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Real-time x-ray investigation of aluminium foam sandwich production

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Sandwich structures with an aluminium foam core and aluminium face sheets were produced by making use of the powder compact melting method. For this, metal powders and a powdered blowing agent were mixed and densified to a foamable precursor material which was then bonded to two aluminium face sheets by roll-cladding in a second step. Finally, the resulting composite was foamed by heating it up to the melting point of the foamable core layer. Foam evolution was monitored in-situ and in real-time by synchrotron x-ray radioscopy with a spatial resolution of 40 μm and a time resolution of 500 ms. The various stages of the sandwich formation process could be identified. Tests at different temperatures allowed for a discussion of technologically relevant process parameters. Finally, the evolution of a crack in the foamable precursor could be investigated.

1 Introduction

Cellular metals can be produced by a variety of different methods [1-3]. Among these the actual foaming methods are especially attractive because they allow for manufacturing fairly inexpensive closed-cell materials with promising mechanical properties. By „foaming“ one usually means releasing gas in a liquid, ensuring that the gas bubbles do not escape and by finally stabilising the resulting liquid foam by cooling. A manufacturing method for producing closed-cell metal foams from metal powders was developed some years ago [4-6]. The process consists of mixing powdered metal and a powdered blowing agent and compacting the mix to a dense semi-finished product (called „foamable precursor material“) by hot pressing, extrusion, powder rolling or some other method. In a final step, the precursor material is foamed by heating it up to its melting point. This softens the metal and simultaneously forces the blowing agent to decompose and to release gas, thus forming bubbles in the semi-molten metal and creating a highly porous structure. For aluminium alloys titanium hydride (TiH_2) is the preferred blowing agent. TiH_2 releases hydrogen gas already at 400°C, but the main decomposition takes place between 550°C and 600°C. The content of TiH_2 in the alloy is usually 0.5 wt.%., corresponding to a total released hydrogen volume of about 20 times the initial volume of the precursor material [6].

For structural applications one often uses foam in conjunction with conventional dense metal structures such as sheets, columns or more complex shaped hollow metal structures. This allows to optimise mechanical properties for a given loading situation [7] and also permits “hiding” the metal foam inside a closed and dense structure which is advantageous, e.g., from the viewpoint of corrosion protection. Such composites containing aluminium foam produced by the method just described can be made in various ways. The most obvious and straight-forward way is by adhesive bonding of pre-fabricated aluminium foams and, e.g., flat

face sheets. However, this approach has certain disadvantages and is not feasible in all cases. An alternative and preferable way is to create composites during foaming. Foam-filled columns can be made by inserting foamable precursor material inside a column and heating up column and foam simultaneously. The foam will eventually rise and fill the column. Another possibility is to start from a foamed part and to coat it with aluminium by thermal spraying [**8**]. This way a dense outer skin can be prepared. Yet another way is to use the foam as a core for die-casting [**9,10**]. Sandwich panels can be manufactured in a very elegant way by roll-cladding face sheets to a sheet of foamable precursor material, to create the desired shape in an optional working step and finally by foaming the entire composite [**11,12**]. Foaming will create a highly porous core structure without melting the face sheets if the melting points of the foam and the face sheets are different and process parameters are chosen appropriately.

Metal foams have been characterised thoroughly with respect to their morphology, mechanical properties and other materials properties relevant for potential applications [**3,7**]. However, there is not much work on how the foam emerges from the liquid, how it changes with time and what mechanisms are responsible for its formation. Most of the work carried out in the past relied on so-called “ex-situ”-investigations: a foam is produced by heating up a precursor. After a given time the foaming process is interrupted and the resulting solid foam is analysed. By varying the time between the beginning of the experiment and the interruption of foaming one can obtain an ensemble of samples which reflect the evolution of the foam and can analyse these samples, e.g., by microscopy or metallography [**4,6,13-15**]. The disadvantage of this method clearly is that each stage of the process is represented by a different sample. As the evolution of foams is a statistical process one cannot follow the evolution of a particular foam feature in this way. If one discovers defects in the material there is neither a way to trace back the origin of the defect nor a way to get a picture of its future evolution because the other samples of the ensemble will usually not show this particular defect. “In-situ” observations in real-time are a way to lift this restriction. A study of the foaming kinetics was carried out by one of the authors by determining the volume and the temperature of an expanding foam during expansion [**13**]. Although very helpful, this method does not yield any information about the internal configuration of the foam and its evolution. By using x-ray radioscopy this problem could be overcome recently and real-time images of expanding metal foams with a high spatial and time resolution could be obtained [**16**].

