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Ústav struktury a mechaniky hornin AV ČR
Oddělení uhlíku – současný výzkum

Výzkum je zaměřen na procesy přípravy, hledání aplikací, a především na studium vztahů mezi strukturou a vlastnostmi moderních uhlíkových materiálů: vláknových kompozitů uhlík-uhlík a uhlíkových mikroporézních membrán.

K přípravě kompozitů uhlík-uhlík jsou používána různá uhlíková vlákna či tkaniny. Vliv jejich parametrů (modulů pružnosti a pevnosti) na mechanické vlastnosti kompozitů je studován metodou rezonančních frekvencí. Biokompatibilita vyvíjených materiálů a jejich možné využití jako implantátů je zkoumáno ve spolupráci s externími pracovišti.

Pro kompozity s maticí odvozenou z termosetových pryskyřic nebo černouhelné smoly jsou hledány optimální procesní parametry karbonizace. Jsou též sledovány vlivy vysokoteplotního zpracování, densifikace pomocí impregnace a infiltrace pyrolytickým uhlíkem z plynné fáze.

Je studována oxidační odolnost kompozitů připravených z jemných keramických vláken a maticí odvozenou z polysiloxanu.

Výzkum mikroporézních uhlíkových membrán pro membránové reaktory sleduje vliv volby polymerních prekurzorů a podmínek přípravy membrán na jejich sorpční a transportní vlastnosti.

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Výzkum kompozitních materiálů pro aplikaci v medicíně

Současný výzkum na našem pracovišti je mimo jiné zaměřen na vývoj kompozitních materiálů na bázi uhlík-uhlík (C/C) a kompozitních materiálů na bázi skelných vláken jako biomateriálů perspektivně využitelných v ortopedii.

Jeho součástí je vývoj kompozitů C/C jako biomateriálů perspektivně využitelných v ortopedii ve formě mezitělových rozpěrek, používaných při ošetření úrazů lumbální páteře. V současné době jsou nejvíce rozšířené mezitělové rozpěrky na bázi titanových slitin (např. slitina Ti Al V). Na trhu se v posledních letech objevují mezitělové rozpěrky na bázi polymerů (např. PEEK), které mají menší tuhost než kovy, nevykazují stíny na rentgenových snímcích či CT projekci a magnetické resonanci, atd. Stávající operační techniky, aplikující kovové rozpěrky, používají kostní štěpy spongiózní kostní drtě k dosažení srůstu dvou obratlových těl, což s sebou přináší oslabení organismu pacienta při dalším operačním zákroku pro odběr kostní tkáně, nebo riziko infekce při využití kostní tkáně od jiného dárce. Aplikace uhlíkových kompozitů, které jsou netoxické, vyznačují se velmi dobrou biokompatibilitou a tkáň i kost jimi velmi dobře prorůstají, slibuje spojení uvedených výhod plastů s výhodou náhrady kostních štěpů.

Další součástí našeho výzkumu je vývoj kompozitních materiálů na bázi skelných vláken. V současné době umožňuje výroba kompozitů připravit kompozitní materiály s vlastnostmi vhodnými pro použití v medicíně. Pokročilá technologie povrchových úprav, znalost biokompatibility a schopnost produkovat materiály s konkrétními vlastnostmi přináší širokou různorodost nových lékařských inovací. Nové kompozitní materiály, jako kompozity na bázi skelných vláken, nalézají vysoké uplatnění v medicíně, a to v podobě náhrad nebo spojovacích elementů. V tomto projektu byla provedena analýza mechanických a povrchových vlastností navržených typů skelných kompozitů k dosažení požadovaných fyzikálních a biokompatibilních vlastností, jmenovitě vhodné mechanické charakteristiky, blíží se vlastnost

CAGES BASED ON THE CARBON-CARBON COMPOSITES

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Abstract

The project aimed at developing a cage (for use in spine treatment) made of a carbon-carbon composite in two modes: 1) a cage composed of a bearing core made of titanium alloy with the surface contacting the bone made of the C/C composites in order to ensure elastic linkage of two vertebral bodies resulting in good bonding with the bone and 2) after testing the first mode, a cage based only on the C/C composites was designed.

It may be stated that the construction of the implant as a combination of a titanium alloy cage with a biologically favourable C/C composites solves the problems with the strength of the core whereas its biological benefit remains preserved. On the basis of mechanical tests and simulation of the strain of the spinal segments it was found that as a self-supporting component the C/C composite is not a material sufficiently suitable for the construction of intervertebral implants from the mechanical point of view.

Keywords: carbon-carbon composites, cage, spine treatment, titanium alloy, mechanical and biological properties

1. INTRODUCTION

The project was aimed at the investigation of carbon-carbon (C/C) based composite materials as biomaterials with prospects of application in orthopaedics in the form of interbody (intervertebral) cages applied in the treatment of injuries of the lumbar spine. At present, interbody cages on the basis of titanium alloys (e.g. the Ti, Al, V alloy) are most widely spread [1]. In the recent years, interbody cages made of synthetic materials (e.g. PEEK) appear on the market; they have a lower rigidity than metals, exhibit no shadows in X-ray pictures, CT projection or magnetic resonance, etc [2]. All operation techniques which apply the interbody cages in question use bone grafts of spongy crushed bone in order to achieve the adhesion of two vertebral bodies. This technique brings about a weakening of the organism of the patient in case of another operative intervention for the collection of bone tissue or the risk of an infection when bone from another donor is used. The application of carbon composites, which are non-toxic, exhibit a good biocompatibility and are well penetrated by both tissue and bone, gives the prospect of the combination of the advantages of the plastics mentioned above with the advantage of substituting the bone grafts. The subject of this project was to verify these assumptions and to attempt to develop an applicable interbody cage on the basis of a C/C composite.

2. MATERIALS AND METHODS

2.1 Materials

The aim of the experiments was to prepare - on the basis of the previous experience - a 2-D C/C composite which would exhibit the optimum mechanical bending strength of at least 200 MPa (that of human bone equals 150-200 MPa) with a bending modulus as low as possible (that of human bone equals 15 - 25 GPa). At the same time a technology should be chosen which would guarantee a release of carbon particles as low as possible especially under mechanical stress. Further, it was necessary to treat this material in order to make it ready for compression tests. The basic precursors for the preparation of the carbon-carbon composites were:

carbon fabric (Nr. 46281, cloth), produced by HEXCEL on the basis of the fibre TORAYCA T 800 (Tab. 1).

number of strands in the warp/welf/cm	mass	weight (g/m ²)	thickness (mm)
3.5 / 3.5	6 kHr	285	0.29

Tab. 1 Properties of carbon fabric used

phenolformaldehyde resin UMAFORM LE, produced by Lučební Závody Kolín (Tab. 2).

Resin content in the solution (%)	58
Viscosity (Pas), 20 °C	75
Density (g/cm ³)	1.090

Tab. 2 Properties of resin UMAFORM LE

2.2 Technology of preparation

A necessary number of laminae of carbon tissues saturated with formaldehyde resin was folded into the pressmould and cured at 150 °C under the pressure of 0,5 MPa. The cured material was cut into the desired shapes (according to the requirements of the mechanical tests) and carbonized.

2.2.1 Carbonization

Carbonization at 1000 °C in nitrogen. Properties after carbonization: open porosity of 28 %, density of 1,27 g/cm³, bending strength of 80 MPa.

2.2.2 Impregnation and subsequent carbonisation

The open porosity was reduced by a 2- or 3-step impregnation again with phenolformaldehyde resin under the following conditions. The carbonised samples underwent in an autoclave an alternation of pressure between vacuum and the pressure of 0,5 MPa at the temperature of 60 °C. The samples treated in this way were cured again (see above) and carbonized. The resulting samples exhibited the open porosity of 13 % and the density of 1,47 g/cm³.

