C. R. Physique 15 (2014) 674–682

Contents lists available at ScienceDirect

Comptes Rendus Physique

www.sciencedirect.com

Liquid and solid foams / Mousses liquides et solides

Structural characterization of solid foams

Caractérisation structurale des mousses solides

Éric Maire *, Jérôme Adrien, Clémence Petit

INSA de Lyon, MATEIS CNRS UMR5510, Université de Lyon, 69621 Villeurbanne, France

ARTICLE INFO

Article history:
Available online 26 September 2014

Keywords:
X-ray tomography
Cellular materials
Image analysis
SEM
3D

Mots-clés :
Tomographie aux rayons X
Matériaux cellulaires
Analyse d'images
MEB
3D

ABSTRACT

For being a useful contribution to the understanding of the properties of solid foams, the characterization of the structure of solid foams has to be performed at different scales. The microstructure of the solid part of the foams has to be analyzed. For this, standard SEM observations are often used. The most important aspect (and the most problematic) remains the characterization of the porous architecture of these materials. The methods introduced in this paper concern both scales and the article discusses the specificity of the experiments in the case of porous materials. X-ray tomography is described in more details because it becomes widely used for this purpose. The paper also shows how the obtained 3D images (sometimes obtained during deformation) can be processed to yield important morphological parameters describing the foams.

RÉSUMÉ

Pour être utile à la compréhension des propriétés des mousses solides, la caractérisation de la microstructure de ces mousses doit s'effectuer à différentes échelles. La microstructure du matériau constituant la phase solide doit être connue. Pour ceci, le MEB est le plus souvent utilisé. L'aspect le plus important (et le plus problématique) est la caractérisation de l'architecture poreuse de ces matériaux. Les méthodes de caractérisation présentées dans cet article concernent ces deux types de caractérisations et les spécificités expérimentales liées à la nature poreuse des échantillons. La tomographie aux rayons X est décrite plus en détail, car c'est la méthode la plus utilisée. L'article montre aussi comment les images 3D obtenues, y compris en cours de déformation, sont traitées pour obtenir les paramètres décrivant la morphologie poreuse des mousses solides.

1. Introduction

Solid Foams (SF), the subject of a part of this thematic issue, are open- or closed-cell foams, having their solid part made of metal, ceramic, or polymer. They form a widely expanding new category of materials available for engineers. Polymer foams have been used for many years in the packaging industry, at a large industrial scale, for thermal insulation of buildings or for comfort applications (seats etc.). Ceramic foams are employed as high temperature filters and gas burners in

* Corresponding author.

http://dx.doi.org/10.1016/j.crhy.2014.09.001
1631-0705/

several devices or processes for many years as well. The most recent family of these materials is that of metallic foams, the properties of which have been reviewed and presented in [1,2]. The possibility of using these materials for new applications has drawn a lot of attention on the family of SFs in the recent years.

The literature on this category of materials is now relatively rich. The book *Cellular Solids* by Gibson and Ashby [3] provides for instance a broad survey of the understanding of the mechanical behavior of a wide range of SFs. It is apparent in this literature that for a good analysis of the macroscopic properties of this type of materials, two scales of microstructure have to be distinguished and analyzed:

- the microstructure of the solid part of the material, at various scales,
- the cellular microstructure also referred to in recent studies as the "architecture" of the SF.

The microstructure of the solid phase in a SF is important to characterize, because most of the time, it differs from its counterpart in the equivalent bulk material. Relying on published and tabulated values of the properties of the bulk solid can then lead to important mistakes. The analysis of this microstructure in terms of inclusions, pores, precipitates, grains, dislocations is very similar to the standard analysis of the bulk equivalent. There will be no description in this paper of all the different techniques available to characterize the solid part at different scales, because this is a rather straightforward conventional discipline generally referred to as "microscopy". In the first part of this paper, we will then give brief examples of the peculiarity of the microstructure of the solid part of SFs, with no emphasis on the technique.

As already mentioned, the architecture will be the term used in the present paper to designate the morphology of the arrangement of the solid and gaseous phases. This has a first-order importance on the properties and has then to be characterized in details. All the crucial morphological parameters (such as orientation, size, shape of each cell) are important to measure for a correct quantification of the cellular microstructure. Ideally, these measurements should be performed on a three-dimensional image of the sample. The second part of this paper will then describe the methods available to obtain a 3D image of a sample at various scales. X-Ray Computed Tomography (XRCT) has appeared in the last two decades to be a very powerful tool allowing one to characterize the architecture of SFs [4–12]. It is nowadays a versatile technique capable of providing non-destructive three-dimensional images of a SF. Some of the most important studies of the architecture of solid foams using X-ray tomography are reviewed in the third part of this paper. In the fourth part, it will be shown how the tomographic images can be used quantitatively to extract key parameters for the cellular microstructure. In the final fifth part, we will give examples of deformation modes studied using dedicated experimental loading rigs.

2. 2D microscopy of solid foams

2.1. SEM observation of the structure

The complex 3D architecture of solid foams requires observation techniques permitting a high depth of field. Amongst the standard available techniques, only SEM shows enough depth of focus to apprehend the structure of these materials. SEM is then commonly used, with a moderate resolution, to measure apparent strut or wall thickness, pore size, etc. Any paper dealing with the characterization of solid foams usually uses SEM to describe the material qualitatively, and many examples can be found in the literature. For illustration purposes, we give in Fig. 1 a visual example obtained on a Fe–Cr foam processed by de-alloying in a molten metallic bath [13]. Note for example a work [14] where the resolution power of the SEM is used to observe nanoporous metal foams. In terms of quantification, SEM can also be used to measure some parameters. The size of the walls or struts can be measured like in the case of nanoporous gold in [15], but one has to be careful not to overestimate the dimensions and to quote these as "apparent" sizes. The size of the windows connecting two pores is often an important morphological parameter to measure [16]. This can be derived reasonably well using SEM with a little bit of tilting if necessary.

Standard SEM is systematically used for ceramic foams. In [17] and [18], EDX has been used to analyze the composition of the foam after fabrication. Although more sensitive to electron irradiation damage, polymer foams have also been studied by SEM for example in [19] where the morphology of the porous network is visualized after cryofracture to obtain a nice and clean surface.

SEM is commonly used to observe the fracture surface of materials. It applies also for solid foams and has been used to help understand the fracture process. In [20], the fracture process of a metal hollow sphere structure in tension fatigue is elucidated using the morphological analysis with SEM. In Goussery et al. [21], the creep and high-temperature deformation behavior is also studied using this method on nickel foams. In [22], this is used to analyze the mechanical properties of replicated Al foams in creep and fatigue. In [23], this is achieved for analyzing the fatigue fracture surface of ERG aluminum foams.

2.2. Polished sections

Standard microscopy techniques can be used to analyze the microstructure of solid foams on polished sections. To achieve this normally simple and rather standard observation, in the special case of solid foams, there are a number of experimental difficulties related to sample preparation. If the fraction of solid is small, the foam is sometimes too weak to be correctly