DESIGN FOR A FILLER OF AN INTERVERTEBRAL CAGE FOR SPINE TREATMENT ON THE BASIS OF FIBERS AND PARTICULATE COMPOSITES

KAREL BALÍK*, TOMÁŠ SUCHÝ*,**, ZBYNĚK SUCHARDA*, ŠÁRKA RÝGLOVÁ*, FRANTIŠEK DENK***

*Institute of Rock Structure and Mechanics v.v.i., ASCR, Department of Composite and Carbon Materials, V Holešovičkách 41, 182 09 Prague 8, Czech Republic

**Laboratory of Biomechanics, Czech Technical University, Faculty of Mechanical Engineering, Department of Mechanics, Biomechanics and Mechatronics, Technická 4, 166 07 Prague, Czech Republic

***MEDIN Orthopaedics, Inc., Jinonická 329, 150 07 Prague, Czech Republic

E-mail: suchyt@irsm.cas.cz

Submitted August 28, 2009; accepted November 9, 2009

Keywords: Aramid Fibers, Polymer-Matrix Composites, Mechanical Properties, Hydroxyapatite

Composite materials have been developed for applications in the form of an intervertebral cage (for use in spine treatment). The intervertebral cage is composed of a bearing cage made of PEEK and a composite core with the surface contacting the bone surface and ensuring elastic linkage of two vertebral bodies, resulting in good adhesion to the bone. Mechanical analysis of four different kinds of composites (particulate composites with polysiloxane matrix and/or hydroxyapatite (HA)/ tri-calcium phosphate (TCP) nano particles, fabric composites based on polysiloxane matrix and polyamide fibers, and fabric composites with matrix modified by addition of HA nano particles) was performed. The changes in the mechanical properties of fiber composites with the matrix modified by nano hydroxyapatite and the effect of HA and TCP nanofiller volume fractions on the mechanical properties of particulate composites were investigated. For particulate composites, the differences between HA and TCP additives appear only in the case of compression strength. This fact is probably caused by different character of adhesion on the interface between the particulate reinforcement and the matrix. Particulate composites showed mechanical properties similar to a trabecular bone. In the case of fabric composites, a 15 vol.% addition of HA nanoparticles has a favourable effect on both modulus and strength which reach values nearly comparable with a cortical bone.

INTRODUCTION

Degenerative diseases of the spine are one of the most widespread diseases of civilization. Surgical treatment is based on removing the damaged disc and substituting it by an artificial disc or by an intervertebral cage and subsequent symphysis of two adjacent vertebrae. At present, interbody cages on the basis of titanium alloys (e.g. the Ti, Al, V alloy) are most widely used [1]. In recent years, interbody cages made of synthetic materials (e.g. PEEK) have appeared on the market. They have lower rigidity than metals, and exhibit no shadows in X-ray pictures, CT projection or magnetic resonance, etc. [2]. All operation techniques that apply these interbody cages use bone grafts of crushed spongy bone in order to achieve adhesion of two vertebral bodies. This technique leads to weakening of the organism of the patient when there is another operative intervention to collect bone tissue, or there is a risk of infection when bone from another donor is used. The application of composites as non-toxic and biocompatible materials, which can be well penetrated by both tissue and bone, gives the prospect of combining the advantages of the plastics mentioned above with the advantage of avoiding bone grafts.

In earlier investigations performed at our Institute, we have developed composite materials for application in the treatment of specific kinds of spine diseases where intervertebral cages are used. The aim of the research was to find a material for application as a filling element for intervertebral cages, which would meet the requirements of both a suitable biological response of the organism and suitable mechanical behavior, i.e. which would exhibit similar mechanical properties to those of the human cortical bone and which would adhere to the bone in the course of a relatively short healing period.

Carbon/carbon (C/C) composites were the first materials to be investigated. The C/C core can be applied as a biologically "friendly" material. Its biocompatibility and it capacity for osseointegration (giving the possibility of avoiding autospongioplasty and homospongioplasty in the implantation of interbody cages) has been verified in vitro and in vivo. It may be stated that in the construction

of the implants a combination of a cage with a biologically favorable C/C composite core solves the problems of the strength of the fragile core, while its biological benefit remains preserved. Unfortunately, the relative fragility of C/C composites and above all their carbonized matrix leads to a risk connected with the potential release of particles into the organism, either in the course of introducing the implant into the intervertebral space or due to the effect of micromovements during the first period of healing [3, 4].

