Materials and Design 105 (2016) 190–200

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Tensile failure observations in sintered steel foam struts revealed by sub-micron contrast-enhanced microtomography

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ARTICLE INFO

Article history:
Received 27 March 2016
Received in revised form 4 May 2016
Accepted 19 May 2016
Available online 20 May 2016

Keywords:
Synchrotron radiation μCT
Metal foam
Micro-tensile testing
Finite-element simulation
Crack propagation
Microporosity

ABSTRACT

Powder metallurgical open-cell steel foams made of hollow struts are of great interest for impact resistance. Here we report experimental results of high-resolution characterisation of single struts, extracted from a commercially available steel foam. Synchrotron radiation X-ray microtomography reveals that the hollow struts are made of triplets of lens shaped rods, connected to each other along the long axis, appearing triangular in cross-sections. The struts have a non-uniform geometry and they include macro and micropores as well as high-density inclusion powder particles, poorly fused with the matrix. Micro-tensile testing showed that tensile failure is accompanied by longitudinal unzipping due to shear along the thinner material found near the hollow corners. Included, higher-density powder particles deflect crack propagation along the interface with the matrix, where much porosity is observed. Results of finite element simulations well match our mechanical tests, revealing plastic deformation due to bending and significant shear stresses arising between neighbouring rods within the same strut. The failure behaviour and the mechanical response of sintered struts in steel foams produced using non-uniformly coated polyurethane templates is the result of an interplay between the triplet geometry, sub-micron pores, precipitates and high-density inclusions.

1. Introduction

Open-cell steel foams are multifunctional materials combining the advantages of cellular structures with superior specific strength [1]. Such foams are used for extremely demanding applications, for example as impact absorbers against micrometeorites and orbital debris [2]. They are also excellent for acoustic and thermal management, for instance when used as liners of jet engine cases where they significantly reduce noise [3]. Both impact resistance and energy absorption are of great interest to the automotive, defense and other industries.

Open-cell stainless steel foams were developed as alternatives to foams made of other metals, e.g. nickel (Ni) or aluminium (Al) [4–6]. Although steel oxidation is of concern, the low costs, recycling ease and wide alloying capacity are huge advantages [4]. Using a pressureless powder metallurgical route [5] it is possible to manufacture a whole range of steel foams, including 316L, 314, 430L, Fe0·6P martensite, and FeCrAl.

Typically, powder materials are compacted before sintering because the density of the sintered steel parts strongly influences the mechanical properties [7]. Indeed, as to be expected, for example increasing the density of sintered Fe-0.85Mo-Ni steels led to an increase in tensile strength and ductility. Further, the distribution of the pores was shown to strongly affect the strain concentration: large and interconnected pores led to more severe strain concentrations than homogeneously distributed small pores [8]. Due to technical limitations, however, open-cell steel foams are produced without pressure so that there is no powder densification [5] and, thus, only lower strut densities as compared to the corresponding bulk sintered steels can be reached, resulting in higher porosities.

Mn serves as an excellent alloying element in powder systems [9–12]. Mn vapour, escaping during heating and reacting with oxygen (O), has been proposed to protect the material during sintering from further oxidation by an effect sometimes termed “self cleaning” [9,10]. However, in addition to the outer surfaces, Mn condenses on inner free surfaces, such as in pores or on included iron (Fe) particles. Because of the high affinity of Mn to O, a continuous oxide network may form during sintering [10] which must be considered when analyzing the microstructure.

Metal foams have been studied in a whole range of settings aimed at understanding the failure behaviour on the macroscale [16–19] and on the microscale [20–25]. Most investigations report on the compressive properties of closed-cell foams [18–20,22–25]. Reports on the fundamentally-different open-cell foams are however scarce [16,17,21]. Further, the majority of publications deals with Al or Ni based foams, and only a handful of studies directly address steel foams [28–32]. The effects of micropores or microstructural inclusions, such as coarse Si

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http://dx.doi.org/10.1016/j.matdes.2016.05.069

phases within the Al-Si eutectic or occluded oxides, were addressed as important factors determining the cell-collapse behaviour of Al foams [20,21,33]. Closed-cell Al foams were studied by means of X-ray microtomography (μCT) [22–25] and 3-D local strain mapping [24–25], and it was shown that cracking initiated at larger micropores with diameters above 30 μm in the cell walls due to significant strain concentrations in the surroundings of such pores [22–25]. Solute segregation was found to be another reason for crack initiation in cell walls [25]. In single struts of open-pore A356 foams, the failure mode was strongly influenced by the amount of included brittle phases, mainly eutectic Si, as shown by SR-μCT and SEM investigations [21,33].

On the cell walls of closed-cell steel foams, micropores and large powder particles were observed [30,32]. Possible effects of these microstructural and sub-micron features on the failure behaviour of the steel foams have, however, not been reported, to the best of our knowledge.

Several studies [17,20,26–27] have reported the tensile failure behaviour of metal foams. For instance, hollow struts of Ni-Cr foam specimens failed in tension by an unzipping process along their longitudinal axis [26]. These hollow struts consist of three solid parts that are connected and form a triangle in cross-section. Unzipping was proposed to arise at imperfections in the strut microstructure in areas where grain boundaries existed at one of the three thin corners of this triangle. The small area of the grain boundary was assumed to induce failure due to shear forces arising as a result of bending and torsion of struts during tensile loading of the foams.

