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Response of 45S5 Bioglass® foams to tensile loading

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Abstract

The tensile strength test of highly porous ceramic foams has been developed and first results have been obtained on bioactive glass foams. The tested material was a 45S5 Bioglass® derived foam-like scaffold intended for use in bone tissue engineering which was manufactured by Bioglass® slurry coating of polyurethane foam and subsequent sintering. The Bioglass® foam structure was investigated in two states: uncoated (as fabricated) and with a PDLLA polymer coating. The tensile testing procedure is based on fixation of the foam into aluminium pots by a suitable adhesive. Tensile test samples having cross-section of $10 \times 10\ mm^2$ and a length of 30 mm were used for the experiments. Basic fractographic analysis was applied to get relevant information about specimens' behaviour during tensile loading. In Bioglass® based scaffolds, the presence of PDLLA coating led to a significant increase of the fracture strength, which is attributed to the interaction of the polymer phase with propagating cracks, e.g. enabling a crack bridging mechanism to take place.

Keywords: 45S5 Bioglass®; Scaffolds; Foams; Open porosity; Tensile strength

1. Introduction

Gaining full understanding of the response of highly porous (cellular) ceramic foams to different mechanical loading conditions is important because of their potential use in several industrial applications and in the biomedical field. At present, it is common to estimate the mechanical properties of ceramic foams by compressive tests only. To enable the uniform transmission of the applied load to the foam structure different inserts are commonly applied, e.g. thin rubber strips and/or epoxy bonding [1]. The most frequently used mechanical property is the crushing strength being associated in some way with the compression test curve [1–4]. When a compressive load is applied to the foam structure, it will initially yield elastically. At some strain, depending on the sample size, the foam structure begins to buckle and collapse continuously at a relatively constant stress. Depending upon the initial relative density of the foam, this constant collapse will proceed to a relatively high strain level. At a certain point, the stress–strain curve begins to rise, i.e. the compressed foam enters the so-called "densification" phase. The point in the stress–strain curve where it transitions from the elastic to plastic deformation phase (if there is any) defines the crushing strength of the ceramic foam [5]. A similar concept has been exploited to investigate the response of bioactive glass foams to compressive loading [6–8].

Very limited number of works has applied also a modified three or four point bending test to investigate the mechanical behaviour of foams [5,9–13]. The complication with these tests is related to appropriately fixing the samples to avoid the crushing between the rollers (supports) and the tested ceramics foam. Usually suitable interfaces between the roller and the specimen surface are obtained using a thin sheet made of a polymer material which must be sufficiently rigid and tough but not too much to enable load transfer [14].

Tensile loading of ceramic specimens is challenging, being an even more difficult task when highly porous materials must be tested [15]. For example, in brittle materials it is necessary to ensure efficient load transfer and suitable alignment of the specimen with the loading axis of the system. Indeed the brittleness of porous ceramics brings complications in fixating the specimen in the test rig and it is impossible to use standard fixtures based on compression, friction, threaded joint and their combinations. One possibility is to facilitate the adhesion by applying suitable adhesives or resins [15]. Another difficulty

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encountered when testing brittle foam-like materials in tension is the measurement of the uniaxial deformation (elongation). Strain values read by crosshead displacement transducers are not suitable for determination of deformation characteristics and direct placement of contact strain gauge on the specimen is generally not possible due to the risk of specimen surface damage.

It is noteworthy that data on the tensile strength of ceramic foams are almost completely missing in the literature. However, for a complete interpretation of the mechanical behaviour and to validate models predicting their response in given applications, information on the tensile strength of ceramic foams is needed [5,16]. The aim of this study was thus to investigate for the first time the response of brittle bioactive glass foams under tensile loading conditions. These foams are of interest for bone tissue engineering [6]. The effect of polymer coating on the foam performance is also addressed, noting that polymer coating of highly porous scaffolds is being proposed as a suitable approach to enhance the fracture toughness of this type of scaffolds for bone engineering [17].