Another material under investigation for this application was a composite on the basis of glass fibers and polysiloxane matrix [5]. This composite exhibited suitable mechanical properties in comparison with cortical bone, especially almost identical rigidity. In the course of the biological evaluation, the glass composite exhibits a behavior that is in principle inert. However, when the composite structure is modified by adding hydroxyapatite (HA) particles (10 wt.%, particle size 5 μm), creating optimal open pores on the composite surface, the stimulation of the adhesion of the bone cells to the composite can be increased. It has been found that, apart from other factors such as surface energy and chemical composition [6], surface characteristics play an important role in fixing the implant in the patient's body [7]. Osseointegration, leading to a good union of the implant with the surrounding bone tissue, is thus greatly affected by the surface porosity (larger adhesion area) and also by the mechanical stability of the implant [7]. Histological examinations have also shown that the material is not susceptible to abrasion.

The effect of modifications of the fiber composites by enriching their matrix with calcium phosphate powders was further studied in the case of composites based on a polysiloxane matrix and on a balanced aramid fabric [9]. The aim of this investigation was to find a suitable ratio of the additives in the matrix and to verify their effect on the mechanical properties of the composite. In this case, four types of fillers were tested, namely nano and micro HA and nano and micro tri-calcium phosphate (TCP). It has been shown that in general both the microfillers and the nanofillers reduce the modulus of elasticity in bending. The bending strength increased with the addition of nanopowders. Microparticles tend to produce a negative effect of bending strength decrease, which is probably caused by their non-uniform dispersion in the composite matrix or by the formation of aggregates. The addition of nanopowders results in a positive effect on the mechanical properties compared to microparticles. From the point of view of mechanical properties, the addition of 10 – 15 vol.% of nanoparticles appears to be the optimum amount for achieving suitable optimization of the mechanical properties without any changes in the inner structure of the composite.

This paper describes the preparation of two composite materials for application in the form of intervertebral cages fillers: a composite on the basis of an aramid fabric, and polysiloxane matrix added with an optimum amount of a nanofiller. The paper goes on to deal with the preparation of a particulate composite or of an added matrix alone, on the basis of polysiloxane added with nano HA or nano TCP. The effects on the mechanical properties of the volume and the type of additives are investigated.

EXPERIMENTAL

Cores on the basis of fiber composites

Composite materials reinforced by a polyamide fabric (Aramid balanced fabric, fibers HM 215, Hexcel, FR) and Lukosil M 130 polysiloxane matrix (Lučební závody Kolín, Czech Republic) were prepared. In order to increase the osseointegration of the final material, an optimum [9] quantity of nano HA powder additives in the amount of 15 vol.% filler/matrix ($Ca_{10}(P04)_6(OH)_2$, particle size of 50 – 150 nm, Berkeley Advanced Biomaterials Inc., San Leandro, USA) was added to the matrix. The preparation of composites modified in this way consisted mainly in preparing the matrix with the added powders, subsequent impregnation of the fabrics, and assembling the composite from the prepregs. The cores of the interbody cages, identical in shape and dimensions, were prepared from these prepregs. The impregnated fabric was wound on the assembled uncured composite with dimensions of 11×16×3 mm. This procedure provides the possibility to wind the band of impregnated fabric (11×107 mm) to the shape, with dimensions corresponding to the core of the interbody cage (Figure 1), without forming large pores in the bending places of the fabric. In this way, strong connection of the individual layers is ensured.

