COMPARISON OF BIOMECHANICAL PROPERTIES OF CARBON-CARBON AND GLASS COMPOSITES

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Introduction

Composite materials based on various precursors have been tested for several years as biomaterials in medicine, esp. in bone surgery. New technologies allow to produce composites with required mechanical properties close to those of human bones, of a suitable shape and size of the pores, facilitating or preventing the ingrowth of the bone cells, and with excellent biological properties. They are non-toxic, bio-neutral as well as bio-active and their advantages include the fact that they do not form shadows during the X-ray and CT screenings or magnetic resonance [1].

Carbon-carbon (C/C) composite materials have been tested for a number of years as connecting elements and replacements of the bones in orthopaedics [2, 3, 4, 5, 6]. Due to their composition, i.e., of nearly pure carbon [3], they have a very good biotolerance. However, a certain C/C composite disadvantage consists in releasing carbon particles from its brittle matrix, especially when loaded [7, 8], and in a complex preparation [2] resulting in a high price.

In this study, we deal with the development of a C/C composite in a form of intervertebral cages to be applicable for the treatment of the lumbar spine disorders, without releasing carbon particles. At the same time, we compare the preparation and biomechanical properties of the above mentioned C/C composite with a newly developed *composite based on glass fibers* considered as replacements and connections of long bones.

Experimental

Intervertebral Cages Based on C/C Composites

Intervertebral cages, currently used in the spine surgery, are based mostly on titanium alloys (e.g., alloy of Ti, Al, V), while those based on polymers (e.g., PEEK), being neither so rigid, nor showing shadows in the X-ray or CT screenings and magnetic resonance, in comparison to metal cages , have appeared on the market in the recent years. Current operation techniques use bone grafts to achieve osteosynthesis of two neighboring vertebral bodies which results in weakening of patient's organism during a consecutive bone tissue auto-sampling or in the risk of infection and immunity reaction while using bone tissues from a donor. Application of bio-neutral carbon composites, showing a very good biotolerance and integration into the tissues and bones, offers the advantages of plastic materials mentioned above and no bone grafts will be needed.

Materials and Preparation

The basic precursors for the preparation of the C/C composites were:

- carbon fabric (Nr. 46281, type plain cloth), produced by HEXCEL, France, on the basis of the fiber TORAYCA T 800
- phenolformaldehyde resin UMAFORM LE, produced by SYNPO, Ltd., Czech Republic

Carbon composite samples were prepared as coiled reinforcements combined with parallel laminae in the center of the sample, where the first sample had 2 and the second one 4 parallel laminae in the center (see Figure 1). Curing of the sample was performed in a mould of silicon rubber, in a metal frame, in an autoclave (air pressure of 0.6 MPa, temperature of 125 °C, dwell of 90 minutes). This was followed by slow carbonization (heating rate in the slowest period 8°C/hour, maximum temperature 1000 °C, dwell 60 minutes) and by double impregnation. The two series of samples were finally graphitized and some samples were also *coated* with PyC (pyrolytic carbon) and with p(HEMA) (poly [2-hydroxyethylmethacrylate])+ collagen. The following values were obtained:

- with graphitized samples -open porosity 16.5 %,apparent density 1.43 g/cm^3
- with PyC-coated samples -open porosity 9.5 %,apparent density 1.53 g/cm^3