In this paper we study the foaming behaviour of aluminium-based sandwich panels using x-ray radioscopy. Whereas the more fundamental work presented in Ref. **16** aimed on clarifying the processes occurring in a foaming metal, the present work is more technologically oriented. We observe sandwich samples foaming at different temperatures and identify the various stages of their formation. Moreover, we discuss the evolution of a defect in the precursor material and demonstrate how radioscopic methods can be used in developing the new technology.

2 Preparation of foamable precursor material

Figure 1 shows the process steps needed for making metal foam sandwiches: aluminium powder (Al99.74, <160µm) was mixed with silicon powder (<100 mm) in a weight ratio 93:7. Taking into account the residual Mg content in the Al powders, this yields a casting alloy which is similar to commercial A356 alloys. To this mix 0.5 wt.% titanium hydride (TiH_2) powder were admixed. For extrusion 50 kg of the powder mix were first compacted to cylindrical billets of 70 to 80% theoretical density by cold isostatic pressing (CIP) at Schunk Sintermetalltechnik GmbH (Gießen, Germany) [**6,13**]. These billets were then pre-heated to temperatures between 350 to 400°C and extruded to rectangular bars of

160x20mm^2 cross-section in a second step in which the horizontal 25 MN direct extrusion machine at Honsel AG, Meschede (Germany) was used. The resulting foamable precursor material was then roll-clad to two AlMn1 (wrought alloy AA 3103) sheets in a series of warm and cold rolling steps. The thickness of the sheets was chosen such that the final rolled product had the desired ratio between core layer and face sheet thickness, namely 1.50 mm for the foamable AlSi7 core layer and 1.35 mm for each face sheet. The foamable AlSi7 core has a melting range from 577 to 620°C while AlMn1 melts at 659°C [**17**]. This difference in temperature is sufficient for foaming the core while keeping the face sheets solid. Metal foams are usually manufactured in a furnace which is kept at a higher temperature than the melting point of the alloys to be foamed in order to minimise the times necessary for heating up the samples. Foaming sandwich panels is therefore a race against time because one must permit the core to expand fully while stopping the process before the face sheets start to melt.

3 Radioscopic Measurements

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 **Figure 2**). The beam, mono-chromatised 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 every 500 ms. The entire foaming experiment took a few minutes, corresponding to 500-950 radiographs for each of the 18 individual experiments which were carried out with varying process temperatures, sample dimensions and orientations of the sandwich composites. 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 [**18,19**], but they do not allow for obtaining sharp images of the thin foam structures and exposure times well below one second.

For the experiments the furnace was pre-heated to a given temperature. Three different values were chosen: 650°C, 700°C and 750°C. The foamable sample was placed onto a sample substrate which was open in the direction of the beam and lowered into the furnace by means of a motor-driven transfer bar. X-ray monitoring was started after about 2 minutes when the foaming process was expected to commence.

4 Results and Discussion

4.1 Stages of foam sandwich evolution

Figure 3 shows a series of radiographs taken from a total of 920 images. The most important stages of foam evolution can be identified, starting from the unfoamed sample in the first frame. One can see the steel substrate on which the sample was supported at the bottom, the right end of the substrate which can be used as an orientation point and a steel pin which held the sample. The pin was inserted into a hole drilled into the foamable sample and creates a slight absorption contrast. The second frame already shows the onset of foaming. The emerging pores are predominantly oriented in horizontal direction which is perpendicular to the foaming direction and also to the direction of powder consolidation. Foam rise is only in one direction at this stage. The upper face sheet freely floats upon the emerging foam layer. The third frame shows that the foam starts to push to the sides and forms a meniscus-shaped lens on the right edge. The fourth image reveals the beginning softening of the face sheets as they start to melt, a corresponding curvature of the upper face sheet and partial melting at the edges of the sandwich. In this stage the foam structure is over-expanded. For making engineering structures the foaming process must be interrupted shortly after the third picture. Nevertheless it is interesting to follow the further process. In the fifth frame the lower face

sheet has almost melted while the dissolution of the upper face sheet is proceeding. Foaming is still ongoing and one observes considerable expansion. Frame 6 shows no more face sheets. The material of the sheets has amalgamated with the foam structure and formed an alloy of an average composition. The foam remains rather stable even after 5 minutes (last frame). Some drainage can be seen but this effect is rather limited at the temperature of the particular experiment (700°C).