2. Experimental details

2.1. Material

A 45S5 Bioglass® based foam was considered, which is a suitable scaffold structure being investigated for bone tissue engineering applications [6]. The scaffold fabrication was carried out by the foam replica method introduced in 2006 [6] involving the use of a Bioglass® powder slurry to coat a sacrificial polyurethane (PU) sponge which is then burn out to leave a typical trabecular structure. For the scaffolds used in this study a PU sponge of 60 ppi (pores per inch) was used. Scaffolds produced by this method are highly porous ($P > 90\%$) exhibiting open and interconnected porosity. In addition, the struts of the foams are usually hollow, the result of the presence of the PU sponge template. SEM micrographs of the material structure showing the typical pore size and morphology of the both the uncoated and PDLLA coated foams are shown in Fig. 1.

45S5 Bioglass® foams with polymer coating were also fabricated. For this coating, poly(D, L-) lactic acid (PDLLA) was used. The coating was prepared by immersing foams in a PDLLA solution following the procedure described elsewhere [18].

2.2. Test methodology

For mechanical testing, samples of nominally $10 \times 10\ mm^2$ in cross-section were considered. Typical sample length was 30 mm what allowed a gauge length of about 20 mm. An electromechanical testing machine (Instron 8862) with 1 kN loading cell was used. A cross-head speed of 0.1 mm/min was applied. For transfer of the load to the glass foam specimen a special fixture and a new testing rig were developed. The sample gripping is schematically shown in Fig. 2.

The foam specimen was embedded into two tusked aluminium pots by using an adhesive which ensured homogeneous transfer of loads from the machine fixtures to the specimen. Difficulties connected with the alignment of the specimen and loading axis were tackled by designing special aluminium pots holders [19]. The upper holder was supplied with cardan shaft and claw which was mounted by pivot and the lower claw was connected by a bulb to the shaft.

The adhesive medium used for fixation of the specimen in the pot was Duracryl Plus (Spofa Dental, Czech Republic). This epoxy type adhesive is composed of a powder medium and a liquid activator. The advantage of this composition is an easily controllable viscosity of the fixative liquid and thereby the controllable wettability of specimens with different cell size. This configuration produces suitable conditions for load transfer to the struts of the specimen. The other advantage is the fact that the hardening time of the resin does not exceed 20 min. The shrinkage of the adhesive during solidification and possible formation of tensile stresses imposed on the separate struts must be also taken into account. The adhesive must be thus selected in correspondence with the expected foam properties.

Fig. 3 shows the typical gripping of the struts by the adhesive. In the optimal case the residual stresses associated with adhesive soldering and shrinkage are sufficiently low and thus no microcracks are initiated.

Tensile strength values determined from the maximum force in the loading diagram divided by the cross-sectional area of

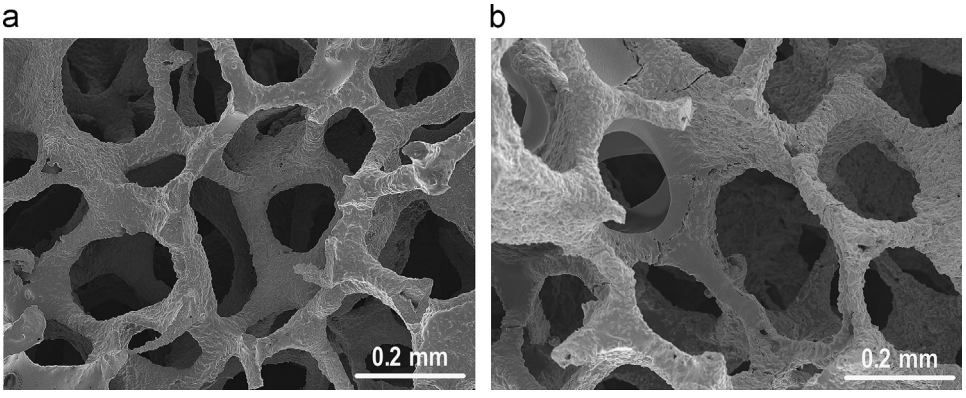

Fig. 1. SEM micrographs showing the microstructure of uncoated (a) and PDLLA coated (b) 45S5 Bioglass® foams after tensile testing. It is revealed that the coating does not reduce significantly the scaffold porosity or pore size.