2.2.3 Graphitization

The next step of treatment was the graphitization of the components up to the maximum temperature of 2200°C in argon medium. The graphitization changes the structure of the composites i.e. of the matrix around the fibres. The process results in a further decrease in open porosity to 11-12 % and an increase in density to 1,5 g/cm³. The bending strength rises up to 180 MPa.

2.2.4 Impregnation with pyrolytic carbon (PyC)

The method CVD, i.e. the chemical vapour deposition in a reactor with a revolving bed under atmospheric pressure at the temperature of 850 °C for 36 hours, was applied. The layers of pyrolytic carbon, deposited both on the surface and in the open pores of the composite, reduce again the open porosity to 8-9 % and increase the density to 1,6 g/cm³. Moreover, these layers - especially on the surface - reduce the release of carbon particles. The resulting bending strength is equal to about 240 MPa.

2.2.5 Impregnation with HEMA-C

Poly- (2-hydroxyethyl methacrylate) collagen, HEMA-C was applied in order to reduce the release of carbon particles as well as to verify the effect of this layer on the ingrowth of the bone tissue [19, 20, 21,]. These layers were again treated in an autoclave by alternating vacuum and the pressure of 0,4 MPa at the temperature of 40°C for 60 min. This process was followed by drying the layers in a vacuum drier again at 40°C for 24 hours.

2.2.6 Type of the reinforcement of the composites

The composites destined mainly for compression tests were prepared with a planar reinforcement. The samples destined for self-supporting interbody cages and for C/C cores + Ti-cages were prepared by coiling the carbon tissue around two parallel textile laminae in the centre of the composite (see Fig. 1). This coiled reinforcement eliminated the potential irritation of the neighbouring tissue by the protruding fibres and, besides that, the tissue coating reduced the release of carbon particles especially from the matrix.

2.2.7 Sterilisation of the composites

For the sterilisation of the samples of carbon-carbon composite materials without pHEMA layers, vapour under pressure in an autoclave at the temperature of about 120°C was used. When the pHEMA layer was applied, a sterilisation under milder conditions had to be chosen. The sterilisation with a germicidal tube (UV rays) in the Rheumatological Institute Prague was used; this technique, however, did not guarantee the ideal sterilisation of the whole surface. Finally, for the sterilisation of all samples destined for

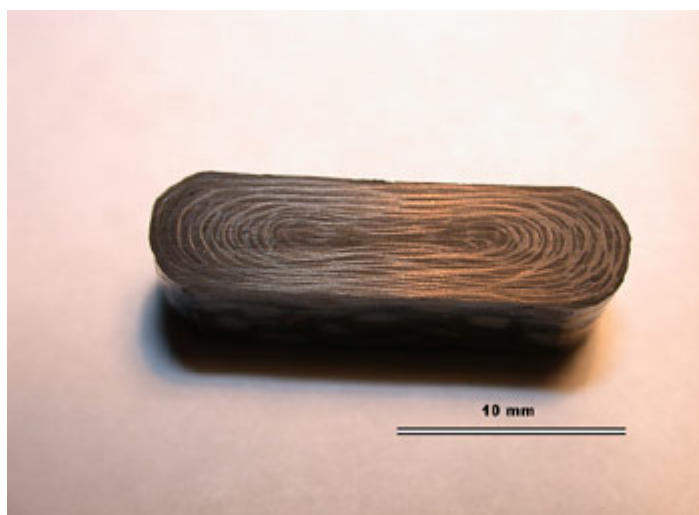


Fig. 1 Composite core of inbody cage implants the system STERRAD (Military Hospital Prague-Střešovice)- which uses the plasma of hydrogen peroxide at 45°C – was applied.

2.3 Technology of the production of final composite cores

The samples were prepared as coiled reinforcements combined with parallel laminae in the centre of the sample, where the first sample had 2 and the second one 4 parallel laminae in the centre. The curing 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 carbonisation (heating rate in the slowest period 8°C/hour, the maximum temperature 1000°C, the dwell 60 minutes) and by double impregnation. Both series of samples were finally graphitized and some samples were coated with PyC. The following values were attained:

2.3 Technology of the production of final composite cores

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- with graphitized samples - porosity 16,5 %, density 1,43 g/cm³,
- with PyC-coated samples - porosity 9,5 %, density 1,53 g/cm³.

2.4 Analysis of the mechanical properties

The analysis of the mechanical properties of the composite tested represented the testing of several types of samples, in the production of which various technologies of the final treatment were used. The aim of the tests performed was the verification of the mechanical properties of the composite on the basis of theoretical hypotheses about the elastic behaviour of orthotropic composite materials and applied standards [3,4,5].

2.4.1 Background of the performed mechanical tests

All mechanical tests were performed in the Laboratory of mechanical tests, the Laboratory of biomechanics of man (Faculty of Mechanical Engineering, CTU Prague). The main device of the laboratory is the testing system MTS 858.2 Mini Bionix. In the domain of the axial force the range of the system is 0-25 kN. The laboratory has obtained the certificate of accreditation according to the international standard ČSN EN ISO / IEC 17025. In this way also all requirements of the international standard ISO 9002 are met.

2.4.2 Description of the mechanical tests performed

The possibilities of the production of samples complying with the standards for mechanical tests of the composite according to ČSN and ISO were limited, especially the production of samples which just according to the tests used as standard ones should have relatively high dimensions. In the course of the production, deformations of their shape I as well as worsening of their structure took place. In order to preserve the homogeneity of the composite structure, samples with untypical dimensions were designed, see Fig. 2. However, the samples used and the way of loading corresponded to the character of the currently applied standards. For the mechanical tests various dimensions of the samples and the way of their loading were chosen, see Tab. 3. The aim of the application of various technologies in the production was to compare the properties of the same composite with a different final treatment and in this way the determine the most suitable way of production with regard to the needed mechanical and biocompatible properties.

A	Carbonized+3×impregnated+graphitized
B	Carbonized+3×impregnated+graphitized+PyC

Tab. 3 Materials of specimens

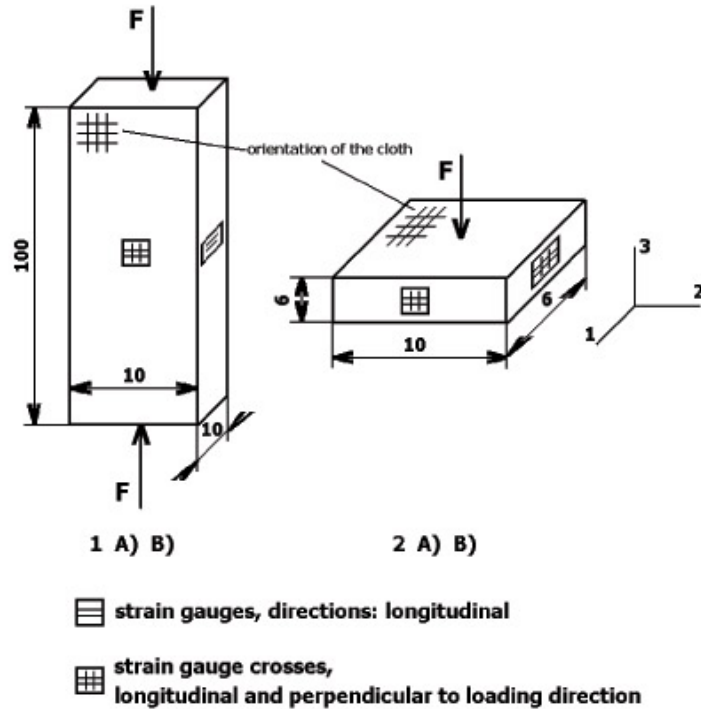


Fig. 2 Specimens for mechanical testing

The dimensions of the samples gave the possibility of sticking tensometers on them. This technique of measurement of the composites required the development of a quite new method. In these measurements, it must be taken into account that measurements using tensometers of very small dimensions (needed with regard to the total small dimensions of the samples) means that properties of the C/C composite are to a certain extent influenced by the local properties of the composite. These properties result already from the inhomogeneous character proper of the sample and for this reason in order to determine the quantitative level of integral deformations the results had to be verified also by comparing the results obtained from the shift of the testing jaws. The test was performed according to the test regulations of ASTM for the testing of composite materials.