4.2 Metallographic study of sandwich formation

In order to obtain a complementary view of expanding metal sandwich structures, metallographic images were made of samples which were foamed to a given expansion stage and were then quenched. Three of these images are shown in **Figure 4**. The unfoamed sample shows a sharp boundary between the foamable core – characterised by the angular-shaped grey silicon particles embedded in the light aluminium matrix - and the dense face sheets at the right. The foamed sample in the middle, which is in an expansion stage corresponding approximately to the third frame in **Figure 3**, shows the typical microstructure of an undereutectic aluminium-silicon alloy. One can easily identify light aluminium-rich grains surrounded by the eutectic phase. The dense face sheet is virtually pore free and shows no structure in the low magnification chosen. The interface between foam and face sheet lies on a straight line and is well defined. The foamed sample on the rhs. of **Figure 4** represents a later expansion stage roughly corresponding to the fourth image in **Figure 3**. It exhibits a notably coarser grain size distribution in the foam and a slightly diffuse boundary between foam and face sheet. The eutectic phase has grown into the former face sheet material by diffusion processes and has locally amalgamated with the face sheet alloy. This is the reason for the very good bonding between foam cores and face sheets in properly manufactured aluminium foam sandwich parts and explains the absence of face sheet delaminations in tensile tests on sandwich structures.

4.3 Temperature dependence

Foaming is strongly temperature dependent. We carried out foaming experiments on identical precursor samples (30 x 11.5 x 4.2 mm^3) at 650°C, 700°C and 750°C nominal furnace temperature and observed the expansion process by means of x-ray radioscopy. The rise of the expanding sandwich was measured quantitatively for each sample. For this, the distance between the upper side of the expanding sandwich and the sample substrate was determined as a function of time. As the upper face sheet is not entirely parallel to the substrate at all times and even becomes vaulted after face sheet melting, three measurements were carried out and averaged: one in the middle and one on each edge of the sample. **Figure 5** displays this quantity as a function of time. For each temperature the origin of time was set to the first visible sign of foam expansion.

The most obvious effect of temperature is its influence on the time scale of foaming. Higher temperatures lead to a much quicker rise of the foam core after foaming has started. A feature common to all three experiments is that expansion levels off after about 30-40 seconds. At this stage core expansion has reached its maximum and the sandwich should be cooled if real components are to be manufactured. The level of this expansion stage strongly depends on foaming temperature. The lowest temperature of 650°C results in a quite marginal foam expansion, while a temperature of 750°C causes very strong expansion. This effect is well known from AlSi7 foams without face sheets [**13**]. After the plateau regime during which the alloy in the foam core gradually melts while the temperature slowly increases, two effects take place: firstly the face sheets start to soften and gradually melt as the temperature starts to approach the furnace temperature and diffusion of alloying elements from the foam core into the face sheet decreases its melting temperature. Only at a furnace temperature of

650°C this effect cannot be observed, because the melting point of the face sheets is not reached. Secondly, foam expansion accelerates due to an increasingly stronger hydrogen evolution and a lowered viscosity of the alloy at higher temperatures. The accelerated expansion leads to a final rise of the whole structure to about 3 times the original height (the volume expansion is higher, about 4-5 times, because there is also some lateral expansion). An important temperature effect can be observed after maximum expansion: while the expanded foam is quite stable at a furnace temperature of 700°C, the foam which is created in the furnace heated to 750°C completely collapses. One observes strong drainage and a pronounced coalescence of bubbles. The reason is the very low viscosity of the overheated melt which leads to a breakdown of the cellular structure. In contrast, drainage effects are rather weak for 700°C and only a gradual coalescence of bubbles can be observed. Only occasionally one observes a certain downward movement of liquid, mostly in form of short pulses after the rupture of a film.

