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USANS investigation of early stages of metal foam formation

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Abstract

Metallic foams are on the verge of being used in industrial applications. However, the mechanism of foam creation, especially the early stages, are still unexplored. Ultra small-angle neutron scattering (USANS), performed with the double crystal diffractometer (DCD) at the Geesthacht Neutron Facility (GeNF), is a promising method for obtaining a three-dimensional average of a pore size distribution in a wide size range from about 100 nm to about 20 microns.

1 Introduction

In order to understand how the structure of metal foams is generated it is useful to investigate not only fully expanded foams [1–3],but also foams in the early stages of evolution. The individual samples were prepared by interrupting the foaming process at a given time. In order to study the very early stages of foaming, one has to apply methods with act on the appropriate length scale which ranges from some tens of nanometers to several micrometers. Optical or electron microscopy are suitable for investigations if two-dimensional information is sufficient. Often, however, one is interested in a three-dimensional average of a pore size distribution. Such distributions can be useful as input data for model calculations of the foam generation process [4]. Ultra small-angle neutron scattering (USANS) is a promising method for obtaining this information, because scattering of cold neutrons at very small wave vectors provides exactly the size range required. USANS was therefore chosen for the present study. In order to encounter a fairly simple situation in this study, zinc foams were selected because zinc is known to show a very simple bubble formation pattern when foamed with zirconium hydride, i.e., the bubbles generated are almost spherical.

2 Sample Preparation

Zinc foam samples, Table 1, were prepared in a three-stage process, comprising powder mixing, hot pressing and foaming. Zinc powder was mixed with 0.3 wt.% of zirconium hydride (ZrH_2) powder acting as blowing agent. A comprehensive description of the sampe preparation process is given in [6]. For the USANS measurements 0.4 mm thick foamed samples were used. The densities of the thin slices were determined by buoyancy measurement [6].

composition of sample	foaming time (s)	density (g cm^{-3})	porosity (%)
Zn+0.3 wt.% ZrH_2	0	7.05	1.1
	60	7.06	1.0
	70	7.04	1.2
	80	6.98	2.1
	90	6.99	1.9
	100	6.99	1.9
	110	6.94	2.6
	115	6.43	9.8
	120	5.82	18.4

Table 1. Investigated zinc samples.

3 Experimental Method

The experimental setup of the double crystal diffractometer (DCD) used for USANS studies is schematically shown in figure 1. A part of the neutron beam coming from the cold source of the reactor is reflected by a pre-monochromator (perfect silicon Si(111) crystal, Bragg-angle 45°) and then monochromatised using a quintuple-bounce channel-cut perfect Si(111) crystal with cadmium inserts [5].

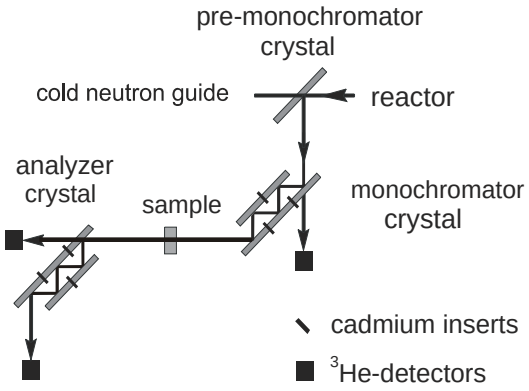

Figure 1. The experimental setup for USANS measurements.

After passing a holding device for changing samples the angle distribution of the neutrons (wavelength λ=0.443 nm) is measured by rotating the analyser crystal which is identical to the monochromator crystal. The average flux at the sample position is about 500 cm^{-2} s^{-1}. The accessible scattering vector range is 10^{-5} - 10^{-1} nm^{-1}. By this inhomogeneities (particles, pores, etc.) from 24 μm down to 0.03 μm in size are detectable.

4 Results

4.1 Foaming behaviour

The volume distribution curves for scattering centres (differential volume fraction *v(D)* expressed in volume percent per unit size of pores and/or blowing agent particles) in various foamed zinc samples are shown in figure 2. The entire information is displayed in the upper in a linear plot, whereas the lower part gives insight into the range of small diameters. Foaming times longer than 60 s lead to an increase in volume fraction of scattering centres on almost all length scales. Obviously, for all times the distribution curves show the log-normal behaviour given by the method of the data analysis. For large sizes the distribution curves are limited by the resolution of DCD. The inset in figure 2 gives volume fractions for some fixed diameters of the scattering centres as a function of foaming time. Foaming for 120 s finally leads to the growth of pores with pore sizes larger than 1 μm with a maximum at 7.5 μm. As one sees from the optical micrographs, quite a significant part of scattering centres are larger than 24 μm [6].

