of foams produced by powder metallurgical or gas injection methods.

1.1 Production process

Metallic foams were produced according to the powder metallurgical route developed by the Fraunhofer institute [1]: a metal powder is mixed with a blowing agent and then compacted. This semi-finished product can be brought into a customizable shape. The blowing agent sets free gas when the precursor material is melted. The resulting porous structure is conserved by cooling down the liquid below its melting point. During the described process, parameters like temperature and foaming duration have major influence on the resulting structure. Also material properties like content of oxides, blowing agent and type of alloy exhibit different pore structures.

1.2 The foaming process

During the melting of the precursor material the expansion of the blowing agent leads to the formation of a metallic foam in various stages. One can distinguish four phases the melt undergoes before it is cooled down to solidification. The steps of foam formation are depicted in figure 1 a)-d):

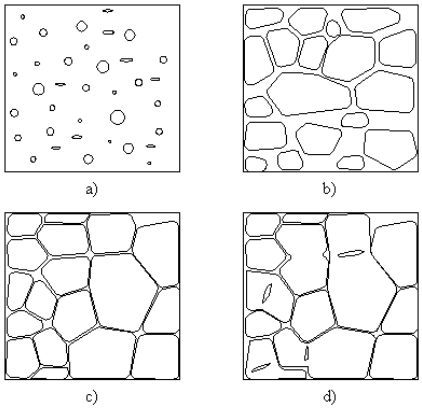


Figure 1. Four steps of foam genesis: a) start of bubble nucleation; b) young foam; c) completed foam; d) coarsened foam

At the very beginning, the material is solid and the blowing agent does not expand. As the temperature increases, the material melts and small bubbles and distortions appear (fig. 1a). These pores are generally of spherical shape since at this stage they do not interact with each other. When the whole sample becomes liquid, the gas expands and the pores start to grow (fig. 1b). In this stage of a so-called young foam we have a two-phase system of gas and liquid metal. Thus the melt is undergoing a change from a wet foam with high liquid fraction to a dry foam with low liquid fraction. This in turn is accompanied by a change of pore shapes: during the growth the former individual bubbles start to interact with each other and undergo a metamorphosis from spherical to polyhedral cells of distributed sizes (fig. 1c).
However, if one analyses a solidified metallic foam one sees a coarse structure and ruptured cells. Also X-ray observations reveal a much more complicated scenario of foam evolution [2]. This can be explained by having a closer look into the two-phase system. In figure 2 the region between gas filled cells and surrounding liquid is shown. This region is built up by the lamellae, which separate three cells and the Plateau-borders at the junctions of four cells. The interface between liquid metal and gaseous cells is characterized by the surface tension γ, which in turn depends on the presence of surface active substances inside the liquid material. As shown in figure 2, the lamellae are bent. Due to the resulting pressure difference, the liquid flows towards the Plateau-borders at the cost of thinning of the lamellae. The junctions

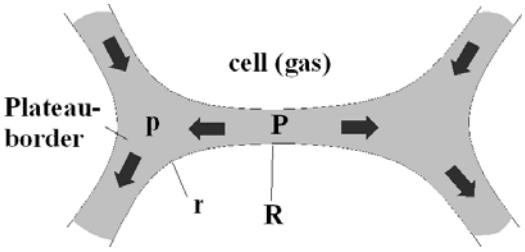


Figure 2. Situation in a liquid metallic foam

between the cells thus contain the major amount of the liquid inside the foam.
In a normal production environment, where gravity is present, the liquid held within the Plateau-borders will flow according to the direction of gravity force. This gravity driven drainage enhances the process described above: the lamellae will rupture and the cells merge. The result is a coarse structure of the foam (fig. 1d).
The longer a liquid metallic foam lasts, the more drainage affects its structure. It can even lead to a collapse of the foam, when all the liquid has drained out. In this context oxides present inside the precursor material play a major role. They are thought to act as surface active

substances and thus stabilize the foam. On the other hand they influence the viscosity of the melted metal which increases the flow of liquid through the junctions. In fact first experiments with precursor materials having a lower than normal oxide content showed a much more unstable foaming process. In addition to this we found evidence for the assumption that oxides also act as a kind of pinning centres inside the liquid.

1.3 Aim of the project

For aqueous steady foams the interaction of drainage and surface tension has been investigated to a certain extent and led to the so-called foam drainage equation (see, for example [3]), describing the liquid flow through a foam. However, in situ observations of metallic foams are much more complicated because of high process temperatures and the opacity of the material. We thus focussed on the investigation of the structures resulting from the foaming process rather than the in situ observation.
The aim of our project is to get a better understanding about the roles surface tension and gravity driven drainage play during the foam formation process. Since both effects are strictly correlated, it is useful to investigate them separately. This was done by processing metallic foams under low gravity conditions during parabolic flights.