4.4 Defect evolution

As the technological implementation of the production process for aluminium foam sandwiches still suffers from occasional flaws which can be traced back to inadequate process parameters and defects in the foamable material, the x-ray radisoscopic investigations were also meant to help identifying such problems. One nice example for this is shown in **Figure 6**. Foaming of this particular sandwich panel was carried out at a furnace temperature of 750°. The first frame shows an early stage of foaming. The foamable core layer already exhibits a very slight absorption contrast versus the face sheets, indicating that some porosity has already formed at this stage. Moreover, a crack is visible on the right upper side running right through the foamable layer. The second frame, representing a situation just 5 seconds later, reveals that foaming of the core layer takes place in a highly non-uniform way. The limited heat flux through the face sheets leads to a temperature gradient and triggers the foaming process near the interface between face sheets and core layer. This is different for the lower foaming temperature used in the experiment shown in **Figure 3**, where the thermal conductivity of the layers was high enough to lead to a sufficiently uniform distribution of heat. As one can see from **Figure 6**, the crack in the precursor material has deepened after foam initiation and still extends over the entire foam layer. After 22 seconds of continued foaming, however, the core layer is fully expanded and the crack has disappeared. Therefore, the type of defect observed did not lead to a visible flaw in the foam sandwich.

5 Summary and outlook

It was demonstrated that x-ray radioscopy is a suitable tool for observing the metal foaming process in general and the production of aluminium foam sandwich panels in particular. The internal structure of the foam core can be clearly resolved. There is a strong temperature dependence of foaming kinetics and expansion behaviour. Too low temperatures lead to insufficient core expansion and long foaming times. To high temperatures lead to inhomogeneous foaming and bear the danger of face sheet melting if foaming times are not exactly complied with. From a technological viewpoint one has to define the maximum foam expansion which is needed to obtain the desired final sandwich thickness first and then select the temperature which yields this expansion. For the alloys used in the present study furnace temperatures slightly above 700°C, e.g. 725°C, should be a good choice in most cases.

Future work will include a more systematic investigation of the influence of defects on sandwich production. Instead of waiting for defects to appear in individual experiments, they could be deliberately induced, e.g., into the bonding between face sheets and core material, thus allowing for a more thorough study of the origins of flaws in sandwich production. Moreover, the behaviour of a foam structure when it is cooled down after foaming is still

unexplored. As one expects effects of cell gas contraction on foam structure, it is planned to allow for cooling samples during the x-ray measurement in future experiments. Furthermore, plans also include measurements with higher resolutions (10 μm or less) and increased imaging frequencies (up to 10 Hz) to capture more details of the metal foaming process.

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Compressed versions of some x-ray movies can be viewed and downloaded from web-site http://www.metalfoam.net/xraymovies.html.

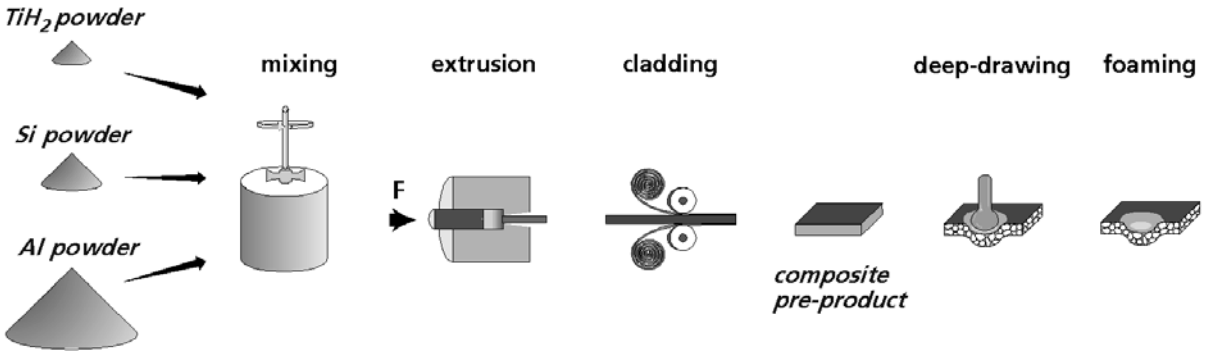

Figure 1. Process steps for making sandwich panels with aluminium foam cores.