The testing machine jaw displacement (stroke) [mm], load [N], angle [deg], torque [Nm] and strain [-] were measured in a configuration where the loading force was either: i) parallel - scheme **1**) or ii) perpendicular - scheme **2**) to the composite laminae (see **Fig. 2**). More complex information about the C/C composite has been obtained: Young's moduli E_1 and E_3 , Poisson's ratio μ_{tp} , $\mu_{31}=\mu_{32}$, and the stress limit values $R_{1,3lim}$ both in tension and compression, provided that $\mu^{ij} = -\varepsilon_i / \varepsilon_j$. To ensure a full contact between the tested samples and the hydraulic jaws, special fixtures were constructed combined with bone cement.

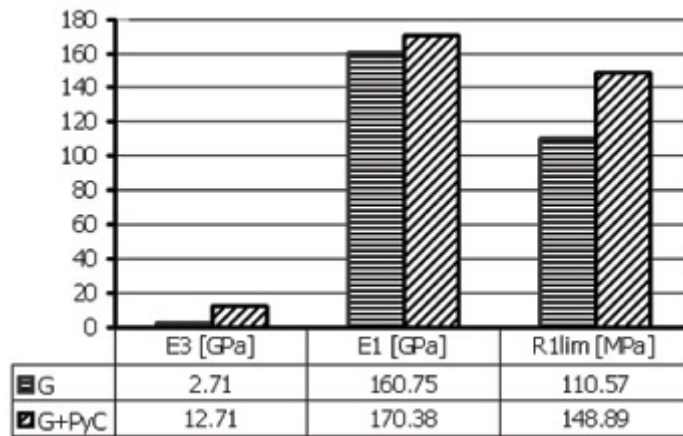


Fig. 3 Comparison of resulting values

The experiments of the first research stages have been designed to provide mechanical characteristics of C/C composite to be applied in FEM models of interbody cages. The problem is complicated due to the fact that the C/C composite examined has been simultaneously developed in order to match two important properties:

- i) suitable mechanical characteristics, to serve as implants, on one hand, and
- ii) a sufficient porosity, to enable a bone ingrowth of good quality on the other hand.

The mechanical characteristic of the C/C composite was determined not only from strain measured with strain gauges, which gives only information from a local part of the sample, but also from the strain measured as integral deformation of the sample (jaw displacement). Other tests have been carried out with a composite core of the same dimensions as in the interbody cage.

Nevertheless, there can be a quite different interpretation of all results: the matrix is damaged from the very loading onset and the curves measured are due to a gradual contact increase of the parallel carbon fabric layers, so that we have got three different data (strain gauges, integral deformations, deformations of the core (see Fig. 3) about the mechanical behaviour of the C/C core (see Fig. 5).

These problems may be solved only after all the experiments and examinations planned have been carried out. The further research stages deal with mechanical testing of interbody cages. These mechanical tests can give us information about the mechanical properties of whole cage consisting of two different materials with different shapes. Mechanical properties of interbody cage must be discussed from the point of view of sufficient stiffness as well as bone-friendly properties of the cage [13, 14, 15, 16, 17].

2.5 Finite elements method analysis

MFE strength analysis of the designed interbody cages on the basis of a C/C composite. The aim of the computational part of the project was the MFE (Method of Finite Elements) analysis of the state of stress 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 [18]. The input parameters needed for the description of the composite were taken with caution from the experimental part of the project. As a constituent of the computational part appears also the MFE mechanics of the motor segment of the lumbar spine L4/L5. The nonlinear analysis of the “physiological” motor segment serves the verification of the formulated boundary conditions and material definitions for the models of stabilised segments. The MFE model of the “physiological” segment was elaborated in principle in two variants differing in the material description of the interbody disc. The first variant is, from the point of view of the material, a linearly orthotropic description of the annulus fibrosus as a composite structure respecting the change in orientation of the collagenous fibres in the different layers of the annulus. The second variant is, from the point of view of orientation, a less perfect nonlinear description of the inner structure of the annulus fibrosus. Although this variant does not respect perfectly the orientation of the binding tissue inside the annulus, nevertheless it gives the possibility of defining the generally known non-linear relation $\sigma - \varepsilon$ of the collagenous fibres of the annulus fibrosus. The results of the MFE models of the motor segment were verified using the data from cinematic measurements of the post mortem dissected motor segments. In order to create both qualitative and quantitative idea about the character of the dynamic loading of the intervertebral articulation as well as to verify these ideas, the analysis of the mobility and loading of the intervertebral articulation was performed on the basis of a special literature search - e.g. [7,8,9,10]. This search did not result in finding any reliable and complex publication which would deal with both the mobility and the mechanical loading of the articulation in question: for this reason to the analysis mainly two literature sources [11, 12] were used. For the determination of mobility it was the article by T. Steffen and coworkers "3-D Lumbar Spinal Kinematic Measurements: Method, Validation and Results". For the determination of the loading of the intervertebral articulation the article by G. Bergmann and coworkers "Load Measurements at Spinal Fixators". For the investigation of the problem of the intervertebral articulation in our Institute a complex procedure has been suggested. The results of this analysis should represent a sufficiently significant input for the numerical calculations of spinal implants in the lumbar segment of the spine as well as a suitable inspiration for the formulation of a plan of laboratory loading tests with the implants. Both MFE models fulfilled the primary aim to verify the assigned boundary conditions. The load and the positioning of the segment was transferred to the models of the fusions.

In the course of the work on the project a number of computational models of the implants and of the fusions with these implants was established. As an introduction to the whole problem the calculations of the titanium PLIF implant produced by the firm MEDIN, which cooperates in the development of C/C implants, were used. The first results showed the reserves in the construction of implants and pointed at their drawbacks. Valuable information obtained from the first models helped us subsequently with the design of the construction of new implants on the basis of a C/C composite.

As the first application of the C/C composite its usage as the core of the commonly used titanium implant PLIF produced by the firm MEDIM was supposed. The mechanical importance of the C/C core in the implant is minimal, as it has been confirmed also by the results of the MFE model of the stabilised segment. The model was established for purely presentation reasons and it has practically no importance for the mechanical evaluation of the fusion of the segment. As a further application of the composite the production of an implant with the shape of a coiled closed profile was considered. By means of calculations the optimum composition of the tissue layers in the whole-composite closed profile was searched for in order to find an optimum from the point of view of the safety of the construction of the implant. The models gave a survey of the possibilities of the turning of the orientation of the individual laminae and of the effect of a change in the orientation of the tissue on the stress distribution in the implant. Unfortunately, the production of a hollow closed profile of C/C composite has proved to be unrealistic and such a construction of a whole-composite implant was rejected.

The limited possibilities of the technology of the production of the C/C composite directed the development of the implant to the formation of a massive composite core. The core was produced as a full-coiled profile of oval cross-section.

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 construction of the intervertebral implant. The compromise variant of the construction is based on the strength stabilisation of the C/C core by means of a titanium cage. It is in principle a comeback of the already previously abandoned concept of a combined Ti-CC implant where the supporting function of the implant is taken over by the titanium cage (see Fig. 4). From the mechanical point of view the contribution of this variant to the safety increase of the whole operation technique is not too high but it contributes to the "bone-friendly" behaviour 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. It represents a benefit especially from the biological point of view.