2. Experimental set-up

The experimental set-up is shown schematically in figure 3. It consists of two main parts: the

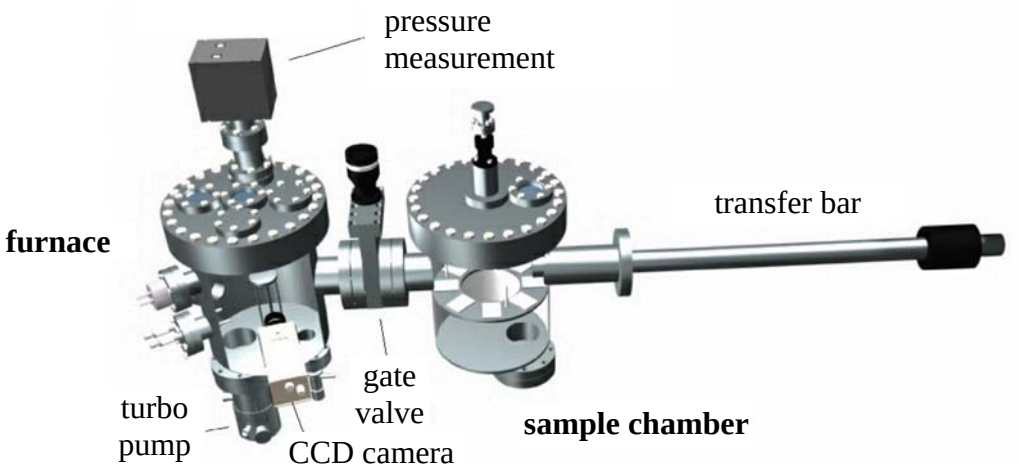


Figure 3. Schematic drawing of the experimental set-up

furnace and the sample chamber. The furnace chamber is equipped with a temperature controlled heating jacket (not shown) to allow air temperatures up to 250°C. The samples are processed on a heating plate inside this chamber, which allows local temperatures of approx. 600°C by means of an infrared light source.
The samples have a size of 20x20x1.8 mm³. They are heated from below and can expand freely. During the process the temperatures of the sample and surrounding air are measured. The stage of foaming can be correlated to the measured temperatures by a video system.
The sample chamber (fig. 3, right side) is needed for keeping the samples during parabolic flights. It is capable to store up to 18 samples in their containers. During the parabolic flight

the samples are transferred manually by a transfer bar into the furnace and placed on the heating plate.

3 Results

In this section we present results obtained from recent parabolic flight campaigns. We processed different types of lead alloys, but we will focus only on the ternary alloy PbSb10Sn10 and lead samples containing different amounts of oxide.

3.1 Ternary alloy

In fig. 4 the temperature and acceleration data of a ternary sample are plotted as an example.

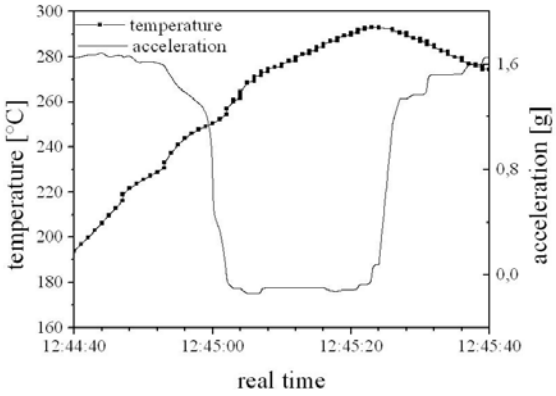


Figure 4. Sample temperature and acceleration for a ternary alloy

Since the sample temperature was measured only by a temperature sensor in contact with the surface of the sample, the melting point is only slightly visible ($T_m \approx 250°C$). The temperature curve was correlated with the video observation to ensure the foaming process took place during the low gravity phase. In the plot the cooling seems not to lead to a decrease of the temperature below the melting point. This has to be attributed to the fact that the container holding the sample is warmed and thus affects the temperature measurement. In several tests we verified that the cooling system is able to freeze the structure in less than 3 seconds so that we are convinced the foaming process was stopped before entering the 2g-phase after the parabola.
In figure 5 photographs of samples processed under different gravity conditions are shown.

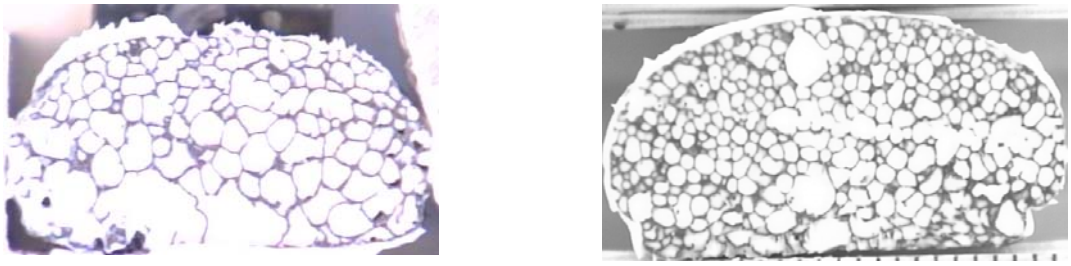


Figure 5. Ternary samples processed under normal g-conditions (left) and low-g-conditions