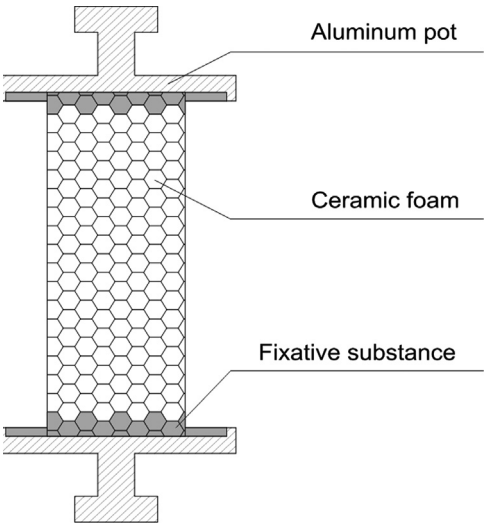

Fig. 2. Schematic drawing of the specimen fixation for tensile strength test of ceramic and glass foams.

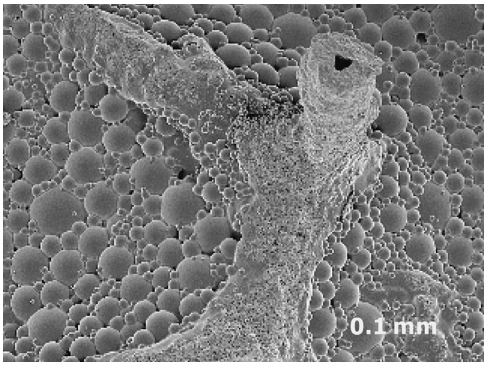

Fig. 3. Example of foam strut coated with epoxy adhesive to achieve suitable gripping of ceramics foam to the aluminium pot and testing rig.

the sample were calculated. The cross-sectional area of the specimens needed for this procedure was measured by digital picture capturing of the fracture surface of the samples by using an optical microscope with digital camera and by carrying out image analysis.

For purposes of comparison with literature data, the compressive strength of uncoated 45S5 Bioglass® foam was also measured. Samples having dimensions $10 \times 10 \times 15$ mm^3 were used for these purposes. The conditions of the compressive strength determination were similar to tensile tests, i.e. loading rate 0.1 mm/min.

3. Results and discussion

3.1. 45S5 Bioglass® foam in compression

Fig. 4 shows the standard response of the non-coated Bioglass® foam to compressive loading. The typical load vs. time curve reflects all the usual stages of the test: linear increase of the load, collapse of one or more struts leading to rapid drop of the load. The maximum stress before this stage was read as the compressive strength value. Struts fractures

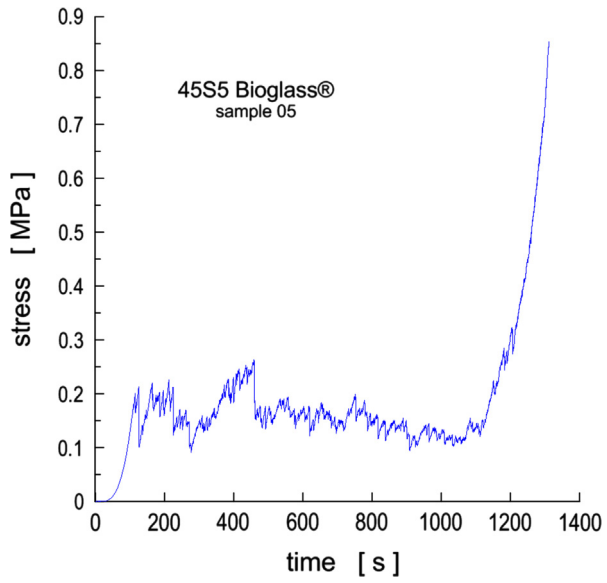

Fig. 4. Typical loading curve in compression of uncoated Bioglass® foam.

continuously go further leading to the structure densification terminated by load increase. The obtained values of compressive strength of non-coated foams are in the range of data published for this type of scaffolds. Based on 6 samples testing the average value of 0.30 ± 0.07 MPa was obtained which is in the range 0.27–0.42 MPa for scaffolds with similar pore size (510–720 μm, [6]).

