



# Crack bridging modelling in Bioglass® based scaffolds reinforced by poly-vinyl alcohol/microfibrillated cellulose composite coating



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## ABSTRACT

The paper deals with crack bridging modelling in Bioglass® based scaffolds due the presence of a special polymer coating. This includes a careful modelling of the scaffold which is based on x-ray computed micro-tomography (micro-CT) scans and identification of bridging mechanism with the aid of extensive fractographic observations of coated, broken struts. A replacement of the real structure of scaffold by a periodic model utilizing Kelvin cell whose size corresponds to the mean cell size of the real foam is discussed. The struts of the idealized foam are modelled using the beam elements. A detailed computational analysis of crack bridging due to coating film fibrils under plane strain conditions is presented and an improvement of fracture resistance of coated scaffolds is explained.

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## 1. Introduction

The open cell ceramic foams are of the technological interest because of their potential use in a number of industrial branches. They still wait for future more extensive applications because the mechanical response is usually below the needed limits. This is also the case of Bioglass® based scaffolds used in tissue engineering to restore and maintain the function of human bone tissues. To accomplish such demanding task, three-dimensional resorbable porous structures, able to trigger the formation of living tissue are required. The main drawback still impairing the use of Bioglass® based scaffolds in load-bearing applications is their intrinsic brittleness. One of the biggest difficulties in the production of porous bioactive scaffolds from 45S5 Bioglass® is due that the crystallization of different phases, which hinder the densification process. By reviewing the huge amount of papers dealing with Bioglass® scaffolds, it is possible to notice how mechanical properties are remarkably scattered, depending on the sintering route used. Properties of sintered Bioglass® can be widely scattered, depending on the amount of crystalline phase in the amorphous matrix, which can be in turn controlled by tailoring the thermal treatment used for sintering. Progresses have been achieved from this perspective,

by understanding how the glass composition can be tailored to prevent crystallization or by modifying the processing parameters in certain way.

However, the scaffolds properties may be optimised also in different ways. A very promising technique is the reinforcement of Bioglass® scaffolds by polymer-based coating. The idea of creating composite scaffolds by combining a stiff mineral phase with a softer and tougher phase such as a polymer was inspired by observing the structure of a real bone. Bone in fact is a composite material itself, composed of carbonated apatite (65 dry wt.%) which confers structural reinforcement and collagen (35 dry wt.%) which contributes to an increase of flexibility and toughness. The deposition of a thin polymeric coating on bioactive ceramic/glass scaffolds in order to increase their strength and crack resistance is an effective method which has been already reported by several researchers (Green, 1998; Maiti et al., 1984). By reviewing the literature on this topic, it can be found that different polymers, either natural or synthetic have been used for this purpose. Among the natural ones, collagen (Xu et al., 2011), alginate (Mourino et al., 2010), silk (Xiaoxin et al., 2014), gelatin (Metze et al., 2013) and chitosan (Yao et al., 2014) can be mentioned. Concerning the synthetic ones, poly(lactic acid) (Yunos et al., 2013), poly(hydroxybutyrate) (Yunos et al., 2008), PHBV (Yao et al., 2013), poly( $\epsilon$ -caprolactone) (Roohani-Esfahani et al., 2010) were reported as well. Previous studies on dip-coated plates have shown that the thickness of deposited liquid film coatings depends on the

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coating solution properties such as density, surface tension, viscosity as well as withdrawal speed from the coating solution (Yunos et al., 2013; Yao et al., 2013). Moreover, the wettability of the polymer solution determines the adhesion of the resulting film on the interface (Deryagin and Levi, 1964). Beside a thermodynamic driving force, the viscosity of the polymeric solution plays also a role in the infiltration capability, which should be as low as possible in order to be able to infiltrate into the cracks and defect at greater extent (Deryagin and Levi, 1964; Scriven, 1988; Levich, 1962). Besides sufficient mechanical capability, coatings for bone replacement scaffolds must also fulfil other requirements such as biocompatibility, biodegradability and tuneable degradation rate. The possibility of functionalization of scaffolds for controlled drug release is also a desirable goal (Metze et al., 2013; Yunos et al., 2008).

Unfortunately, polymers usually exhibit low elastic modulus (below a few GPa), therefore it is desirable to develop improved biodegradable coatings having higher elastic modulus and strength. From this standpoint, the adhesion between coating and substrate plays a fundamental role since the external loads must be transferred from the struts to the coating through this interface (Martínez-Vázquez, 2014). The preparation and mechanical characterization of Bioglass®-based porous scaffolds reinforced by a composite coating constituted by polyvinyl alcohol (PVA) and microfibrillated cellulose (MFC) was described in the previous paper (Bertolla et al., 2014). It has been proven that PVA coating as reinforcement for bioactive glass based scaffolds by mechanical and microstructural characterization leads to approximately 5 fold increase of compressive strength compared to uncoated ones and the addition of 5 wt.% of MFC fibres led to a 10 fold increase of compressive strength. Also tensile strength has been found to be improved by the PVA/MFC composite coating; the PVA/10%MFC-coated samples exhibited more than 20 fold increase of tensile strength compared to non-coated samples.

