In-situ observation of the foaming process in aluminium by means of X-ray radioscopy

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Abstract

Aluminium foams were produced by means of the powder-metallurgical method. During foaming in the furnace the foams were subjected to a homogeneous, synchrotron-generated X-ray beam of 15 mm height and 40 mm width with energies between 30 and 70 kV according to the sample composition and depth of the foam columns which varied between 7 and 20 mm. The radiographic images were recorded with a 1024x1024 pixel CCD detector. Up to 3 images per second were taken. The various stages of foaming and phenomena known from aqueous foams, namely pore initiation, growth, coalescence of bubbles and drainage of liquid metal could be clearly observed.

1 Introduction

While a lot is known about how to produce metal foams, little is known about how to explain the mechanisms governing the foaming process. This applies to most of the production processes [1,2]. Currently a number of research groups including our own one have launched investigations to clarify some of these mechanisms (see papers in Ref. [3]). While some groups have developed tools that allow for observing the foam during the foaming process, namely the shape of the foaming front or even the evolution of the gas bubbles, others have been trying to simulate the growth of the bubbles with computational methods [4-7]. One manufacturing route has gained particular interest in the past few year: the powder-metallurgical route [8]. The term “powder-metallurgical” refers to the first of the two main processing steps which include compacting a mix of metal powders and a small fraction of a blowing agent powder to a dense billet and foaming this billet in a second step by melting it. The foaming is triggered by the decomposition of the blowing agent (usually $TiH_2 \rightarrow Ti + H_2$) during its melting and takes place in the semi-liquid or liquid state of the metal or alloy.

The foaming process is very complex because many phases are involved: there is a gaseous phase, a liquid phase which is usually a low melting eutectic component of the melting alloy, and a solid phase which consists of the residual solid component of the melting alloy and also oxides which are inevitable when processing powders and which are always present in the powder compacts [7]. The complexity of the foaming process becomes evident if one prepares samples in various stages of expansion and carries out metallographic [4,7] or computer-tomographic [9] investigations on them. One can see, e.g., how metal powder mixes form an alloy during foaming or how the cells of the foam are formed. However, the disadvantage of such ex-situ experiments is that each foam stage investigated belongs to a different foam sample and that the evolution of features cannot be followed in one sample. In-situ (real-time) investigations were limited to the measurement of temperature and volume of expanding foams in the past [7]. To obtain more information we carried out real-time X-ray radioscopic investigations of expanding foams and obtained a picture of the internal structure of metal foams this way.

2 Experimental Procedure

A metal powder blend Al + 7 wt.% Si (purity 99.5%, -100 mesh) was mixed with 0.6 wt.% of powdered titanium hydride (TiH_2) after which the mix was hot pressed at 450°C and 120 MPa. The resulting tablets were virtually dense (<0.75% porosity). Foaming of the alloy was triggered by heating tablets to the melting range of AlSi7 (577-620°C) in normal atmosphere. This caused partial melting of the alloy, hydrogen release by the blowing agent and formation and inflation of bubbles. The fully expanded foams contained approximately spherical bubbles of 1 to 5 mm diameter occupying 80-90% of the total volume.

In order to observe the evolution of the internal structure of metal foams in real time, foams were generated in a furnace which was equipped with two water-cooled Al windows through which a synchrotron X-ray beam could pass (see Fig. 1). The beam, monochromatized to 33.17 keV, generated an absorption radiograph which was captured with an electronic detector system based on a 1024x1024 pixel CCD camera with 40 µm pixel size. The CCD camera was read out at frequencies between 2 and 3 Hz. The entire foaming experiment took a few minutes, corresponding to 500-900 radiographs for each of the 60 individual experiments carried out. Synchrotron beams had to be used because of their high intensity and very low divergence. Conventional micro-focus X-ray tubes have been used for real time observations of liquid metals, but they do neither allow for obtaining sufficiently sharp images of the thin structures nor for exposure times well below one second.

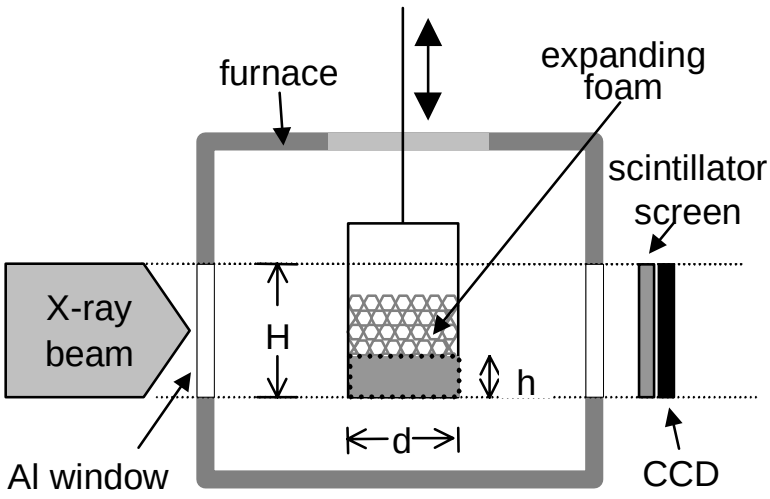

Figure 1. Experimental set-up for real-time radioscopy. The beam is H = 15 mm high and 40 mm wide, the samples were between *h* = 4 and 7 mm high before foaming, their thickness ranged from *d* = 6 to 20 mm.