Fig.4 Model of interbody cage

3. RESULTS AND DISCUSSION

3.1 Results of mechanical testing

The improvement of the mechanical properties of the composite by the coating with pyrolytic carbon is evident especially in the direction perpendicular to its laminae. The compression elasticity modulus is increased up to four times. However, the mechanical properties of the composite in course of the loading in this direction are unsuitable for the application in question. Better mechanical properties are exhibited by the composite when it is loaded in the direction parallel with the laminae. In this direction e.g. the compression elasticity modulus after the application of PyC was increased by only about 10 %. However, the benefit of the PyC coating consists in a noticeable prevention of the release of carbon particles. The tested samples were subjected to structural analyses (using the polished surface microscope scanning). After evaluating the scanning results some structural changes of the sample without PyC were found, different from those of the sample covered with PyC, which confirmed a better behaviour of the C/C composite with PyC and its better ability to be used in the developed interbody cages. Lower porosity of samples covered with PyC (i.e. a negative feature for its integration in the bone) can be improved by covering the C-C composite with HEMA-C which stimulates the bone ingrowths into the composite.

The relatively high values of the Young's moduli obtained by tensometric measurement are reduced when the relative deformations obtained from the shift of the jaws of the loading machine are considered (see Fig. 4). From the measurements performed directly on the composite coiled cores the decrease of Young's modulus down to 2,9 GPa with graphitized and to 3,8 GPa with PyC-coated samples is observed. When the samples are loaded in the direction parallel with the laminae, the upper layers of the composite in the vicinity of the contact planes between the samples and the jaws open. This effect influences probably the decrease in the modulus. The parting of the upper layers of the composite has a favourable influence on the increase in porosity and thus also on the enhancement of the ingrowth of the bone tissue into the composite.

An example of assesment of Young's moduli (Batch 1)

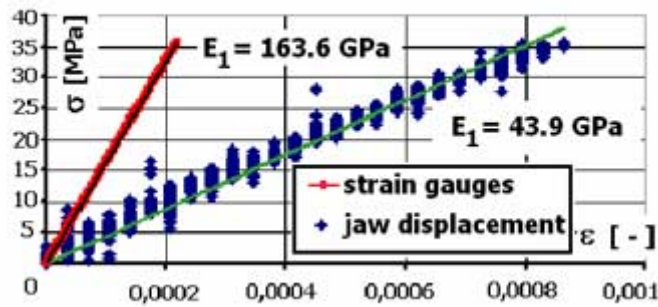


Fig. 5 An example of assessment of Young's moduli (Batch 1 A) Mechanical compression tests of interbody cages based on C-C composite materials, performed by various technologies, served for selecting an optimum procedure yielding suitable mechanical characteristics for the use in human lumbar spine injuries which preliminary resulted in the preference of C-C composite infiltrated and covered with PyC. To obtain statistical informative values [6], a sufficient number of samples to be mechanically tested has been prepared.

3.2 Tests of the biocompatibility of the material of the interbody cages
The biological properties of the materials of the C/C composite (C/C) and of the C/C composite coated with HEMA-C were tested in experiments:

- 1) in vitro (cell cultures),
- 2) in vivo (pigs).

3.2.1 Results of the tests in vitro

By coating the material with a layer of HEMA-C the metabolic activity of the fibroblasts increases several times and the content in both inflammatory cytokines in the medium is reduced.

3.2.2 Results of the first tests in vivo

In contrast with the pure implant of C/C composites, the bone tissue in the vicinity of the carbon implant coated with HEMA-C contains only a very low number of pieces of crushed bone, practically no carbon abrasive wear and the bone stroma has the tendency to surround the implant. In the vicinity of both types of carbon implants haematogenous bone marrow appears, which has not been observed in the vicinity of the titanium implant. It seems that for potential use of the C/C material a suitable porosity of the implant seems to be critical which gives the possibility of the penetration of the bone tissue into its interior.

3.2.3 Compatibility

From the point of view of the integration into the tissue, titanium seems to be a worse material than the carbon composite and distinctly worse than the carbon composite coated with HEMA-C. The testing of the biological properties of a variously treated C/C composite was in the year 2002 performed "in vivo" with laboratory rats and pigs. Both investigations confirm our preceding result from the first approximative experiments "in vitro" and "in vivo" in the year 2001. The order of the materials according to the degree of biocompatibility is as follows:

- 1) C/C composite material coated with HEMA-C
- 2) C/C composite material (exhibits a better integration with the bone than the titanium alloy),
- 3) titanium alloy (exhibits better properties than C/C composite alone only in the subcutis of rats).

The coating of the C/C composite materials with HEMA-C containing a collagen protein causes more extensive changes in the tissue which concern osteoinduction. Collagen as a biodegradable material causes a higher degree of bioactivity of the system which gives the possibility of a more rapid ingrowth of the bone into the implant. A more detailed analysis of the system implant - bone tissue on acrylate sections of implants with the tissue in the vicinity (where the bone proliferation in the immediate vicinity of the surface of the material is investigated) is now being performed continuously and will be presented later.

4. CONCLUSIONS

On the basis of mechanical tests and simulations of the strain of the spinal segments it was found that as a self-supporting component the C/C composite is not a material sufficiently suitable for the construction of intervertebral implants from the mechanical point of view. The direction-dependent mechanical properties represent a significant restriction for its applicability from the point of view of the shaping of the construction. The applied carbon fabric with the tow width of about 2 mm does not give the possibility of producing shape details needed for the connection of the introducing instrument to the implant with the dimensions of about 10 x 10 x 20 mm. It is practically impossible to form in the C/C composite a hole or another structure for the connection with the instrument without endangering the integrity of the composite. The construction of the implant with the titanium cage tries to compensate this drawback.

The titanium cage is designed in such a way that its critical cross-sections correspond to the well-tried implant produced by the firm MEDIN, i.e. so that it should be able to support by itself the strain of the stabilised segment. In this case the composite serves as the core, the supporting function of which is taken over by the titanium cage.

The C/C core remains to be applied as a biologically “friendly” material. Its biocompatibility and capability of the osteointegration (giving the possibility of the absence of autospongioplasty and homospongioplasty in the implantations of the interbody cages) has been since the beginning of the work on this project verified by the research team at the Rheumatological Institute Prague, in cooperation with the Anatomical Institute, Charles University Prague. As stated above, the samples of C/C composite coated with HEMA-C developed for their application into the Ti core meet the requirements of prevention of the intrusion of carbon particles into the organism. The reduction of the total volume of the implant for the bone tissue represents a certain disadvantage of this solution. The titanium cage also represents a protection against the damaging of the fragile composite core during the introduction of the implant. The intervertebral PLIF implants are introduced through a limited space after laminectomy relatively “deep” into the body of the patient by means of the corresponding instrument. The cage gives the possibility of transferring the contact with the instrument from the C/C composite to the resistant titanium and in this way hinders the damage of the fragile core. The function of this “bone-friendly” interbody cage may be in principle described as follows. The loading of the interbody cage by the vertebrae will cause an initial slight intrusion of the titanium tips into the compact and the subsequent contact of the vertebral surface with the C/C insert: in this way the contact area is increased and the contact pressure is substantially reduced. This results in reducing the danger of the penetration of the interbody cage into the bone of the vertebrae. The action of the forces of the vertebral column on the C/C core causes a microscopic opening of the laminae of the composite and the increase in porosity of the fronts of the core created in this way has a favourable effect on the integration of the core into the bone tissue of the vertebrae.

Finally, it may be stated that the construction of the implant as a combination of a titanium cage with a biologically favourable C/C-composite core solves the problems with the strength of the core whereas its biological benefit remains preserved. The suggested construction of the implant is not final and will be further optimised from the point of view of the ratio of the supporting titanium component and of the biologically prospective C/C composite. The aim is to find a suitable ratio of both parts so that both the, mechanical potential of the composite core and the stabilising effect of the titanium cage on the implant may be exploited.

