Available online at www.sciencedirect.com
SCIENCE DIRECT
Acta Biomaterialia 2 (2006) 103–107
Acta BIOMATERIALIA
www.actamat-journals.com

Brief communication

Mechanical properties of bioactive glass 9-93 fibres

E. Pirhonen [a,*], L. Moimas [a,c], M. Brink [b]

[a] Inion Oy, Lääkärinkatu 2, FI-33520 Tampere, Finland
[b] Sydväst Polytechnic, Malmgatan 5, FI-20100 Åbo, Finland
[c] University of Trieste, Dimca, Via Valerio, IT-34127 Trieste, Italy

Received 11 November 2004; received in revised form 12 August 2005; accepted 18 August 2005

Abstract

Fibres were manufactured from bioactive glass 9-93 by melt spinning. The manufactured fibres were further characterized by measuring their mechanical properties. The tensile strength of 9-93 glass fibres with a diameter between 20 μm and 140 μm and the flexural strength of glass fibres with a diameter of 500–800 μm were measured. The tensile strength of fibres was highly dependent on fibre diameter. Thin fibres possessed the highest strength, 1625 MPa, compared to the strength of the thickest fibres tested, which was 617 MPa. The flexural strength of glass 9-93 fibres was approximately 1000 MPa and the flexural modulus 64 GPa. The Weibull modulus for tensile and flexural strength values was rather low, at about 2–4.

Keywords: Bioactive glass; Fibre; Mechanical properties; Strength; Weibull modulus

1. Introduction

The need for better biomaterial scaffolds for bone and cartilage tissue engineering applications is currently widely recognized [1,2]. Bioactive glasses have been clinically used over three decades. However, for clinical use, the form of bioactive glasses has been limited to the use of glass as crushed particulates, as the re-processing of bioactive glasses has not been possible due to the crystallization of glass during thermal treatment. Brink et al. studied different glasses in a system which contained boron, sodium, potassium, magnesium, calcium and phosphorus oxides and silica. They extensively studied the crystallization, viscosity–temperature dependence and biological activity of 40 different glass compositions in this system. Ten out of the 40 studied glasses did devitrify when the glass was slowly heated in a high-temperature microscope. Thirty of the studied glasses had large working range properties, which means that the studied glasses did not devitrify when the glass samples were heated slowly to their melting temperatures. The large working range property of those glasses enables the production of fibres from those glasses. Fourteen of these large working range glasses were also bioactive. Glass 9-93 is one of the glasses found from this system which possesses large working range property and is bioactive [3,4]. Use of bioactive glass fibres in manufacturing novel scaffolds may introduce advantages over the materials currently used. Bioactive glass is known to have osteoconductive and also osteopromotive characteristics [5], and shaped as fibres, it shows good strength properties. In using bioactive glass fibres there are also novel methods which can be utilized to easily manufacture open porous structures, a property required for most tissue engineering scaffolds.

The aim of this study was to manufacture fibres from the bioactive glass 9-93 and to study the mechanical properties of these fibres.

* Corresponding author. Tel.: +358 3 230 6600; fax: +358 3 230 6601.
E-mail address: eija.pirhonen@inion.fi (E. Pirhonen).

1742-7061/$ - see front matter
doi:10.1016/j.actbio.2005.08.008

2. Materials and methods

2.1. Manufacturing the glass

Bioactive glass 9-93 contains 12% Na_2O, 15% K_2O, 5% MgO, 1% B_2O_3, 11% CaO, 2% P_2O_5 and 54% SiO_2, all in wt.%. The raw-materials were measured and mixed in a plastic container. The glass was then melted in a platinum crucible for 3 h at 1360 °C. The glass formed was crushed into pieces of approximately 1 cm^3 and re-melted for 3 h at 1360 °C in order to achieve a homogeneous glass block.