3.2. Tensile strength

The characteristic tensile load curves for specimens investigated, both uncoated and PDLLA coated foams, are shown in Fig. 5. It is possible to observe a linear increase of the load up to fracture of the first strut suitably oriented to the acting load which is followed by the crack propagation through several struts both represented by the pop-in marked by black arrows. The individual strut fractures (arrows 1 and 2 in Fig. 5a) did not lead to overall specimen failure however and further increase of the load was possible.

The maximum load indicates the conditions for critical damage accumulation in the structure which is usually related to the fracture of several cells, and it is associated with the observed unstable drop of the load (arrow 3). For the uncoated foams (Fig. 5a), after reaching the maximum load, there is sudden decrease of the load, which is attributed to generalised fracture of the struts and obviously a part of the specimen cross-section is broken at this stage (indicated in the photograph in Fig. 6). A part of the structure cross-section preserves its integrity; however the loading curve continues to increase after the sudden drop. This stage associated with fractures of individual struts at lower applied load leads to generalised fracture of the whole cross-section.

Noticeable scatter of tensile strength data is observed (Fig. 7), which is likely caused by the heterogeneity in the distribution characteristics of the cell sizes in the samples. The observed

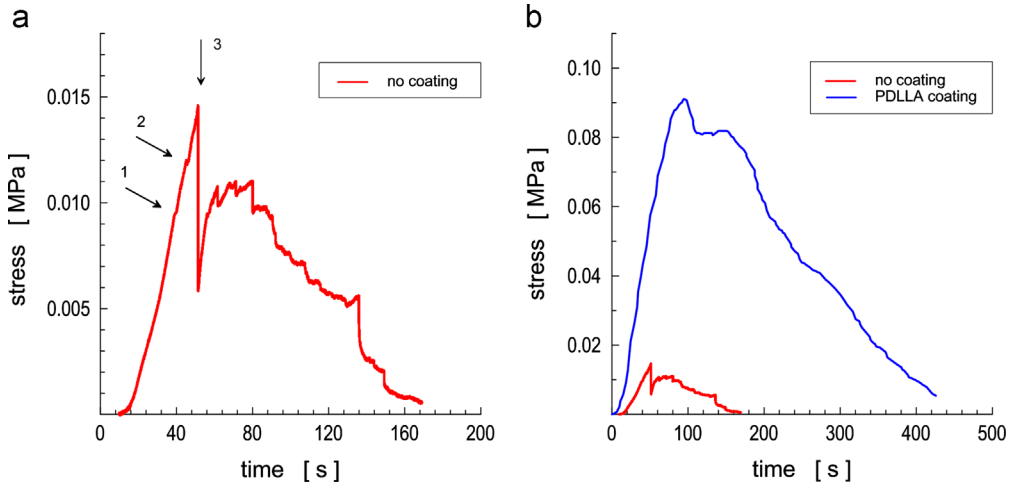

Fig. 5. Loading curves of 45S5 Bioglass® foams: uncoated (a) and with PDLLA coating (b).

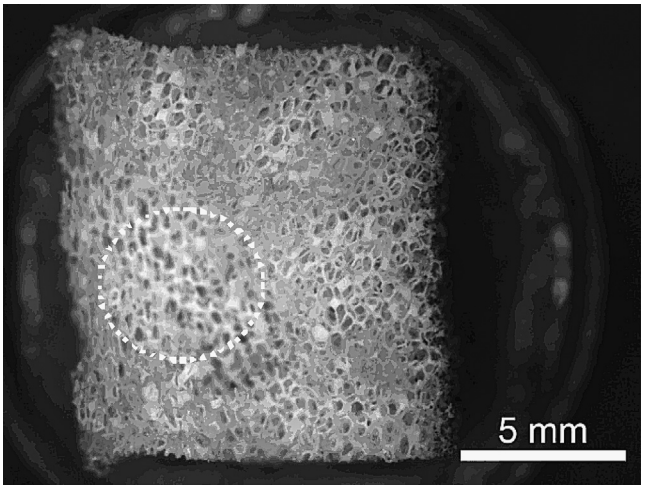

Fig. 6. Macrograph image of the fracture surface of non-coated Bioglass® foam broken during the tensile test.