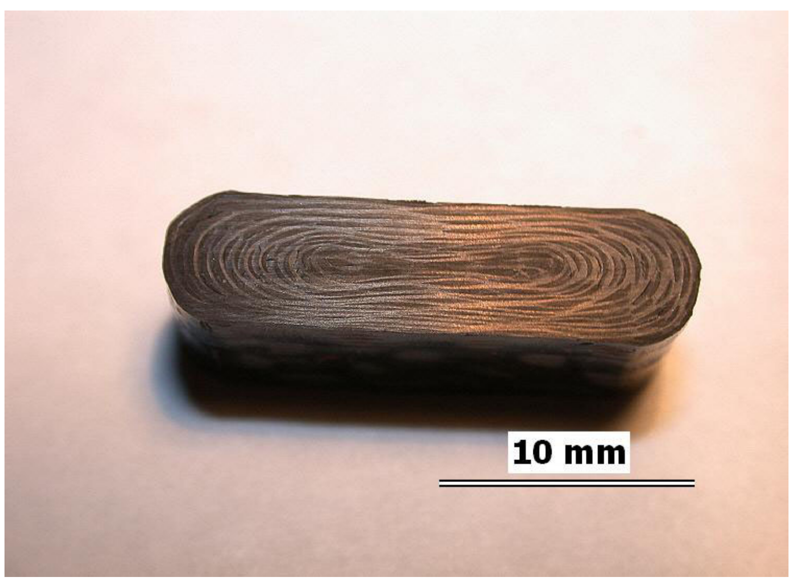


Figure 1. C/C composite core of intervertebral cage

Mechanical testing

Analysis of the mechanical properties of the C/C composite materials consisting in testing several types of samples, which were produced by various technologies to compare their mechanical and biotolerance properties, aimed at the selection of the most suitable composite final treatment. To obtain required mechanical properties of the composite to be used for proposed FEM simulations, concerning the elastic behavior of orthotropic composite materials, two shapes of the samples were designed (see Figure 2). Considerable afford was applied for the samples to comply with the CSN and IS0 standards, respectively, prescribed for the mechanical testing of composite materials. However, in order to preserve homogeneity of the composite structure designed for this specific purpose, the two series of samples had to be produced with atypical dimensions, Figure 2.

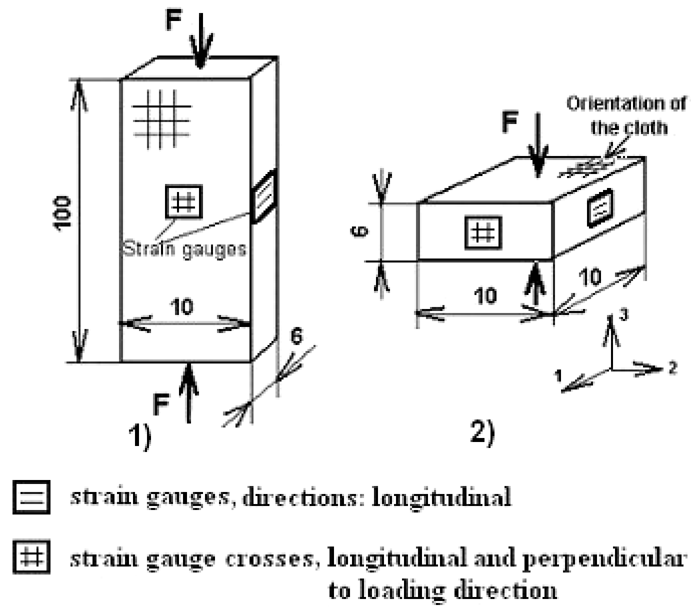


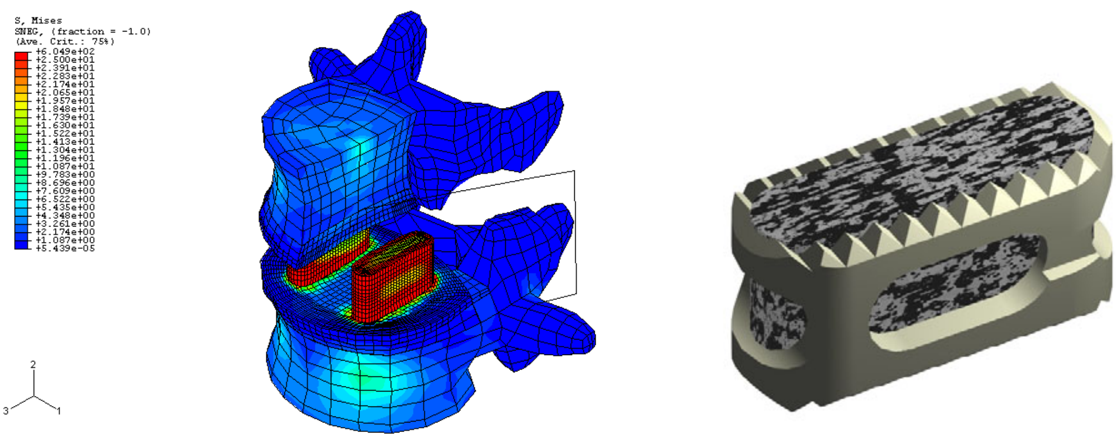
Figure 2. Samples for the mechanical testing of the C/C intervertebral cage core

The tests were performed according to the ASTM test regulations for the testing of composite materials using the MTS 858.2 Mini Bionix testing system.

Finite Element Method Analysis

The aim of the computational part of the project were FEM analyses of the stress state in the composite structures with regard to the interaction of the vertebral bodies with the bone tissue during the spondylodesis fusion of the lumbar segment. The implant with the shape of a coiled closed profile was considered. By means of FEM simulations, an optimum composition of the tissue layers in the whole-composite closed profile was searched for from the point of view of the safety of the implant design. The models gave a survey how a change in the orientation of individual laminae would effect the tissue regarding the stress distribution in the implant. Unfortunately, the production of a hollow closed profile of C/C composite had been proved unrealistic and such a design of a whole-composite implant was rejected. The calculated mechanical properties of the C/C composite which were unsuitable for the application as self-supporting interbody cages led the research team to a compromise variant of the design of the intervertebral implant (see Figure 3a).