2.2. Manufacturing of fibres

The glass manufacturing machinery was built in order to perform melt-spinning trials. The apparatus contained a furnace with an opening at the bottom and a fibre spinning unit which was placed approximately 1 m below the furnace to enable the fibres to fully cool down before spinning to a roll. The glass block was heated in a platinum crucible with 19 orifices at the bottom. Previously in this project the optimum fibrillation parameters were studied by varying the melting temperature and the diameter of the orifices. Furnace temperature of 810 °C and an orifice nozzle diameter of 4 mm were found to be optimal for melt spinning of glass 9-93. The speed of the spinning of fibres could be adjusted by varying the spinning speed and the diameter of the roll and this way the fibre diameter could be adjusted from 15 μm up to 150 μm. The thick fibres for flexural testing were manufactured by slowly pulling the fibres from the nozzle manually.

2.3. Tensile testing

In the tensile testing the ASTM D3379-75 instructions were followed in principle. A gauge length of 50 mm and a testing speed of 1 mm/min were used. A Zwick Z020/TH2A universal materials testing machine with 20 N load cell was used. The diameter of the fibre was measured from both free ends of the fibre sample with a micrometer screw gauge with an accuracy of 1 μm. Fibres with a diameter from 20 μm up to 140 μm were tested. Prior to the testing care was taken, not to touch the fibres to avoid any contamination of the surfaces.

As bioactive glass fibres are brittle they do not have a well-defined strength, but the stress at which they fail mostly depends on the presence of flaws. These may occur randomly along the length of the fibre. The Weibull distribution is a statistical model for describing the scatter in strength data [6]. N strength values are ranked in descending order: the highest strength value having the rank $j = 1$, the next highest $j = 2$, and so on until the smallest, $j = N$. A probability of survivability, $Sj = j/(N + 1)$, is then assigned to each value of strength. A minimum of 30 values of fracture strength are required for statistical validity. A Weibull diagram is obtained by plotting $\ln\ln(1/Sj)$ against $\ln(\text{strength})$. The gradient of the line of regression on $\ln\ln(1/Sj)$ upon $\ln(\text{strength})$ is the Weibull modulus, m, which enables an estimate of the probability that a material will survive a given stress. The Weibull modulus for eight sets of glass 9-93 fibres was evaluated as outlined above. Tensile strength values between the test groups were also analysed with analysis of variance (ANOVA) testing.

In order to study the fracture origins in fibres, 10 fibre samples with diameters between 340 μm and 440 μm were tested as described above and the fracture surfaces were analysed by optical microscope (Smartscope FLASH, Optical Gaging products, Inc., New York, USA).

2.4. Flexural testing

The flexural strength of fibres was measured with a three point bending test fixture with fibres with a diameter between 500 μm and 800 μm. The support span radius of $r = 0.15$ mm was used in the lower jig and $r = 1$ mm for the upper loading nose. The span length was calculated to be 16 × mean nominal diameters for all sample series. The crosshead speed was calculated as presented in ASTM C1161-02c. The crosshead rates were chosen so that the strain rate upon the specimen was of the order of $1.0 \times 10^{-4}\ s^{-1}$. In Table 1 the parameters used in the three-point testing are presented. The strain rate for the three-point mode of loading is as follows:

$$\varepsilon = \frac{6 \cdot d \cdot s}{2 \cdot L} \tag{1}$$

where ε = strain rate, d = specimen thickness, s = crosshead speed, and L = support span.

Flexural strength values between the test groups were analysed with the ANOVA test.

The flexural modulus was calculated by using the following equation:

$$E = \frac{8 \cdot L^3 \cdot (XH - XL)}{6 \cdot \Delta L \cdot \pi \cdot d^4} \tag{2}$$

where L = support span, XH = end of E-modulus determination in kN, XL = beginning of E-modulus determination in kN, ΔL = flexure in mm between XH and XL, and d = specimen diameter. XH and XL were determined from the initial part of the curve where clearly only elastic deformation was present.

Table 1
The table of parameters used in the three-point testing of bioactive glass 9-93 fibres

Fibre diameter (μm)	Number of samples	Crosshead speed (mm/min)	Support span (mm)
500–600	42	0.14	8.8
600–700	45	0.17	10.4
700–800	32	0.19	12.0