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MECHANICAL PROPERTIES OF COMPOSITES BASED ON GLASS FIBERS AND SILOXANES AS BIOMATERIAL

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Abstract

Nowhere is the ability to tailor the properties of materials having greater impact than in the medical devices market. Advanced coating technology, new knowledge of biocompatibility, and the ability to produce designer materials are creating a broad variety of important new medical innovations. New composite materials such as glass composites for implants applications are moving rapidly out of the laboratory and into the hospital and clinic.

They can potentially be used in orthopedic in the form of substitutive or connective elements. Stress analysis, surface analysis and materials designs were performed to reach desired physical and biomedical properties. These properties are namely suitable mechanical characteristics, to serve as implant materials and a sufficient porosity, to enhance a bone growth.



Keywords: biomaterial, composite, glass, siloxane, mechanical properties

Fig. 1 Glass fibers applications (based on [7])

1. INTRODUCTION

The composite materials proposed as substitutive or connective elements in orthopedics must be biocompatible and their mechanical properties should

approach as much as possible the properties of the human bone (the strength characteristics should be at least the same and the modulus of elasticity should be close to the value characterizing the human bone). However, by the biocompatibility is meant now not only a passive biocompatibility or an inertness that is the facilitation of the growth of the tissue around the implant without any signs of toxicity but especially the bioactivity, i. e. the assurance of a specific biological response on the interface of the material, resulting in the formation of a solid bond between the material and the tissue [1].

In the preceding projects where we have tested carbon – carbon composite materials as implants we have prepared materials with the strength in bending and the elasticity modulus similar to the human bone [2, 3, 4, 5]. These values – especially those of strength in bending – were attained by multiple impregnations from both liquid and gaseous phase. However, this procedure led at the same time to a significant decrease in open porosity with a prevailing pore dimension of about 40 μm , this size of pores giving no possibility of the downgrowth of the hard bone tissue. This is possible only in the case of the pores with a minimum size of 150 μm [6]. The implants with 250 μm pores had the strongest biomechanical strengths [8]. Moreover, the relatively complex preparation and the expensive components (the carbon fibers) increase significantly the price of these materials.

In order to obtain the bioactivity of the composite materials, materials based on bioglasses as reinforcement and thermoplastics (polysulfones, polyetheretherketones) as a matrix were prepared in abroad [9]. The prepared composites with reinforcement chopped or braided, combined with carbon fibers in order to increase their rigidity, exhibited according to their authors a good bioactivity. The bioactivity proper of the bioglasses is given by their chemical composition, especially by their content in the oxides SiO_2 , Al_2O_3 , P_2O_5 , CaO and the alkalis. Hench presents in his papers [10, 11] the preparation of glasses, the chemical composition of which – especially the content in Ca and P – was similar to that of bone. These glasses guaranteed a solid bond between the bone and the implant. However, their preparation and the fiber formation proper are very difficult and exacting is according to our opinion also the preparation of the composites proper [11, 9].

In the literature, we have found the application of siloxanes as a biomaterial. In the publication [13] polydimethylsiloxanes alone, hardened with peroxides, introduced for a longer time (up to 105 days) into laboratory rats are tested and in a further work [14] a composite membrane on the basis of polysiloxanes and cholesterol carbonate was prepared. The result of the experiments is the finding that hardened polysiloxane is biocompatible.

Glass fibers are the most common of all reinforcing fibers for polymeric matrix composites. Their main advantages are low cost, high tensile strength, high chemical resistance and good insulating properties. The use of the composite based on glass and polymer as biomaterial is demonstrated in the Fig. 1.

In our study we have prepared glass-siloxane composites, tested their mechanical properties and studied their surface character.

2. MATERIALS AND METHODS

Properties of various glasses and fabrics follow (see **Tab. 1**).

			E-Glass	R-Glass
Glass composition	SiO ₂	[weight %]	53-57	58-60
	Al ₂ O ₃	[weight %]	12-15	23.5-25.5
	CaO, MgO	[weight %]	22-26	14-17
	B ₂ O ₃	[weight %]	5-8	-
	F ₂	[weight %]	0-0.6	-
	Na ₂ O+K ₂ O	[weight %]	<1	-
	Fe ₂ O ₃	[weight %]	-0.5	-
Mechanical properties	Virgin filament tensile test	[MPa]	3400	4400
	Impregnated strand tensile test(calculated on fiber cross section)	[MPa]	2400	3400
	Tensile modulus	[GPa]	73	86
	Tenacity(sized yarns)	[cN/Tex]	Min. 50	
	Elastic recovery	[%]	100	100
Glass fabric	Weight	[g/m ²]	240	300
	Thickness	[mm]	0.3	0.22

Tab. 1 Properties of glasses and glass fabrics

The siloxane precursors LUKOSIL 901 and LUKOSIL M130 resins (commercial products of Lučební závody Kolín, Czech Republic) were used. The composites were prepared from plain-woven cloth V240 (E-Glass, VETROTEX, Litomyšl, Czech Republic), and from satin-woven fabric 21055 (R-glass, VETROTEX, Saint Gobain, France). Properties of the glasses are presented in **Table 1**.

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 Young's modulus of elasticity in tension and in-plane shear modulus were measured using the electrodynamic resonant frequency tester ERUDITE. The flexural strength was determined with groups of samples processed under identical conditions by a three-point bending arrangement on the material tester MINIMAT.

The character of surface was studied by using the image analysis system LUCIA.

3. RESULTS AND DISCUSSION

Mechanical testing of glass composite samples, dimensions of which enabled to use strain gauges, while applying loading forces in parallel direction to the composite laminae, has been prepared. More complex information about glass composite will be obtained (E, G, Poisson's ratio μ_{tp} , stress limit values $\sigma_{1,3lim}$ both in tension and compression, provided that $\mu_{ij} = -\varepsilon_i / \varepsilon_j$) by three-point bending tests, four-point bending tests, flexural tests and resonance measurements. To ensure a full contact between the tested samples and the hydraulic jaws, special fixtures were manufactured combined with bone cement. First results from mechanical tests are listed below in **Tab. 2**.

	E-Glass +L130	E-Glass +L901	R-Glass +L130	R-Glass +L901
Young's modulus E [GPa]	39.93	20.45	42.92	41.23
Shear modulus G [GPa]	2.39	2.77	3.17	4.60
Flexural strength Rm [MPa]	200.81	195.75	391.76	443.02