The mechanical properties and the failure behaviour of open-cell metal foams are strongly influenced by the mechanical properties of the strut material. A very relevant test for evaluating the function of foams is therefore micro-tensile testing of single struts. Such experiments are challenging, and they have been reported in several studies [13–15], mainly, however, on Al foam struts. In such studies it was found that the strength of convex triangular Al 6101 foam struts was higher than values reported for bulk material [13]. This was attributed to the different grain structure of the struts where typically a single grain is observed as compared to the usually polycrystalline bulk material specimens. The stress-strain properties of the single crystals in such struts were, therefore, assumed to depend on the size of the specimen, the crystallite orientation relative to the loading axis and the purity of the metals [13]. Further, precipitates and surface-oxide 'scales' were assumed to attribute to strengthening. The fracture strain was also higher than for bulk A356, which may be explained by the size effect and by the smaller reference length as compared to standard bulk specimens [33].

Much remains unknown about the mechanical characteristics and properties of open-cell, sintered steel foams and specifically the contribution of individual hollow struts to the foam behaviour/properties. Open-cell stainless steel foams are nowadays routinely produced via pressureless powder metallurgy production routes resulting in a distinctly different microstructure as compared to bulk material and cast foam struts. Such steel struts have a hollow convex triangular geometry (see Fig. 1, strut cut to reveal interior), similar to the Ni-Cr foams mentioned above [26]. The steel struts contain large numbers of grains, both macro- and microporosity and high-density powder particles [16]. Further, edge defects, i.e. production defects due to insufficient slurry coating of the PU templated, are frequently observed (green arrow, Fig. 1). All these features may yield inferior mechanical properties of the struts as compared with bulk or cast material.

The joint effects of the strut geometry and microstructure on their deformation behaviour have not been reported to date, and the extent to which macro- and microporosities affect the failure behaviour of these hollow struts is still unclear. Here we report on the structure and on the failure behaviour of isolated hollow steel foam struts, tested in tension. Ex-situ micro-mechanical tensile testing of single, isolated struts was performed together with synchrotron-based X-ray micro-tomography (SR-μCT). The results were complemented by finite element (FE) analysis to better understand the experimental outcome and to visualize and quantify the stress/strain distribution within the tested samples.

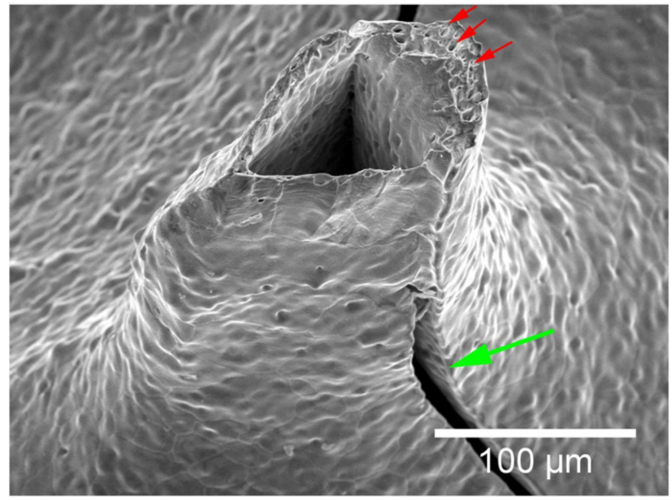

Fig. 1. SEM: typical hollow steel strut surface and cross-section, as visible on a cut surface of a typical foam specimen. The section reveals the distribution of material around a central large void. The strut has a triangular cross-section and consists of inhomogeneous walls. Note the high number of micropores in the three strut walls (red arrows), and the edge defect, an imperfection due to production limitations, seen between two of the three lens-like solid rods comprising the strut (green arrow, note the internal sintered surface in the defect region).

2. Material

Open-cell stainless steel foams (316L) with a density of 10 pores per inch (ppi) were purchased from Hollomet GmbH (Dresden, Germany). The foams were produced by the powder metallurgical method [5]. First, a slurry was prepared from steel powder containing 25 μm sized particles (particle size distribution D90) [16], binder and water. Then polyurethane (PU) foam templates (Foampartner Reisgies, Leverkusen, Germany) were coated with the slurry. After drying the slurry-coated PU foam in air, a two-step heat treatment was carried out: first at ~723 K for 1 h during which the binder and the PU foam burn off in a nitrogen gas atmosphere. Thereafter, further sintering was accomplished at ~1533 K for 1 h in a hydrogen gas atmosphere.

3. Experimental characterisation

3.1. Microstructural investigations

3.1.1. Sample preparation

Two of the steel foam samples were embedded in an epoxy resin for preparation of metallurgical sections. These were ground on SiC paper in several steps (down to an abrasive particle size of 10 μm, corresponding to a grain size of 2400), and subsequently polished for 3 min with a lubricant and diamond suspensions with 3 μm and subsequently 1 μm grain sizes. Final polishing was obtained with an oxide polishing suspension with a grain size of 0.1 μm. While pore[1] visualisation required no further sample preparation steps, some specimens were further etched with V2A etchant at 343 K for 40 s to reveal the microstructure.

3.1.2. Optical and electron microscopy

The etched samples were observed with a Leica DMRM light microscope equipped with a MicroCam 1.3 camera (Leica Microsysteme Vertrieb GmbH, Wetzlar, Germany). SEM (scanning electron microscopy; Zeiss DSM 982 Gemini, Oberkochen, Germany) imaging and EDX (energy-dispersive X-ray spectroscopy; EDAX, Wiesbaden, Germany) mapping of the etched cross-sections were carried out at the Central

[1] Please note that oxides are found in the pores - see Results Section.