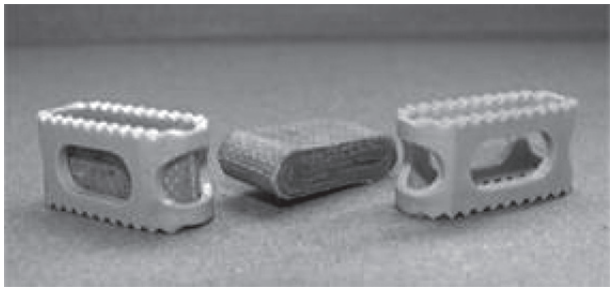

Figure 1. A basic design of the intervertebral cage with a composite core M 130 + Aramid + HA

The sample wound in this way is then inserted into the press mould, and there it is subsequently heated to 135°C for 2 hours and cured under pressure 1.1 MPa at 225°C for 4.5 hours. The temperature and the pressing cycle correspond to the optimum increase in the viscosity of the matrix. The cores cured in this way were then submitted to mechanical compression tests, in order to determine the modulus of elasticity in compression and the compression strength. The course of the tests corresponded to the EN ISO 604 standard.

Cores on the basis of particulate composites

Particulate composite materials based on the Lukosil M 130 polysiloxane matrix (Lučební závody Kolín, Czech Republic) and identical fillers (HA and TCP) as in the case of fabric composites were also prepared. The main problem in preparing composites with a polysiloxane matrix is the content in primary volatile components, i.e. the solvent and humidity, both in the resin and in the particulate reinforcement, as well as the content in the volatile components that are formed, especially water formed in the course of a polycondensation reaction of the resin.

After a series of technological experiments, a compromise technology for preparing the moulding mixture was chosen. It consists in admixing the reinforcement to the solution of the resin for at least 8 hours at room temperature, and subsequent evaporation of the solvent at 60°C for at least 12 hours in vacuum. This intermediate product was then homogenized in a kneading machine at room temperature for at least 4 hours. On the basis of preliminary tests, we chose a curing temperature regime with a starting temperature of 160°C, rising slowly to temperatures of 210 – 220°C, with total exposure time of up to 24 hours. There was a possibility of reducing the time of exposure due to the fact that the preparation is performed at gradually rising temperature and rising pressure, which reduces the volume of the released water vapour. When the sample is cured, it is also sufficiently resistant to the pressure of the enclosed vapours formed at higher temperatures. In addition, a favorable part is played by the particles of the reinforcement, which can absorb a certain amount of the water that forms. It would be advantageous to apply the highest possible pressure, but the increase in temperature at the beginning substantially reduces the viscosity of the resin and there is a danger of it being lost by outflow through the leaks between the pistons and the casing of the mould, as well as settlement of the particulate reinforcement in resin that is not yet cross-linked.

Cylindrical samples cured in this way (10 mm in diameter and 15 mm in height) were subsequently submitted to mechanical pressure tests in order to determine the modulus of elasticity in compression and of strength under compression. The course of the tests corresponded to the EN ISO 604 standard.

An image analysis of the polished sections was performed using LUCIA software, ver. 4.8 (Laboratory Imaging, Inc., Czech Republic). The above mentioned mechanical properties were determined by the Inspekt 100 HT material tester (Hagewald & Peschke, Germany). A statistical analysis for all tests was carried out by nonparametric analysis of variance, at a significance level of 0.05 (Kruskal-Wallis test, Mann-Whitney as a post hoc test).

RESULTS AND DISCUSSIONS

Mechanical properties

Sample designations and summary results are listed in Table 1 and Figures 2, 3.

Table 1. Designation of mechanical test samples

	Sample designation	Material
Fabric composites (FC)	Polysiloxane matrix, Aramid fabric	M130+Aramid
	Polysiloxane matrix, Aramid fabric, 15%vol. nano HA	M130+Aramid+N
Particulate composites	Polysiloxane matrix, nano HA	M130+N
	Polysiloxane matrix, nano TCP	M130+NF

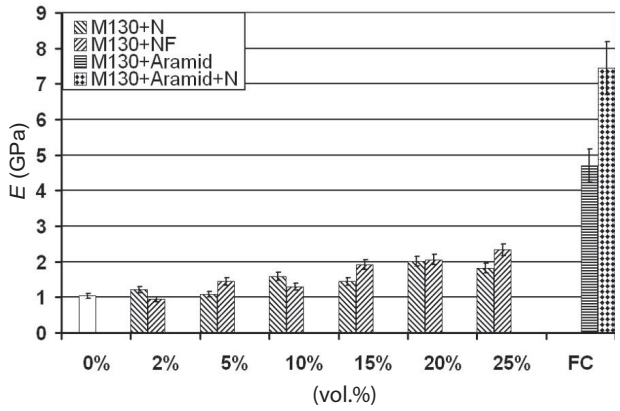

Figure 2. Modulus of elasticity in compression of pure matrix (0 %), particulate (M130+N, M130+NF) and fabric composites (FC).