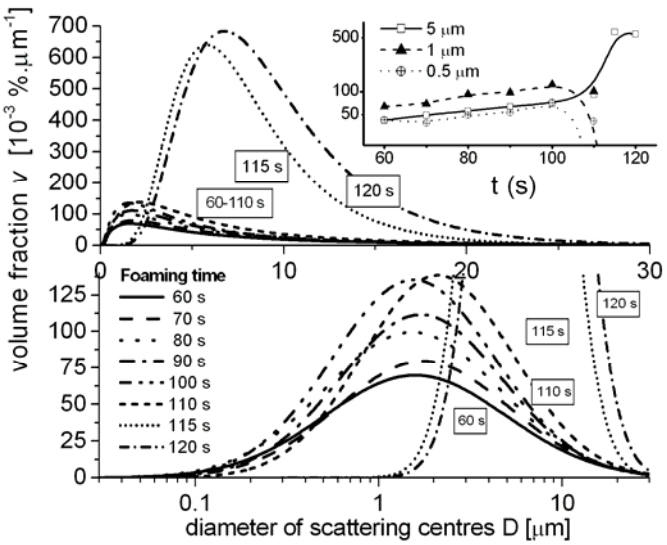

Figure 2. Pore size distributions *v(D)* in zinc foams. Upper: entire size range with inset showing volume fraction of scattering centres with 0.5, 1 and 5 μm size as a function of time, lower: semi-logarithmic representation restricted to scattering centres with small volume fractions.

4.2 Total volume fraction of pores

What becomes clear at this point is that pore inflation starts rather suddenly after a longer stage of gradual expansion and then proceeds very quickly. The macroscopic density shows the same behaviour (Table 1). From the unfoamed sample to the sample foamed up to 110 s the density decreases by just 1.5%, whereas the following 10 seconds of heat treatment result in an expansion of 17%. Similar results can be obtained from the USANS measurements by the calculated total pore volume fraction.

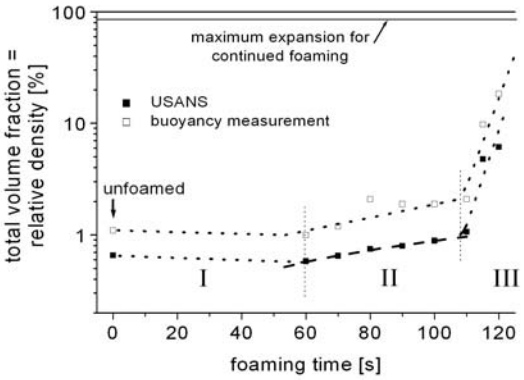

Figure 3. Total volume fraction of pores in foamed zinc as a function of foaming time at foaming temperature of 440 °C as determined by buoyancy measurement and USANS. The dotted lines are merely for orientation, whereas the dashed line represents a linear fit.

The total volume fraction has been primed to emphasise that only the fraction of scattering centres accessible to USANS is included in it (D_{max} = 24 μm in our case). figure 3 compares the total volume fractions determined in this way with conventionally measured relative sample densities. Obviously, the functional dependence of both quantities is very similar, but the total volume fractions determined by USANS are only about half as large. In region I the volume slightly decreases, in region II there is a steady volume increase linear with time, in region III rapid pore formation starts. The discrepancy between the two measurements is up to now not clear, but the inability of this DCD to detect inhomogeneities larger than 24 μm in size may be the reason.

5 Summary

Zinc foams were produced by expanding powder compacts containing zinc powder and zirconium hydride, which acted as blowing agent. By varying the foaming time and quenching the emerging foams after this time, different stages of early foam formation could be prepared. Foaming of powder compacts containing ZrH_2 first leads to a decrease of a sub-micrometer porosity (Table 1) resulting from either solid-state diffusion processes or liquid-phase sintering. After a foaming time of about 60 s a steady creation of porosity in all size ranges is observed. The maximum of the pore size distribution moves to larger diameters while smaller pores disappear. The foaming process is quite slow for a long period, thereafter bubble inflation starts suddenly. In this phase of accelerated bubble growth, a large pore volume is generated with a maximum pore diameter at 7.5 μm for the most mature foam investigated, and many bubbles exceed maximum size measurable using USANS.

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