STRUCTURE AND COMPRESSION PROPERTIES OF IRON AND IRON-PHOSPHORUS PM FOAMS

M. Kupková, M. Hrubovčáková, A. Zeleňák

Abstract
Prospective materials for synthetic bone graft substitutes - iron and iron-phosphorus open cell foams - were prepared by impregnating an open-cell polyurethane sponge with suitable metal powder slurry, subsequent thermal removing the organic template, and final sintering the residual powder skeleton. The structure and compressive properties of resultant metallic foams were investigated. The results show that the samples behave as if they were solids made up of an interconnected network of ductile struts.

Keywords: open cell foams, iron based, static compression test, stress-strain curve

INTRODUCTION
Biodegradable open cell metallic foams have the potential to serve as scaffolds for engineered bone tissue [1,2]. The reasons for this are clear. If the foam is made of biogenic elements, the body might tolerate both implant and products of its degradation. Mechanical properties of foams can be tailored to match those of healthy bone tissue, thus reducing the problems associated with stress shielding. Open cell metallic foams exhibit a natural bone-like structure, which enables ingrowth of bone tissue and blood vessels. However, to take full advantage of potentially valuable properties of metallic foams, there is still a lot to study.

A feature that characterizes materials with a foam-like structure is their very low density. The big pore volume minimizes the load-bearing cross-sectional area. Consequently, the foams usually cannot withstand large tensile forces. Therefore, deformation experiments are carried out primarily in compression and bending.

When a foam undergoes compression, the stress-strain curve exhibits universal behaviour, no matter what material the foam is made of [3,4]. This curve generally consists of three distinct regions: 1) quasi-elastic linear increase in stress for small compressive strains, 2) “plateau” region characterized by no or very small slope of the stress-strain curve, and 3) region of densification with rapidly increasing stress [3,4].

The quasielastic linear response of foam to increasing strain is controlled by cell edges bending or cell faces stretching. The plateau is associated with a collapse of cells. There are several modes of cell collapse: elastic buckling, plastic yielding or brittle crushing of cell edges depending on the properties of the material the foams are made of. For ductile cell edges, the stress-strain curve is quite smooth in the plateau region, while for brittle edges the stress-strain curves are rather rough. The slope of the plateau region depends on whether the cells are open or closed. Open-cell foams collapse at almost constant load, providing a long flat plateau. For closed-cell foams, the deformation of cell faces and compression of gas within the cells cause a slight increase in stress with...
increasing strain. But for all foams, the lower the foam density the longer the plateau region [3,4].

In the regime of densification, the cell edges and faces touch each other. Consequently, further strain corresponds to compression of the solid itself, providing the final region of rapidly increasing stress.

Iron-phosphorus foam is being actively considered as a potential scaffold for bone tissue engineering [5]. Both iron and phosphorus are biogenic elements, so the scaffold itself and the products of its decomposition are expected to be compatible with the environment of the living human. Phosphorus seems to have a beneficial effect on the interaction between Fe-P foam and osteogenic cell populations. Alloying of Fe with P enables a manipulation of the corrosion rate and mechanical properties of the foam matrix. If Fe-P foam is prepared by a powder metallurgy route, phosphorus enables liquid-phase assisted sintering, which increases sintering density and leads to strengthening of the material. But a higher content of phosphorus causes the formation of brittle intermetallic phases, which results in embrittlement of the material.

This article addresses the mechanical properties of iron and iron-phosphorus open-cell metallic foams, namely their behaviour under compression. We have performed preliminary simple compression tests on Fe and Fe-0.5 wt.% P metallic foams prepared by powder metallurgy replication route from bare and phosphate-coated iron powders. Obtained experimental curves show all aforementioned characteristic features and favour the conclusion that the samples tested are open-cell ductile metallic foams.

EXPERIMENTAL

The carbonyl iron powder (CIP) by BASF (type CC, d50 value 3.8–5.3 μm) consisting of spherical particles composed of 99.5% Fe, 0.05% C, 0.01% N and 0.18% O was used as the starting material. The particles of this powder were coated in a phosphating solution using a modified precipitating method. Phosphated iron powders were dried and calcined in air. Details of the applied method were presented in [6,7]. The content of phosphorus in the final coated powder (determined by a photometric method) was 0.5 wt.%

A reticulated polyurethane sponge (Bulpren S 28133) with the cell size of 1060–1600 μm was entirely impregnated with metal powder slurry. In the next step, the impregnated sponge was kept in a tube furnace Aneta for 2 h at 450°C in a nitrogen atmosphere to remove the organic template by thermal decomposition, and finally the residual metal structure was sintered in reducing atmosphere (10% H2 and 90% N2) to produce open cell iron or iron-phosphorus foam. The sintering temperature has to be carefully selected with respect to the P content. For the Fe materials the temperature was 1120°C, for Fe-P, 1050°C.

The microstructure of experimental samples was observed by a scanning electron microscope (JEOL JSM-7000F, Japan).

Uniaxial compressive tests were performed on cubic specimens of foams on INSTRON equipment. The used ram speed was 0.1 mm/s. The samples had dimensions of about 10 mm x 10 mm x 10 mm and density of 0.29 g·cm⁻³ (Fe material) or of 0.37 g·cm⁻³ (Fe-0.5wt.% P material).
RESULTS

The structure of iron-based foams

The used powder metallurgy replication procedure transformed the polymeric foam template, representing the open network of polymeric struts, to the metallic foam.

![Fig.1. Morphology of metallic foams produced by powder metallurgy route from Fe (a, b) and Fe-0.5 wt.% P (c, d) powders (SEM).](image)

The structure of resultant metallic foams closely resembles the original polyurethane sponge structure, with polymeric struts replaced by hollow metallic struts. Sintering did not change the dimensions of Fe foams, but Fe-P foams shrank by about 10%.

Figure 1 shows the surfaces of metallic foams prepared from bare carbonyl iron powder (Fig.1 a, b) and from phosphate-coated carbonyl iron powder with the resultant content of phosphorus of 0.5 wt.% (Fig.1 c, d). In the microstructures, high fractions of large, almost spherical, macropores with size up to 800 μm are clearly visible. The strut thickness between individual macropores is around 150 - 250 μm. Spherically shaped micropores (size of about 3-5 μm) were also found in all samples.

Compression of metallic foams

During deformation caused by axial compression, the collapse of foam cells was observed to progress layer-by-layer from sample ends until practically full densification occurred. The fully flattened samples still remained in one piece.
The compressive stress-strain curves obtained for our samples are presented in Fig.2. One can see that the curves are smooth and contain a plateau region. Within the plateau region the stress remains constant or increases very slightly with strain.

There are several reasons (or their combinations) for such a behaviour of the stress-strain curve (see Introduction): small relative density of the samples, open-cell character of the solid, ductile material of the cell edges for pure iron and iron-phosphorus alloy containing small content of P, etc.

CONCLUSIONS

The iron-based open-cell foams were prepared by a powder metallurgical replication route from both bare and phosphate-coated iron powders. How the addition of 0.5 wt.% P to the carbonyl iron powder affects the response of resultant metallic foams to the compressive load was investigated. It showed a better response of material to compression in the sense that the plateau region for the Fe-P foam is flatter and longer than that for the Fe foam and the „plateau load” is also higher for the Fe-P sample. Fe-P foams seem to be more suitable for service as load bearing scaffolds in engineered synthetic bone replacements than Fe foams. To obtain quantitative values for relevant parameters, more sensitive measurements are now in progress.

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REFERENCES