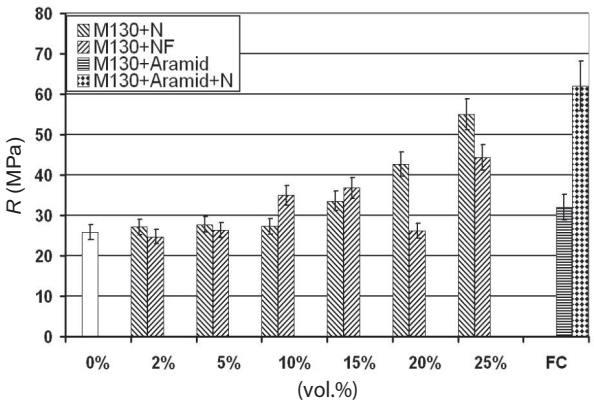

Figure 3. Compression strength of pure matrix (0 %), particulate (M130+N, M130+NF) and fabric composites (FC).

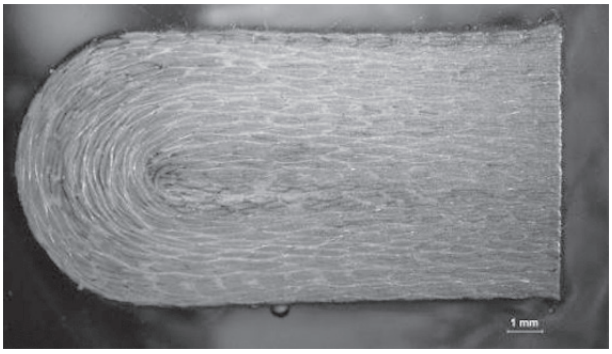

Figure 4. Core of an intervertebral cage made of a composite on the basis of Aramid and a polysiloxane matrix modified with HA.

The addition of a powder additive to the matrix results in an increase in both the modulus of elasticity in compression and the compression strength. This finding does not correspond fully to the changes in the mechanical properties measured by means of a bending test [9]. We can assume that with another type of loading no other course of the dependence on the volume of fillers in the matrix will be obtained. On the basis of the image analysis performed here (Figures 4 and 5), it can be stated that this effect is caused mainly by optimizing the physical properties of the matrix containing additives. This probably results in higher saturation of the reinforcing component and lower loss of matrix in the course of curing. This fact is illustrated by the findings of the image analysis, where we observe not only a higher amount of pores in the bends of the reinforcement but also local delamination in the central part of the core.

In the case of particulate composites (Figures 6 and 7), both the modulus and the strength increases together with increasing volume filling with HA or TCP. Here, for the absolute majority of series, the linear regression with confidence bands is satisfactory. This confirms that these reinforcements also have a generally reinforcing effect for this type of composite. In this case, values in the range of 0.8 to 2.3 GPa are attained, which is a value approximately one order of magnitude lower than the modulus given for the cortical bone. However, we can assume that if the material is applied in the form of a filling of an intervertebral cage, this system will exhibit higher overall rigidity. When comparing nano HA and nano TCP particles, no statistically significant differences in reinforcing effect were observed. We can sum up that the value of the volume filling has a substantial effect on

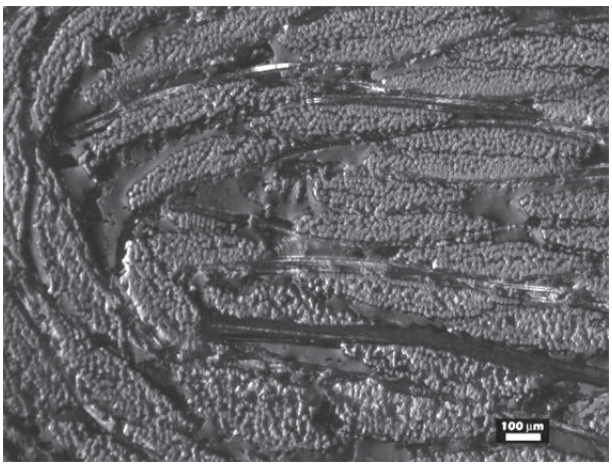

Figure 5. Detail of a wound core (location of the reinforcement bending). The technology of the production ensures strong binding of the individual layers at the site of the fabric winding.

the resulting modulus of elasticity in compression.