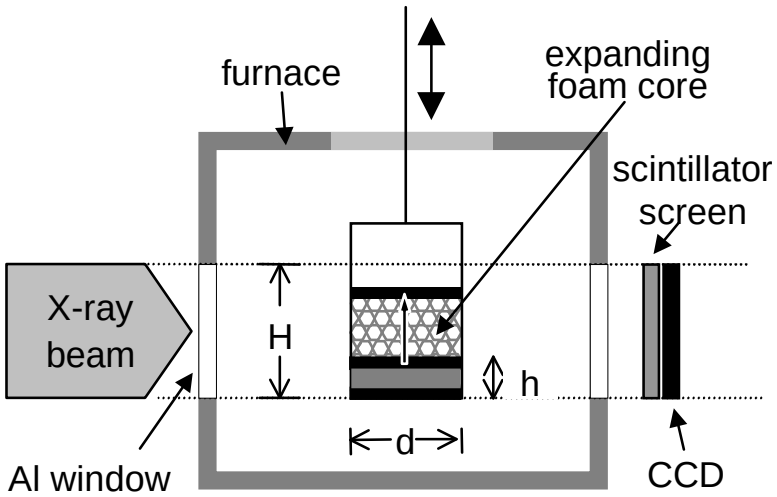

Figure 2. Experimental set-up for real-time radioscopy. The synchrotron beam is H = 15 mm high and 40 mm wide, the samples height *h* was 4.2 mm before foaming, their thickness was *d* = 11.5 mm.