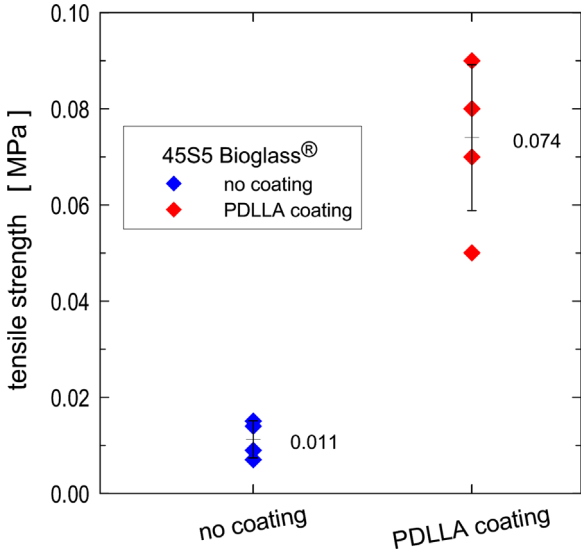

Fig. 7. Comparison of tensile strength values for uncoated (left) and coated (right) 45S5 Bioglass® foams.

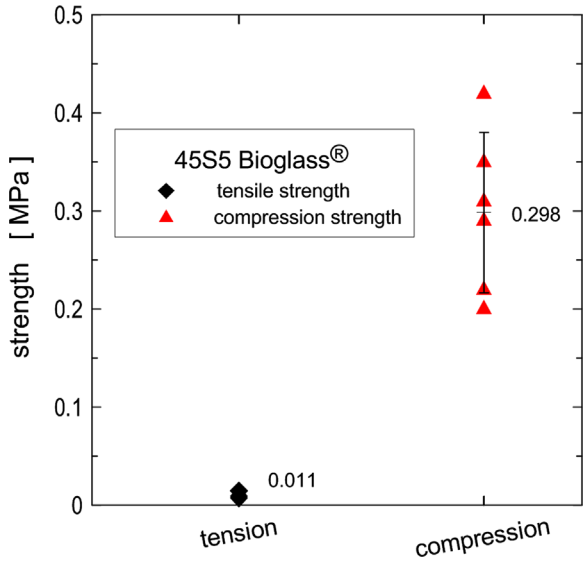

Fig. 8. Fracture strength data for uncoated Bioglass® foams obtained for loading in tension and in compression.

differences in tensile strength values for different foam microstructures confirm the adequate susceptibility of the tensile test measurements to changes of the scaffold strut characteristics, e.g. in this case the presence of the polymer coating.

The comparison of tensile strength and compressive strength values on non-coated foams is presented in Fig. 8. The compressive strength is about one order of magnitude higher compared to tensile strength data. The data reflect the well-known better response of ceramic foams to compression when compared to the response to tensile loads.

3.3. The effect of polymer coating on tensile strength

The tensile strength of the coated material is found to be about five times higher than that of the uncoated structure. The average tensile strength value of uncoated 45S5 Bioglass® foam is 0.011 ± 0.004 MPa (from 4 samples) whereas coated foam