Tab. 2 Mechanical properties of glass composites

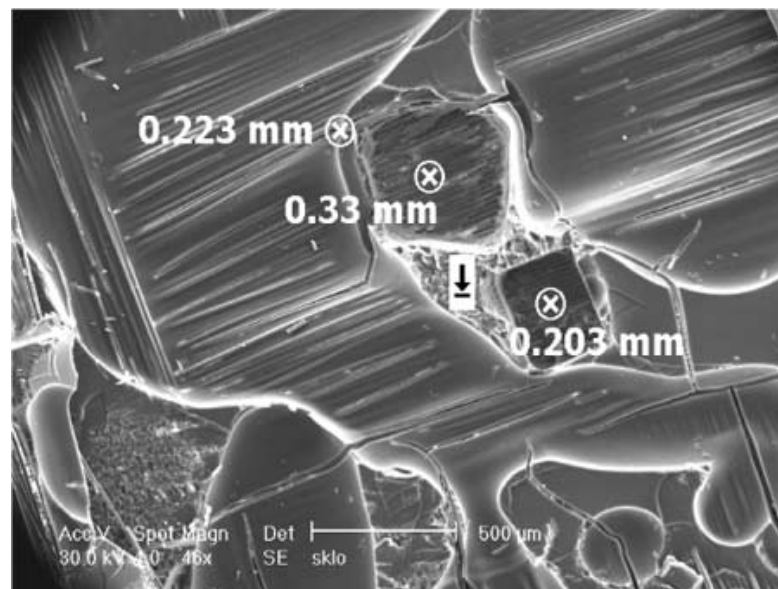
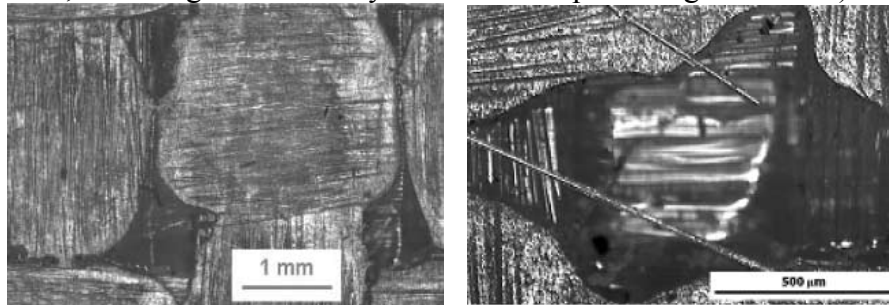
If we compare the mechanical properties of both glass composites and carbon-carbon composites with mechanical properties of human bone-see Table 3, we can see first of all sufficient strength and a relatively low value of modulus of glass composites.

	Composites		Hard tissues [15]		
	R- Glass +L901	C/C(Toray800)	Cortical bone (longitudinal dir.)	Cortical bone (trasverse)	Enamel
Young's modulus E [GPa]	41.2	160.8	17.7	12.8	84.3
Shear modulus G [GPa]	4.6	-	-	-	-
Flexural strength [MPa]	443.0	110.6	-	-	-
Tensile strength [MPa]	-	-	133.0	52.0	10.0

Tab. 3 Comparison of mechanical properties of various materials
The Figures 2, 3 and 4 demonstrate the sufficient pore dimensions. This size of pores over, above cited, 250 μm is giving good presumption of the downgrowth of the hard bone tissue.

4. CONCLUSIONS

Not only a composite material exhibiting high strength values has been looking-for. Based on a complex analysis, the glass composite exhibits a compromise between required both mechanical properties (a relatively sufficient strength value and a low modulus of elasticity, comparable with that of human bone, and biological properties (a sufficient porosity), which would be favorable for tissue and bone in growth, has been developed. Next step of our project will be also biotolerance testing. The biotolerance testing of our glass composites have two parts, tests in-vitro and tests in-vivo (implantation into rats) namely cytokine level observation (observation in the extract of newly formed tissue surrounding the implant the inflammatory cytokines interleukin-I (IL-1 β) and the tumor-necrosis factor (TNF- α)) and histological observation (standard histological examination (painted with haematoxylin-eosin), creation and a quality of capsular connective tissue, including inflammatory cells in the implant neighbourhood).



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Další práce týkající se současného výzkumu Oddělení uhlíku jsou shrnuty v příloze.

Oxidačně odolné kompozity s keramickou maticí

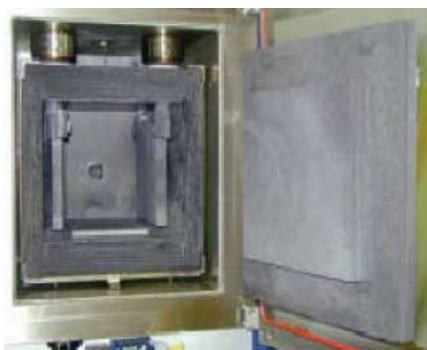
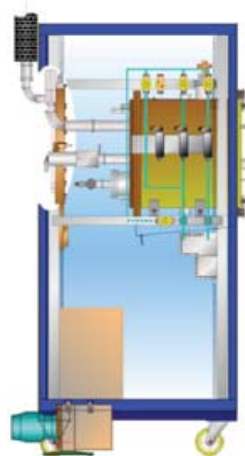
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Vlastnosti některých silikonových pryskyřic za různých teplot

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High Temperature Chamber Furnace Type HTK 8 GR/25-16 man



Pracovní prostor:

šířka 150 mm

výška 150 mm

hloubka 200 mm

Maximální pracovní teplota:

2500 °C

Médium: Argon

Výrobce: GERO GmbH

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info@gero-gmbh.com

Ve dnech 11.-12. října proběhl na našem ústavu Česko-polský workshop zaměřený na testování kompozitních materiálů. Byly předneseny následující příspěvky.



Mechanical properties of polysulfone/short carbon fibres composites
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Polysulfone-based composites with carbon fibres play significant role and are in the field of interest of many researchers. The main advantage for investigation of such composites is ability to modify their microstructure and resulting mechanical properties. Polysulfone is one of well-known polymers in medical applications. This polymer belongs to thermoplastics and its mechanical properties can be shaped with various carbon fibres and different ways of preparation.

The work deals with manufacturing and mechanical properties assessment of polysulfone-based composites with short carbon fibres as a reinforcement. Two kinds of multidirectional polysulfone composites made of short high strength fibers have been manufactured. To prepare the multidirectional prepreg tape, the fibers have been mixed with polymer solution and cast into Petri's dishes. One part of such prepared composites has been stacked and molded in a metal die. The specimens were heated up to 240°C. Tensile tests to determine mechanical properties of two types of polysulfone-based carbon composites (tensile strength, Young's modulus) have been made.

Effect of interface of polysiloxane - based composites reinforced with carbon fibres on their mechanical and thermal properties

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Composites based on polysiloxane resins and carbon fibres as reinforcing elements have been investigated. Wet – winding technique was employed to prepare the samples. Mechanical properties of the unidirectional composite samples after each stage of thermal treatment (curing, HT at 1000°C, HT at 1700°C) were studied. Relation between the nature of interfacial bonding and resulting mechanical properties of composites after various stages of processing was analysed. To determine mechanical properties of interfacial bonding (ILSS) three-point bending test was applied. It was found that mechanical properties, such as bending strength and Young's modulus, and environmental characteristics depend on the characteristics of the interface. Moreover, oxidation resistance of the composites after HT of organic matrix at 1000°C and 1700°C in relation to ILSS parameter was analysed.

Impact testing of composite materials

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Impact tests of composite materials must be performed by instrumented impact equipment because is necessary to take apart phase of fracture and determine both initiation E_i and propagation E_p energy. Ductility index is then given by relationship:

$$DI = \frac{E_p}{E_i}$$

The total impact energy is given by:

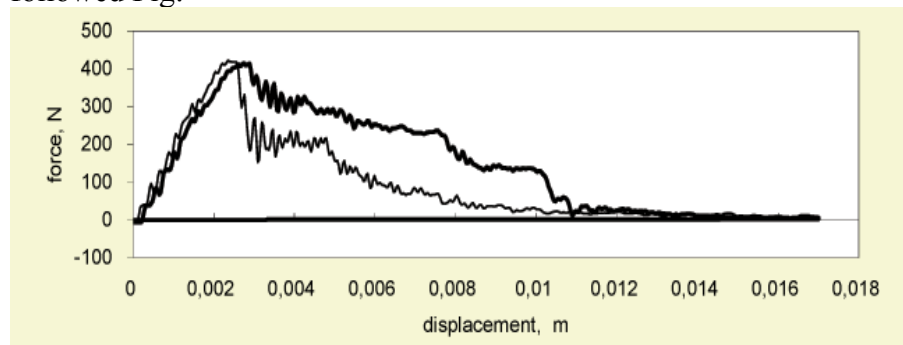
$$E_a = E_i + E_p$$

In our institute we use instrumented pendulum hammer CEAST Resil. Charpy tests are performed accordingly ISO 179 standard. Specimens of laminates are tested in flatwise position, when laminate plane are normal to direction of blow. Changes of voltage on a strain gauge in the striker edge are recorded by A/D converter (sampling time 5 μ s, Czech product) working under Disys software (Czech product). The strain gauge is calibrated statically and the voltage is converted into force. The displacement of pendulum is calculated by double integration of area bellow force-time trace:

$$d(t) = v_0 t - \frac{1}{m} \int_0^t \int_0^t F(t) dt dt$$

here v_0 is the initial pendulum velocity, t is the time and m is the pendulum mass. Dissipated energy are calculated by integration of area below force-displacement trace.