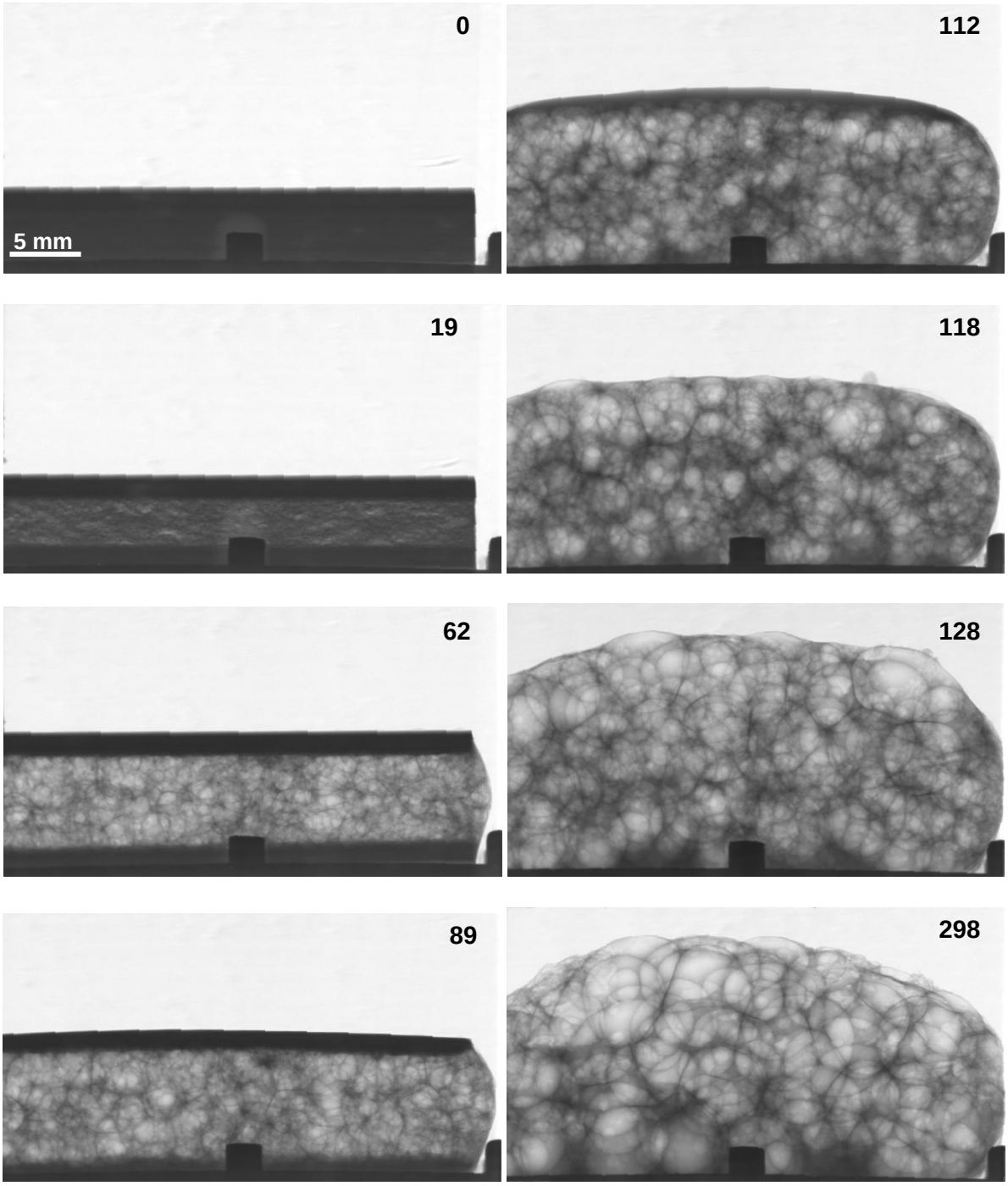

Figure 3. Series of radioscopic images of an expanding AlMn1 / AlSi7 foam / AlMn1 – sandwich. The width of each image is 31.5 mm. The dimensions of the original sample as defined in Fig. 1 were b×h×d = 30×11.5×4.2 mm^3, furnace temperature was set to 700°C. Foaming time is given in seconds with respect to the beginning of the foaming process (about 2 minutes after the cold sample was inserted into the pre-heated furnace)

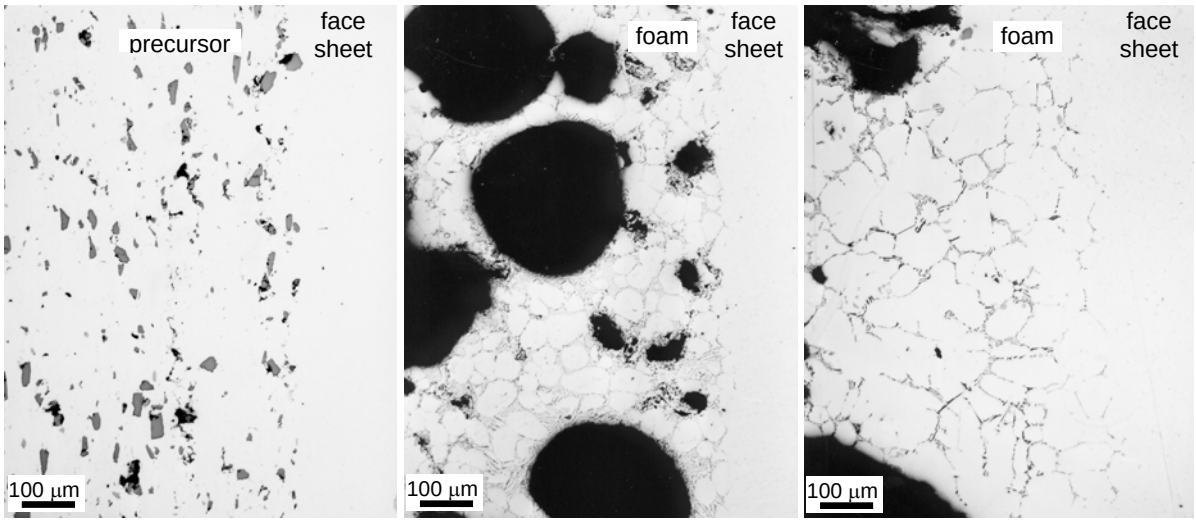

Figure 4. Metallographic images of sandwich structures with an AlSi7 core. Three different expansion stages are shown: left: unfoamed precursor material, middle: sandwich shortly before maximum expansion, right: sandwich at the onset of face sheet melting. The width of each image is 0.75 mm.