This remarkable increase has not been analysed theoretically yet. Hence, the paper aims to shed a light on this issue by investigating crack bridging in Bioglass® based scaffolds due the presence of a special polymer coating. A suitable computational model is developed which allows analysing the crack stability in coated Bioglass® based scaffolds.

## 2. Observed effects of polyvinyl alcohol/microfibrillated cellulose composite coating on mechanical and fracture properties of 45S5 Bioglass® foam

As reported in the previous paper (Bertolla et al., 2014), a fully reticulated polyurethane (PU) foam having 45 pores per linear inch (PPI) was used as sacrificial template for the foam replication method. The PU foam was immersed into ethanol-based Bioglass® slurry, dried and then sintered at 1050 °C for 1 h. The volumetric shrinkage occurring after sintering has been computed from tomography results (See Section 3) and was observed to be nearly isotropic and equal to 26.6%. The distribution of cell sizes and strut diameters was optimally described by the normal distribution function. Mean cell size  $D_c$  and mean strut diameter  $D_s$  after sintering measured by image analysis of low magnification SEM micrographs resulted respectively as 530  $\mu\text{m}$  and 67  $\mu\text{m}$ . The addition of a polymeric coating on the scaffold's surface increased the strength and the work of fracture of the structure. The novelty is the use of a composite coating (Bertolla et al., 2014), comprised of polyvinyl-alcohol (PVA) and micro-fibrillated cellulose (MFC), in order to obtain superior reinforcing effect in comparison with neat-polymer coatings commonly reported in literature. By observing the stress-strain behaviour of polymer-coated ceramic scaffolds obtained either from compressive or tensile tests, two different contributions to toughening and strengthening were detected. The first one was identified as a remarkable increase of the stress value at which the

first fracture event occurred in comparison with non-coated samples. This behaviour has been ascribed to healing/bridging of existing defects on the strut surface and, consequently, reducing the stresses around the defect/ flaw tips which allowed further increase of the remote load without any failure occurring and with the consequent extension of the elastic domain (Gerhardt and Boccaccini, 2010). As the stress peak was reached, cracks initiated and propagated across the struts under the influence of local stress concentrations, the coating film underwent progressive deformation forming thin fibrils which were capable of bridging the crack edges, opposing the crack opening process and preventing the catastrophic fracture of the whole structure (Maiti et al., 1984; Hagiwara and Green, 1987). Deformation and tearing of coating fibrils and debonding of film from the scaffold were responsible for energy dissipation, causing an increase of the overall fracture energy. This phenomenon corresponded to the plateau region which was usually observed on the load displacement curves from tensile and compressive strength tests (Peroglio et al., 2007). The magnitude and appearance of the plateau primarily depended on the mechanical response of the coating to the applied stress (Young's modulus, tensile strength, strain to fracture, damage mode).

The tensile strength of the coated material was found to be about 5–20 times higher than that of the uncoated structure (Bertolla et al., 2014). The average tensile strength value of uncoated 45S5 Bioglass® foam was  $0.011 \pm 0.004$  MPa (from 4 samples) whereas coated foam reached up to  $0.35 \pm 0.05$  MPa for composite coating constituted by polyvinyl alcohol (PVA) and microfibrillated cellulose (MFC). This result is important evidence of the positive effect of the polymer coating on the structural integrity of this type of scaffold, which has been suggested previously based on compression strength tests only (Yunos et al., 2013; Yunos et al., 2008; Bertolla et al., 2014). The polymer layer filled surface defects of separate struts and formed, at the same time, a continuous film on the struts which contributed to a decrease in the effect of the defective (microcracks containing) nature of the sintered bioactive glass structure. Fig. 1 shows how the polymer coating acted at the final stage of the strut fracture, when the brittle glass phase was broken but the catastrophic opening of the struts was avoided preventing scaffold fracture. This crack bridging effect, which has been discussed by other authors in similar polymer coated, highly porous structures (Mourino et al., 2010) causes that the decrease of the loading curves is comparably slower compared to the uncoated material. The experimental results reported in Řehořek et al. (2013) show that non-coated samples turn out to be extremely brittle and weak. For coated samples, pop-in peaks are not observed during the first stage of load increase. Initial fracture events take place only at a remarkably higher load in comparison with non-coated samples and they are followed by a less drastic drop of load. After this point, a plateau stage can be observed, corresponding to toughening mechanisms enabled by deformation of the polymer coating. Fig. 2 shows a fractured strut coated by PVA/MFC. The coating appeared to be homogeneously distributed along the strut surface, forming a continuous thin film (thickness was estimated on the order of few microns) without reducing the porosity of the whole structure.

In previous works, the reinforcement of scaffolds due to the presence of coating was ascribed to a lowering of the stress concentration at the defect sites on the strut surface, allowing a further increase of the remote load without occurrence of any failure (Bertolla et al., 2014; Feng et al., 2011; Bulota et al., 2011). Results from FEM on the two dimensional strut (Bertolla, 2016) confirmed this mechanism, demonstrating that the dominant criterion for the sake of strengthening is the extent of coating infiltration into surface microcracks, see Fig. 3, which is maximized as the polymer reaches the crack tip. In fact, as the polymer reaches the microcrack tip, the highest reduction of stress concentration ahead of the

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