The compromise variant of the construction is based on the strength stabilization of the C/C core by means of a titanium alloy cage (see Figure 3b). From the mechanical point of view the contribution of this variant to the increase of safety of the whole operation technique is not too high but it contributes to the “bone-friendly” behavior of the interbody cage by the fact that after the contact of the vertebrae with the teeth of the titanium cage the load is distributed also to the C/C core so that the contact pressure is reduced. This fact and also the C/C composite stimulation of the intervertebral osteosynthesis (i.e., no bone graft is needed) represent benefits especially from the biological point of view.



a) b)

Figure 3. a) FEM model of L4/L5 motion segment with C/C intervertebral cage; b) Model of interbody cage (C/C core + Ti alloy cage)

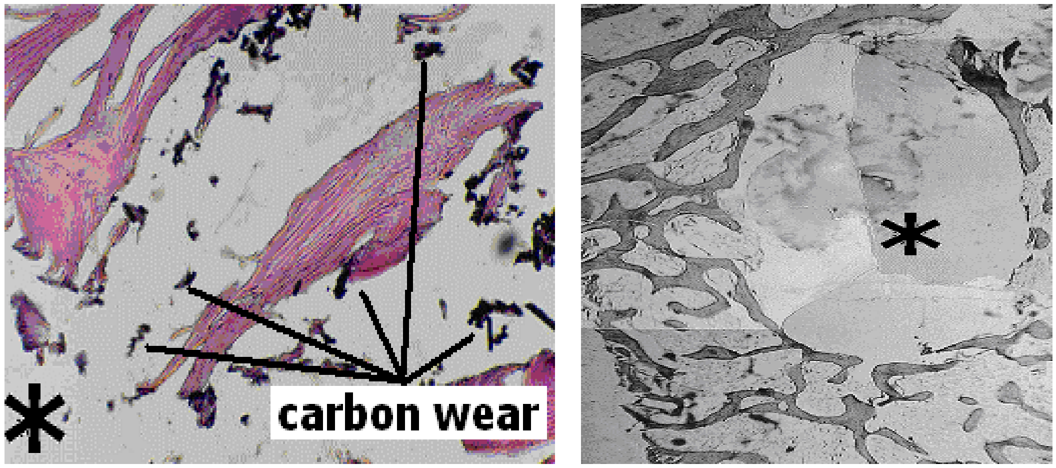
In vitro, In vivo Analysis

The biological properties of the samples of the C/C composite and of the C/C composite coated with p(HEMA) were tested in experiments:

(i) in vitro, biological properties were observed – adherence, proliferation and metabolic activity of cells growing on tested materials and levels of inflammatory cytokines in the cell medium.

(ii) in vivo conditions, the connective tissue surrounding the implants embedded in the bone artificial defect was investigated by the standard histological procedures three months after surgery of pigs. The results are shown in Figure 4 a, b.

Figure 4 unambiguously shows the contribution of the p(HEMA) layer which prevents the release of carbon particles.



a) b)

Figure 4. * the place of the embedding of the C/C implant
a) without p(HEMA); spongiosis tissue (trabecula) with carbon wear,
b) with p(HEMA); implant circled by spongiosis bone, without carbon wear.

Composites Based on Glass Fibers

A relatively complex preparation and expensive components (carbon fabrics) increase significantly price of the C/C composites. Therefore, our research team has aimed simultaneously at developing an applicable biocompatible composite with relatively inexpensive both preparation and components. Glass fibers, being common reinforcing fibers for polymeric matrix composite mailnly for their low cost, have at the same time a high tensile strength and a high chemical resistance. In our study we have prepared glass-siloxane composites and tested their mechanical and biological properties.

Materials and Preparation

The LUKOSIL 901 (L901) siloxane precursors and LUKOSIL M130 (M130) resins (commercial products of Lucebni zavody Kolín, Czech Republic) were used. The composites were prepared from plain-woven cloth V240 (E-glass, VETROTEX, Litomysl, Czech Republic), and from satin-woven fabric 21055 (R-glass, VETROTEX, Saint Gobain, France). The soaked prepregs were stacked, cured at 250°C, then cut to pieces of the required size (40×8×2mm), and cured / pyrolyzed at 200-350°C in nitrogen. The analysis of the properties of the glass composites tested represented the testing of four types of samples in the production of which various combinations of the basic precursors were used, see Table 2.

1.	**R-glass + M130**
2.	**R-glass + L901**
3.	**E-glass + M130**
4.	**E-glass + L901**

Table 2. Tested samples

Mechanical Testing

Mechanical properties of glass composites were obtained by two methods. Young's modulus $\mathbf{E_{res}}$ and shear modulus in elasticity $\mathbf{G_{res}}$ were measured by using the electrodynamic resonant frequency tester ERUDITE. Young's modulus $\mathbf{E_{4p.b.}}$ and the flexural strength $\mathbf{R_m}$ were determined by a four-point bending arrangement on the material tester INSTRON, see Table. 2.