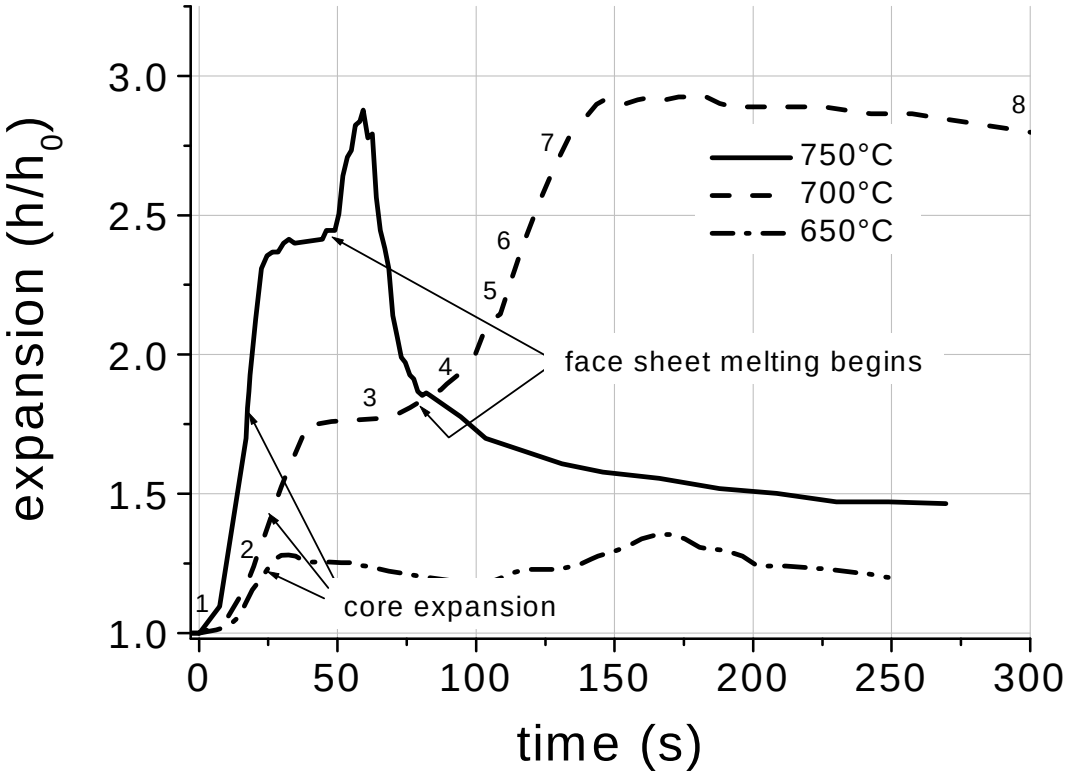

Figure 5. Expansion kinetics of AlMn1/AlSi7-foam/AlMn1 sandwiches for three different furnace temperatures. The averaged expansion height h was directly determined from the radioscopic images. The original samples were h_0=4.2 mm high. The sample labelled "700°C" is shown in **Figure 3**. The 8 expansion stages shown there are marked in the diagramme by black squares and corresponding numbers.

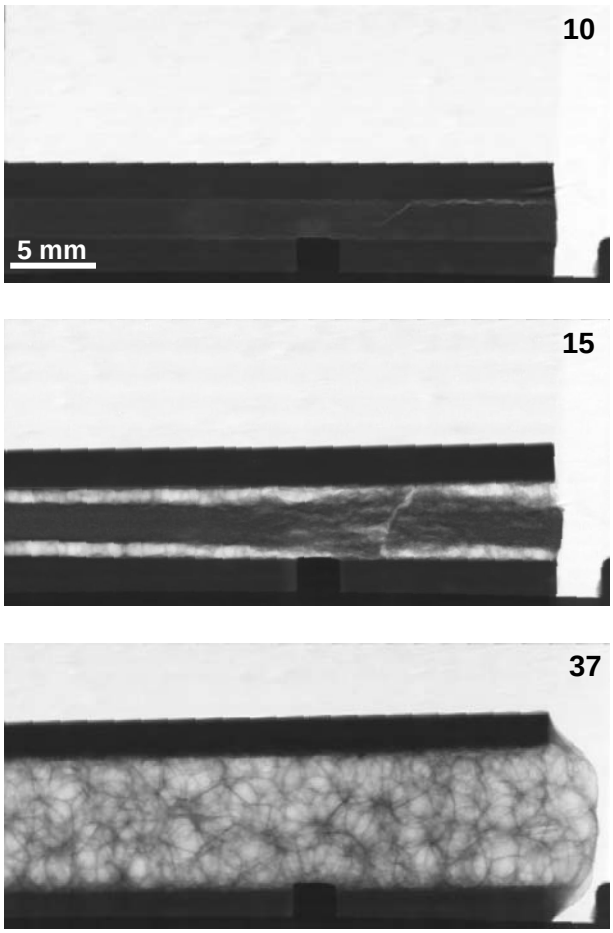

Figure 6. Series of radioscopic images of an expanding AlMn1/AlSi7-foam /AlMn1 – sandwich. Same as in **Figure 3**, but foamed at a furnace temperature of 750°C.