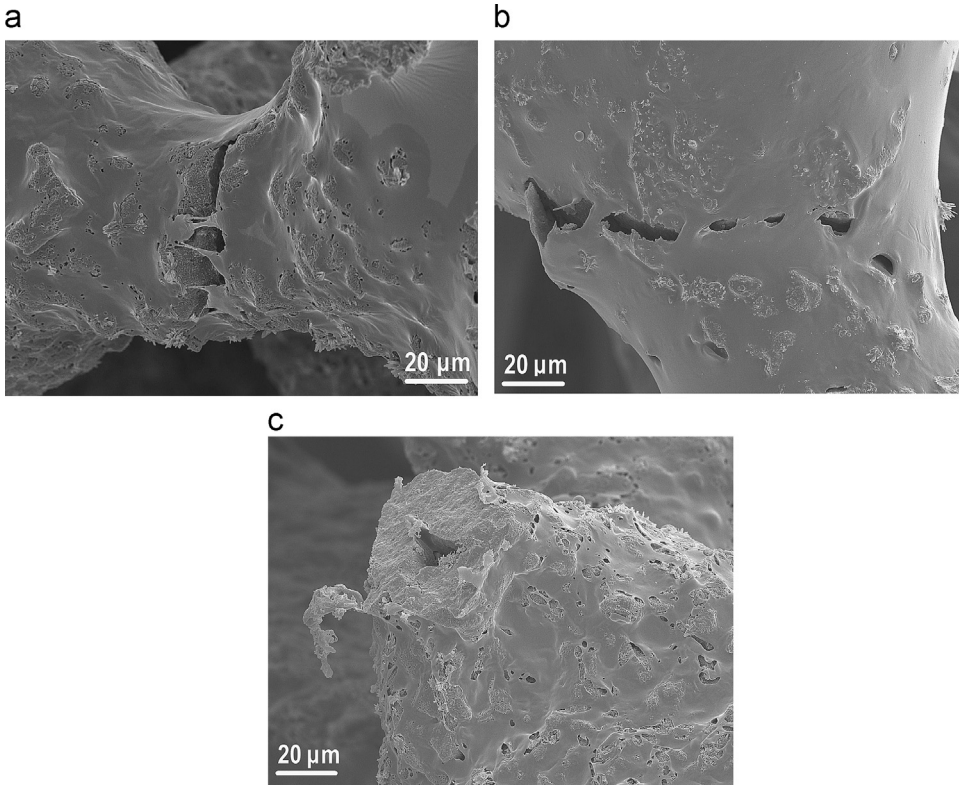

Fig. 9. (a, b) Details of the crack bridging effect caused by PDLLA coating on Bioglass® based scaffold, (c) fracture surface of the strut indicating that the polymer has not infiltrated the central hole.

reaches 0.074 ± 0.016 MPa (from 5 samples). This result is important evidence of the positive effect of the polymer coating on the structural integrity of this type of scaffolds, which has been suggested previously based on compression strength tests only [17,18]. The polymer layer filling surface defects of separate struts and forming at the same time a continuous film on the struts contributes to decrease the effect of the defective (microcrack containing) nature of the sintered bioactive glass structure. Fig. 9 a, b shows how the polymer coating acts at the final stage of the strut fracture when the brittle glass phase is broken but the catastrophic opening of the struts is avoided preventing scaffold fracture. This crack bridging effect, which is discussed also by other authors in similar polymer coated highly porous structures [20,21,18], causes that the decrease of the loading curves is comparably slower compared to the uncoated material (see Fig. 5b). Analysis of fracture surfaces by SEM also revealed the presence of the remaining central hole in the struts (Fig. 9c) indicating that the polymer has not fully infiltrated the (rather dense) struts. A possibility of increasing the amount of polymer incorporated in the struts, e.g. by leaving open pores and cracks in the sintered struts which would enable ingress of the polymer phase to eventually fill also the internal hole, remains as an interesting research task, as this approach is anticipated to increase further the fracture toughness of the scaffolds.

Some of the values presented in Fig. 7 lie outside the typical scatter range. This result can be attributed to the presence of critical defects in the microstructure which are associated with inconsistencies of the foam structures.

4. Conclusions

A testing methodology to determine the tensile strength of highly porous and brittle cellular ceramics was developed and tested on Bioglass® based scaffolds with and without PDLLA coating. The testing method involved embedding and fixing the specimens into the doublet of alumina pots using a carefully chosen gripping adhesive. The homogenous transfer of loads from the machine fixtures to the specimen is ensured in this way while the special design of loading pot holders ensures the alignment of the specimens with the loading axis. In Bioglass® based scaffolds, the presence of PDLLA coating led to a significant increase of the fracture strength, which is attributed to the interaction of the polymer phase with propagating cracks, e.g. enabling a crack bridging mechanism to take place.

Acknowledgements

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