Materials	V_f	R_m	$E_{res.}$	$E_{4p.b.}$	$G_{res.}$
	[%]	[MPa]	[GPa]	[GPa]	[GPa]
E-glass+M130	51	200.81	25.38	23.70	2.39
R-glass+M130	65	391.76	56.06	55.50	3.17
E-glass+L901	52	195.75	27.70	25.70	2.77

Table 2. Mechanical properties of the glass composites

The R-glass+L901 samples were discarded due to a fairly extensive delamination of individual layers of the composite.

In vitro, In vivo Analysis

Biological properties were observed in tests – adherence, proliferation and metabolic activity of cells growing on the tested materials, and levels of inflammatory cytokines exprimed during the cultivation into the cell medium (see Table 3). The medium of this cultivation experiment was performed for cytokines TNF-α, IL-1β detection using immuno-chemiluminescence method of the analyzer Immulite (DCP, Los Angeles, USA).

Ranking (1.= best, 4.= worst) according to:	**R-glass + M130**	**R-glass + L901**	**E-glass + M130**	**E-glass + L901**
The extracts: the metabolic activity of cells cultured in the medium prepared by using the liquid extracts from composite materials	***1.***	***2.***	***3.***	***4.***
The metabolic activity of the cells adherent to the tested materials	***1.***	***4.***	***3.***	***2.***
Cytokine productions	***1.***	***3.***	***3.***	***2.***

Table 3. Results of in-vitro tests

Results and Discussion

To achieve satisfactory mechanical properties, carbon-carbon composites, serving as biomaterials, require a very complicated and thus expensive preparation, often with disputable results, e.g., when reducing the composite open porosity its mechanical strength increases but, at the same time, a possibility of the tissue ingrowth in the composite decreases during some applications. Our experimental results show that coating the C/C composite by pyrolytic carbon and p(HEMA) prevent one of its disadvantages, i.e., release of carbon particles. Nevertheless, based on our presented results, the replacement bone grafts with the C/C composite cores in metal intervertebral cages has its future. This idea is protected by the Certificate of Utility Model [9].

Composites based on glasses and siloxane resins are promising biomaterials, with a relatively cheap precursor, requiring a simple and thus cheap preparation and showing very good biomechanical properties. Currently, we are verifying a possible bioactivity of these materials.

Conclusions

C/C composite materials can be prepared with excellent biotolerance properties to be used as biomaterials, e.g., as a core in titanium intervertebral cages replacing the bone grafts and reducing contact pressures with the vertebra. However, their mechanical properties do not achieve parameters needed to be used as self-bearing elements in the human body. Besides, their preparation is rather expensive. Composites based on glasses and siloxane resins promise to achieve good biomechanical properties while requiring a significantly lower cost. For facial and dental surgery, the light color of the glass composites is also preferable.

Acknowledgment

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References

[1] Ramakrishna S, Mayer J, Wintermantel E, Leong Kam W. Biomedical applications of polymer-composite materials: a review.Composite Science and Technology 2001; 61: 1189-1224
[2] Fitzer E. The future of carbon-carbon composites. Carbon 1987; 25: 163-190
[3] Savage G. Carbon-Carbon Composites. London: Chapman and Hall, 1992: 356-357
[4] Huettner W, Clara LE. Carbon based materials in medical application. J.L. Figueiredo et al., editor.Carbon Fibers Filaments and Composites. Kluwer Academic Publishers, Netherland, 1990: 337-365
[5] Balik K, Sochor M, Krena J, Cabrnoch B, Glogar P, Vilimek M, Pesakova V. Various reinforcements of the C/C composite bone plates and their influence on mechanical properties. Engineering of Biomaterials 1999; 7,8: 8-10

[6] Krysa J, Balik K, Krena J, Gregor J. Corrosion of carbon-epoxy resin (C/E) and carbon-carbon (C/C) composites. Materials Chemistry and Physics 1998; 57: 156-161
[7] Pesakova V, Klezl Z, Balik K, Adam M. Biomechanical and biological properties of the implant material carbon-carbon composite covered with pyrolytic carbon. Journal of Materials Science: Materials in Medicine 2000;11: 793-798
[8] Buresova M, Balik K, Machovic V, Novotna M, Pesakova V, Hulejova H, Sochor M. Biocompatibility of C-C composites covered with PyC and pHEMA. Engineering of Biomaterials 2002; 20: 3-5
[9] Sochor M, Balik K. Utility Model “Intervertebral Cages” (based on C/C composites), No. of application: 2003-13869, Owner: Czech Technical University