The foaming times for both samples were 30s. The material is the ternary alloy PbSb10Sn10. A first look shows that the major difference is to find in the pore sizes: the pores of the sample produced under low gravity conditions are much smaller. This is confirmed by the results of the digital image analysis shown in figure 6. The equivalent diameter, which is defined as

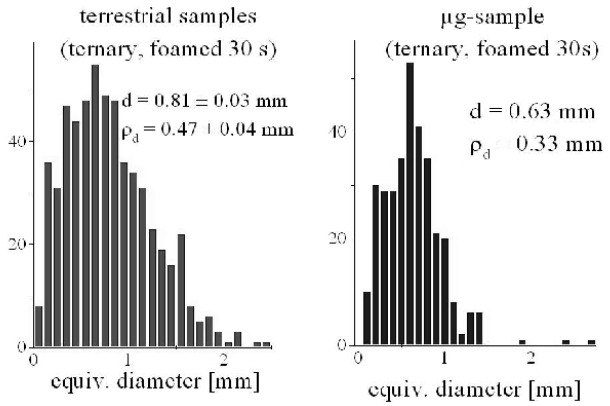
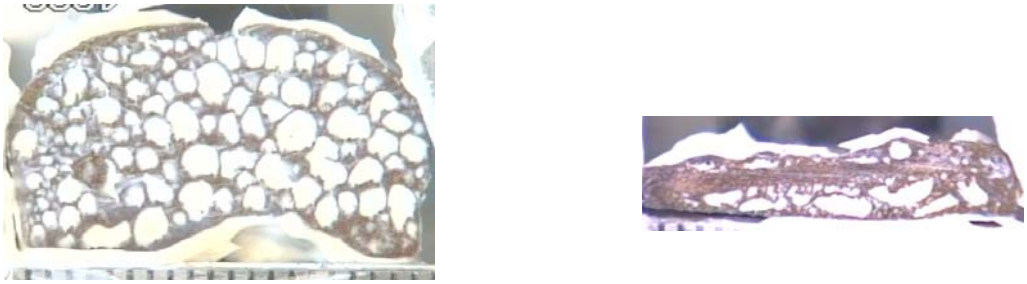


Figure 6. Equivalent diameters for samples foamed under varied gravity conditions.

the diameter of the circle with the same area like the pore, was found to be d = 0.81 mm for the terrestrial sample, while we measured d = 0.63 mm at the µg-sample. The observed difference is most likely due to the fact that the absence of gravity leads to a decrease of liquid flow inside the porous structure and thus reduces the coarsening caused by thinning of lamellae. Gravity driven drainage can thus be assumed to be a major origin of foam coarsening.

3.2 Lead samples with different oxide contents

Oxides present inside the precursor material are known to have major influence on the foamability of a metal. To investigate the role of oxides in more detail, we used normal lead samples on the one hand. For comparison precursor material was produced in a way that assured a significantly lower oxide content. As expected, the material with low oxide content showed a very poor foamability, compared to the material with normal oxide content (see figure 7). During the foaming process this material exhibited a sequence of expansion phases followed by collapses. This leads to distributed expansion factors of the solid samples.



a) b)

Figure 7. Lead samples processed under normal gravity conditions, foaming time 30s
a) normal oxide content; b) low oxide content

In a very recent parabolic flight campaign we were able to produce a small number of samples made of this material under reduced gravity conditions. The statistic is still low and thus the results have to be treated as preliminary. In case of low gravity the expansion behaviour of the low oxide material is steady and not interrupted by collapses, leading to a foam of significantly larger volume, see figure 8b. The material containing a normal amount of oxide seems to be not that strongly affected by the change of gravity conditions (see figure 8a). A detailed analysis of the structures is still in progress, but at this stage we can make the preliminary statement that oxides are not only responsible for the foamability of a material, but also act as pinning centres for growing pores: the structure with low oxide content shows a quite well expanded foam containing only very few pores. Under terrestrial conditions these pores are

a) b)

Figure 8. Lead samples processed under low gravity conditions, foaming time 30s
a) normal oxide content; b) low oxide content (see also fig. 7)

not able to expand against the pressure of the surrounding liquid, while they can expand almost freely under reduced gravity conditions. Thus the absence of oxides seems to lessen the foamability of a material by the reduced number of pinning centres.

4 Summary and outlook

In this article we describe an approach to get a better insight into the details of the foaming process during a powder metallurgical production route. We introduce the separation of surface tensional effects and gravity dependent influences by performing experiments under low gravity conditions. The obtained results give rise to the assumption that gravity driven drainage is mainly responsible for the coarsening of the liquid foam structure. Also recent experiments with varied oxide contents lead to the preliminary assumption that oxides not only influence the surface tension and thus foamability of the material, but also act as pinning centres during the process. In future experiments we plan to investigate the role of oxides in more detail. This will be done by processing more samples during parabolic flights with varied oxide content and, if possible, in nitrogen atmosphere.

Acknowledgements

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