Example of traces force-displacement for composite tetrafunctional epoxy resin–aramid fabric in both original and wet state are shown in followed Fig:



Impact testing can be used for determining the dynamic energy release rate of composite, when delamination is not coming up.

The dynamic energy release rate G_{da} is calculated from the slope of the $E_a - BD\Phi$ plot:

$$G_{da} = \frac{\Delta E_a}{\Delta BD\Phi}$$

where E_a is the total impact energy, B and D is the specimen thickness and width, respectively and Φ is difference of the energy calibration factor given for different a/D ratio by table (a is the notch depth).

Surface Energy Determination of Composites and Reinforcing Fibres

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Technical University of Liberec

Surface energy of solids is a significant parameter for prediction of other useful parameters. A direct surface energy measurement is not possible. The simplest methods use the study of liquid wetting on the solid surface if the liquid surface energy is known. The contact angle is calculated from the Young – Laplace equation describing the equilibrium of a liquid drop placed on a solid surface. The static and the dynamic conditions of wetting must be differed by an experimental measuring.

The three simple methods are used for an usual static contact angle measurement of the solid surface wetted by the liquid:

sessile drop method

solid plate perpendicular immersed into the liquid

incline level method

These three methods are used for flat solid surfaces and solid fibres. The optical scanning of the microscopic image is necessary for the result evaluation.

The changes of liquid body shape when the volume of liquid drop is decreasing, respectively increasing, on the solid surface are used for dynamic contact angle measurement (syringe method). The changes of liquid surface shape when the solid body is immersed into the liquid, respectively the solid body is lifted from the liquid, is observed to measure the contact angle changes by Wilhelmy method. The velocity of liquid wicking into the fibre bundle or powder is used to calculate the contact angle (Washburn method). The evaluated cosinus of the contact angle, from the mentioned measuring methods, is converted to the solid surface energy.

The data converting is possible according to the number of used testing liquids of known surface energies:

one component concept (Neumann, Girifalco – Good)

two component concept (Kaelble, Wu)

three component concept (van Oss – Chaudhury)

The simple experimental tests exist for the solid surface energy determination:

float test for fibres and very small particles

drop test for woven textiles

Zisman estimation of critical surface energy

These mentioned methods were used for testing of carbon, kevlar, boron, nicalon, siliciumcarbide, basalt fibres and etc. The studies of the surface energy of carbon composites with epoxy resin matrix and C/C composites proceed recently.

The project is supported by Research Centre TEXTIL LN 00B090.

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C/C composites with SiC nanofilaments

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Transformation of preceramic polymers into ceramic materials by thermal treatment in a neutral atmosphere is a simple method for preparation of ceramic matrix composites. Preceramic polymers containing high yield of inorganic substances are commonly used for preparation of ceramic powders, coatings and composites. The aim of this work was fabrication porous, fibrous composite consisting of carbon and carbide fibers. Such a dual –fibers composite can be considered as material having improved thermal resistance, temperature - dependent thermal properties and specific friction properties.

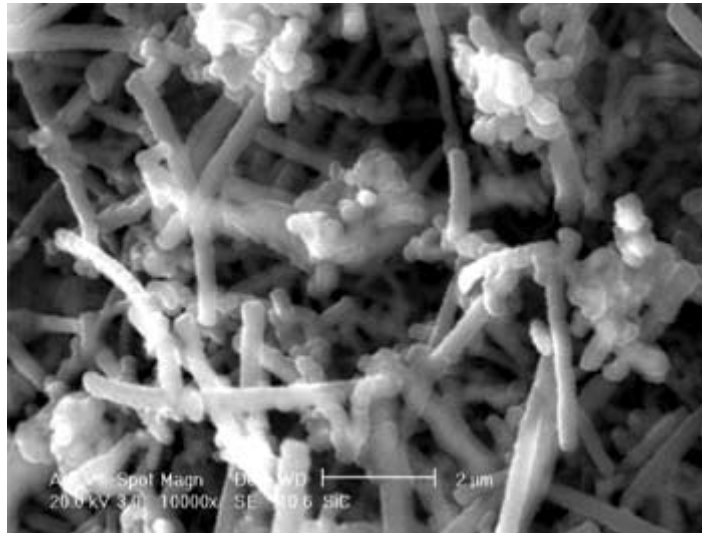


Fig. 1 SEM micrograph of SiC fibers obtained from polysiloxane resin precursor

Oxidation damage of fibre reinforced ceramic matrix composites at elevated temperatures

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Unidirectionally reinforced ceramic matrix composites (CMC) with matrix derived by pyrolysis of methylsiloxane (M130) or methylphenylsiloxane (L901) resin and reinforced with Nicalon NL202 or Nextel720 fibres were investigated with the aim to check and compare the suitability of the resins as matrix precursors. The green composites were processed by pyrolysis in nitrogen to 1000°C during which the polymer matrix was gradually converted to a ceramic glassy material with good resistance to oxidation. The pyrolysed specimens were twice densified by infiltrating with the same resin and re-pyrolysing to 1000°C. In order to assess their mechanical properties and stability when exposed to hot air their dynamic Young's and shear moduli (E and G) were measured prior to and after this exposure. The oxidation was performed during an isothermal exposition to air at 900 or 1200°C for 1 hr. The dynamic moduli were measured at room temperature by means of the resonant beam technique [1].

As expected, the oxidation treatment had nearly no influence on the fibres, thus the E moduli remained almost unchanged. On the other hand, the shear moduli suffered from large deterioration by oxidation (Figure 1), namely after the first oxidation at 900°C. The composites with polymethylsiloxane (M130) - derived matrix revealed generally lower G than their doubles with polymethylphenylsiloxane (L901) - derived matrix. Less decrease of G took place after the subsequent oxidation step at 1200°C; in fact even an increase was observed in case of the Nicalon + M130 composite (Figure 1). Such behaviour can be plausibly explained to result from competition of two processes conversely influencing the shear modulus: a) local ("surface") deterioration of the matrix by oxidation, and b) further stiffening of the incompletely ceramic matrix by its treatment at temperature not experienced so far.

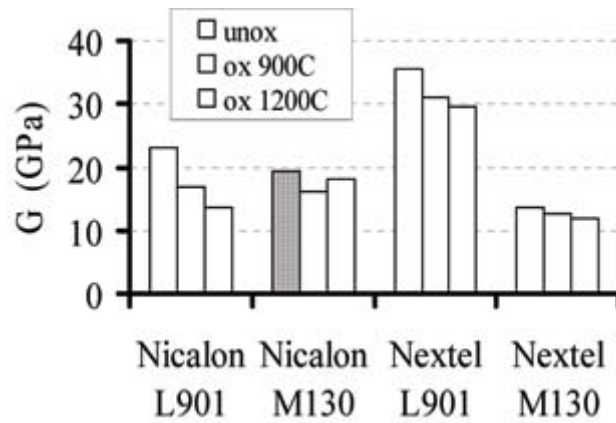


Fig. 1 Mean values of flat-wise shear modulus G of composite specimens after successive oxidation steps in air for 1 hr at 900 and 1200°C.

The performed experiments confirm that the polymethylphenylsiloxane (L901) - derived matrix is more prone to oxidation than the polymethylsiloxane (M130) - derived one.