3 Example

Figure 2 shows 10 evolution steps of a AlSi7 foam. The times given refer to the beginning of the foaming process which was about 2 minutes after putting the foamable sample into the furnace pre-heated to 700°C. The first frame shows first pore formation and some delaminations on the top surface. Quite clearly, the initial pore formation does not create round voids but oblate, crack-like pores. This is a known effect [7] and is due to gas formation in the solid state before the metal starts to melt. The subsequent rise of the foam leads to more and more rounded pores (frames 2 to 5). The

final volume of the foam expansion is reached in frame 7 after 113 seconds. Foams growth comes to an end and only coalescence leads to a further evolution of the structure. One can quite clearly see that the average pore size (although difficult to determine from the radiographs) increases as cell walls rupture. The coalescence rate is highest in early stages and gets lower as the foam gets over-aged. A fairly stable state is reached after about 5 minutes. The remaining cell walls remain unchanged and little drainage and coalescence is observed at this stage. This is compatible to previous measurements with a dilatometer [7]. In the entire foaming experiment little drainage can be seen. This is different at higher temperatures when the viscosity of the melt decreases.

4 Summary

X-ray radioscopic real-time measurements allow for an observation of the internal structure of metal foams during their formation. Details such as individual films and Plateau borders and their change in time can be resolved. Future work will also include further radioscopic studies with better resolutions (10 μm or less) and higher imaging frequencies (up to 10 Hz) to capture more details of the metal foaming process.

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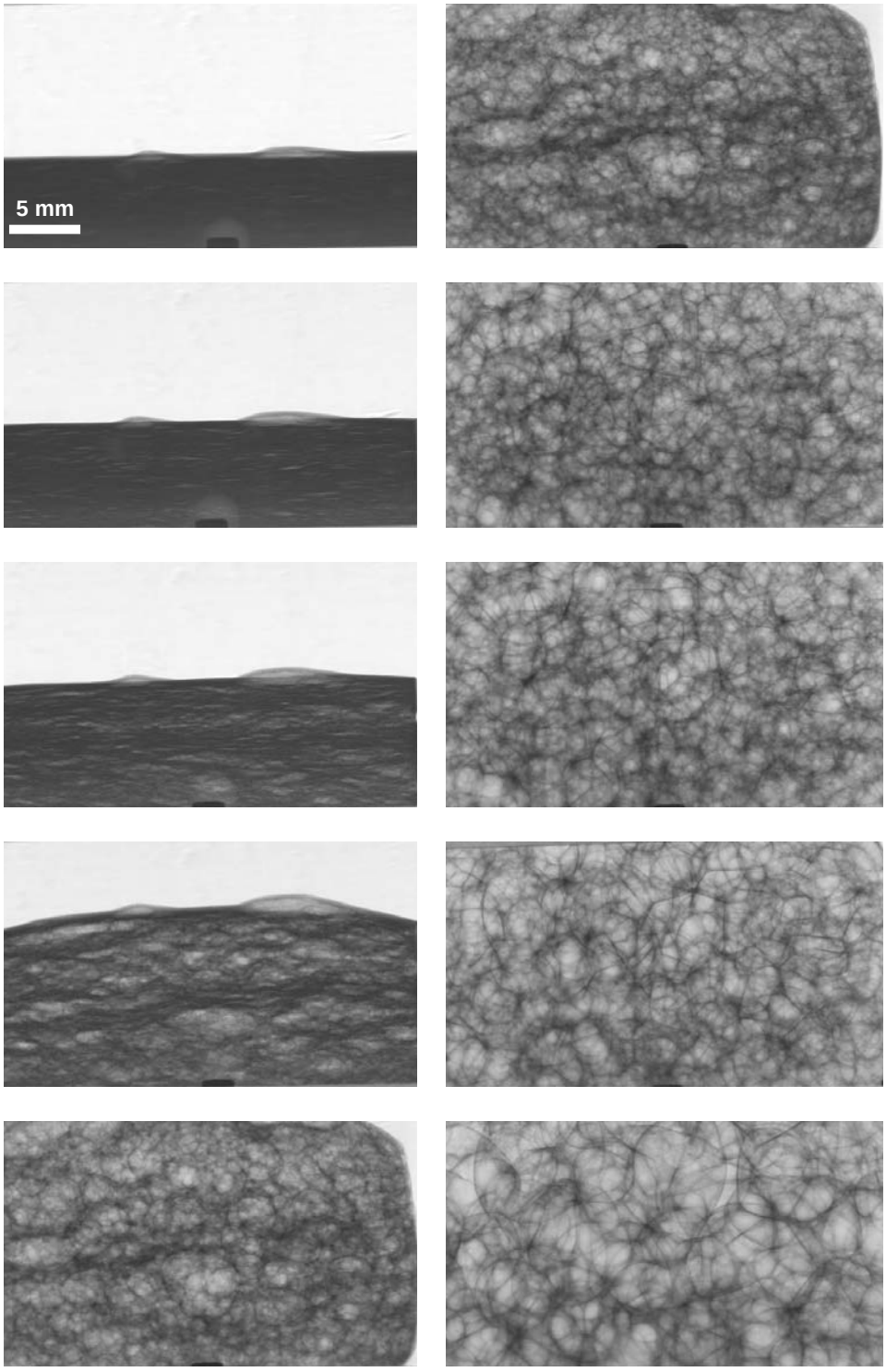

Figure 2. X-ray radiogrammes of an expanding AlSi7 foam. Images are ordered column-wise. Foaming times are: 0, 29, 53, 76, 88, 113, 120, 143, 302 seconds.