In the case of the compression strength of the particulate composites added with nano HA, up to 10 vol.% filling no reinforcing effect of the particles was observed, and with 15 vol.% filling and more the compression strength rises evenly from 25 MPa up to 55 MPa. In the case of nano TCP, the reinforcing effect of the particulate filler appeared in a single jump only above approximately 10 vol.%. In comparison with the nano HA series, the rise in compression strength is slower, and the maximum values attain on average approximately 45 MPa. In general, composites with nano HA particles attain better results than those with nano TCP particles.

This effect is probably caused by a different character of the adhesion between the reinforcement and the matrix on their interface. This emphasizes the difference between the types of particulate reinforcement. This is to be expected, because strength depends on effective transfer between filler and matrix and is affected by particle/matrix adhesion [10]. Differences in particle/matrix adhesion of HA and TCP can possibly be explained by the different character of the interactions at the interface between the matrix and the particles. Unlike TCP, the filler HA in its crystalline form consists of two molecules, hence it contains two OH groups which provide the possibility to form a chemical bond with the OH group

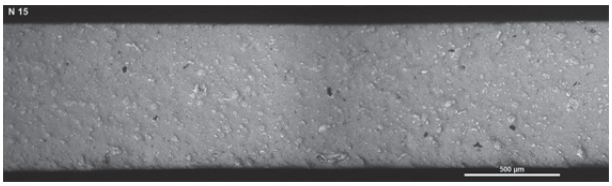

Figure 6. Micrographs of a polished cross-section of particulate composite M130+N (15 vol.%).

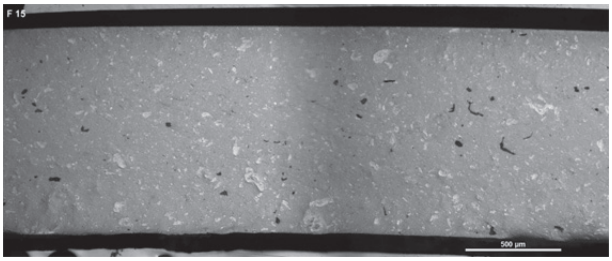

Figure 7. Micrographs of a polished cross-section of particulate composite M130+NF (15 vol.%).

of the polysiloxane under the formation of an -O- bond or of hydrogen bridges. In this case, the quality of the bonding does not depend only on the dimensions of the contact surface (as is apparently the case with TCP, where only the effect of physical or electrostatic interactions at the interface appears). However, composite stiffness is not affected by particle/matrix adhesion, since the fillers have a much larger modulus than the matrix [10], as in this case of polymeric resin and calcium phosphate fillers. This can be illustrated by the fact that there are almost no statistically significant differences in modulus in compression between HA and TCP fillers.

CONCLUSIONS

In the case of fabric composites, 15 vol.% addition of HA nanoparticles has a favourable effect on both modulus and strength which reach values nearly comparable with a cortical bone, but in the case of particulate composites, measured mechanical properties are similar to a trabecular bone. The differences between HA and TCP additives, in the case of particulate composites, appear only in the case of compression strength. This is probably affected by different character of adhesion on the interface between the particulate reinforcement and the matrix. The decision on the sort of composite that will be selected for other tests depends on the results of *in vitro* and *in vivo* tests, which are now in progress.

Acknowledgement

This study was supported by the Ministry of Industry and Trade of the Czech Republic Program – TANDEM, No. FT-TA3/131, Research on problems of spinal diseases focused on degenerative and post-traumatic states of the spine, applying findings in tissue engineering, vertebra biomechanics, osseointegration of artificial replacements and revising their failures, and by the Ministry of Education project Transdisciplinary Research in Biomedical Engineering II., No. MSM 6840770012.

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