References:

W. Lins, G. Kaindl, H. Peterlik, K. Kromp, Rev. of Sci. Instr. 1999, 70, 3052

Measurement of elastic constants of composite tubes

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This paper is aimed at development of the new methodology for determination of elastic constants of thin-walled cylindrical orthotropic composite tubes by resonant frequency method.

Measurement of longitudinal and transversal resonant spectrum of composite tubes allows the evaluation of their axial Young's modulus, tangential Young's modulus, Poisson's ratio and shear modulus. In experimental part of the paper, thin-walled wound composite tubes made of carbon fibres and epoxy matrix were measured. Spectrum of mechanical vibration was observed in range from 1 kHz up to 100 kHz. The proposed method was tested for all discussed cases of measurement of elasticity. Results of measurements of elastic constants were compared with calculated values according to laminate theory. Experiments demonstrate the utility in the research and development of composite materials.

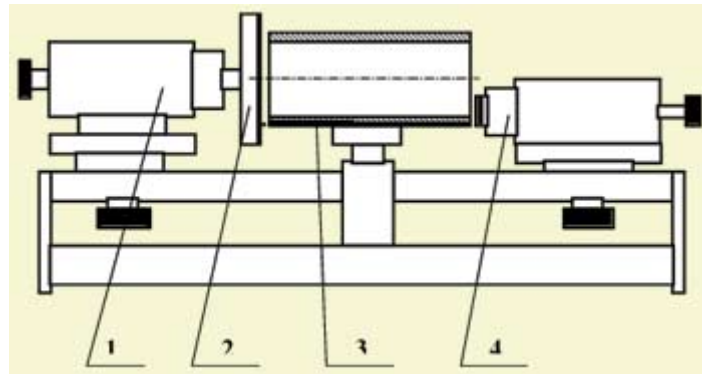


Fig. 1 Measurement of longitudinal resonant frequencies of tubes. (1 body of exciting electrode, 2 plastic exciting electrode, 3 measured tubular sample, 4 receiver)

Mechanical testing of carbon-carbon composite for applications in human spine surgery in the form of intervertebral cages

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Byla zjištěna a vyhodnocena data o IR absorpci a termogravimetrické analýze komerčně dostupných polysiloxanových pryskyřic od 3 výrobců. Vybrané pryskyřice byly použity jako prekurzory matrice při přípravě kompozitních materiálů vyztužených vlákny z R-skla, Nicalonu NL202 a Nextelu 720, které byly následně pyrolyzovány při 750°C (R-sklo) nebo 1000°C (Nextel, Nicalon). Během pyrolýzy se polymerní matrice v různém stupni proměnila na tepelně odolné silikonoxykarbidické sklo. Byl studován vliv procesních parametrů přípravy a řízené oxidace při zvýšené teplotě na mechanické vlastnosti kompozitů (pevnost, modul pružnosti při tahu a ve smyku, charakter lomu při poškození).

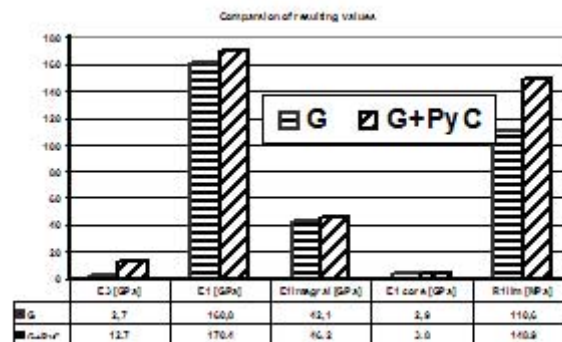


Fig.1 Resulting values

The mechanical characteristics of the C/C composite (see Fig. 1; E – Young's modulus; Rlim – Stress limit value in compression; 1: parallel or 3: perpendicular direction to the composite laminae) was determined not only from strain measured with strain gauges, which gives only information from a local part of the sample, but also from the strain measured as integral deformation (E integral) of the sample (jaw displacement). Other tests have been carried out with a composite core of the same dimensions as in the intervertebral cage (E core).

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který bude pořádat

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AGH – University of Science and Technology

Faculty of Materials Science and Ceramics

Al. Mickiewicza 30

30-059 Krakow, Poland

Další informace budou aktualizovány na www.irsm.cas.cz

22. – 25. May 2004



You are invited to join us at FOA8, the 8th International Conference on Fundamentals of Adsorption to be held on May 23-28, 2004. This conference is organized by the International Adsorption Society. Since the first one in 1983, FOA conferences have been unique in their content and organization. FOAs cover recent advances in all aspects of adsorption from materials to processes. The conference is intended to foster truly international exchange between scientists and engineers of all disciplines, academia and industry, and between novice and expert. Those who have interest in adsorption will find FOA8 well worth attending.

<http://www.csuohio.edu/foa8>

6. – 10. June 2004



The scientific program consists of plenary and keynote lectures and oral presentations in parallel sessions. Posters will be on display and time scheduled for author discussion. In addition, there will be a session devoted to recent research reports.

Preliminary Symposium Topics

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- Conversion of Natural Gas to Energy
- Biological and Biomimetic Conversion Routes
- Upgrading of Coal and Petroleum Using Natural Gas

<http://www.ngcs7.org>

11. – 16. July 2004



Welcome to Carbon2004, the leading international meeting dedicated to the science and technology of carbon materials. Currently there is tremendous excitement in the carbon field, and this meeting serves as an interdisciplinary forum for presentation and discussion of the most recent research results. The meeting covers synthesis, structure, properties, and applications of diverse carbon materials ranging from nanotubes and fullerenes to carbon fiber composites and sorbents; from electrodes, catalysts, and gas storage media to pyrolytic coatings and flame-formed chars and nano-particulate.

The 2004 meeting will be hosted by the American Carbon Society and will be held on the historic campus of Brown University in Providence, Rhode Island (USA). Rhode Island, the smallest of the 50 states by land area and one of the original 13 colonies, is now known for its ocean beaches, historical sites, and the maritime city of Newport with its elegant mansions and large collection of pre-revolutionary buildings. Providence Rhode Island is also close to Boston (100 km) and to the summer playground of Cape Cod (100 km).

<http://www.carbon2004.org>

17. – 19. December 2004



Composite materials have unique advantages over monolithic materials, such as high strength, high stiffness, long fatigue life, low density, and adaptability to the intended function of the structure. Significant achievements have been made in the design/development and application of composites in aerospace applications. Considerable innovative research is still continuing for the development of continuous fiber composites and particulate composites for the use of critical applications. Therefore, this event is aimed at bringing together academicians and researchers in various disciplines to share knowledge and exchange views, for useful industrial applications of composite materials. The conference will discuss various aspects of composite materials by which the participants will be benefited with this recent development and the state of art technology.

<http://www.bhu.ac.in>

1. -5. July 2005



The Carbon Conference 2005, will be held in the old capital city of Gyungju, Korea, July 3-7, 2005. Although many aspects of carbon science have become of age, carbon materials never cease to surprise. Always the quest exists for new materials and an enhanced efficiency for established materials. Carbon 2005 will attempt to promote an interactive atmosphere by facilitating discussion and debate. We will have good discussion leaders and state-of-the art key notes and make maximum use of posters which are essential for interactive debate. The aim of Carbon 2005 is to emphasize the future possibilities of carbon materials and to identify potential "hot topics".
<http://www.carbon2005.com>

September 2005

Carbons for Energy Storage and Environment (exact dates to be announced) Protection CESP '05 to be held in Orléans, France. Further details from Dr. Francois Béguin, CRMD-CNRS, 1b Rue de la Férollerie, F-45071 Orléans Cedex 02, France. E-mail: beguin@cnrs-orleans.fr.
<http://www.gfec.fr>

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