MECHANICAL PROPERTIES OF CARBON-CARBON COMPOSITE FOR IMPLANTS APPLICATIONS

M. Sochor1, K. Balik2, P. Tichy1, R. Sedlacek1, T. Suchy1
Department of Mechanics, Faculty of Mechanical Engineering, Czech
1) Technical University in Prague, Czech Republic
2) Institute of Rock Structure and Mechanics, Academy of Sciences of the Czech Republic, Prague, Czech Republic

Corresponding author e-mail address: balik@irsm.cas.cz

1. INTRODUCTION

New composite materials such as carbon-carbon composites for implants applications are moving rapidly out of the laboratory and into the hospital and clinic. C-C composites to be used in orthopedic in the form of intervertebral cages applied at lumbar spine injuries treatment have been investigated. A complex experimental project has been designed, first results of which being discussed in this paper.
on glass fibers considered as replacements and connections of long bones.

2. MATERIALS AND METHODS

2.1 Materials

Carbon-carbon composite samples are based on plain-woven cloth (Torayca carbon fibers T 800) and phenolic resin. Cured samples are carbonized at the heating rate of 50°C/hr up to 1000°C in nitrogen, see **Tab. I; Batch 1**. The preparation continues with three-step impregnation with phenolic resin and with repeated carbonization, see **Tab. I; Batch 1** and with graphitization at 2200°C in argon atmosphere, see **Tab. I; Batch 2, 3**. The finish step of manufacture is infiltration and covering with pyrolitic carbon in tumbling bed reactor, see **Tab. I; Batch 2, 3**. Our analysis of stress tested three different kinds of this composite with different final production technology applied. Not only a composite material exhibiting high strength values has been looking-for. Based on a complex analysis, the C-C 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 favourable for tissue and bone in growth, has been developed.

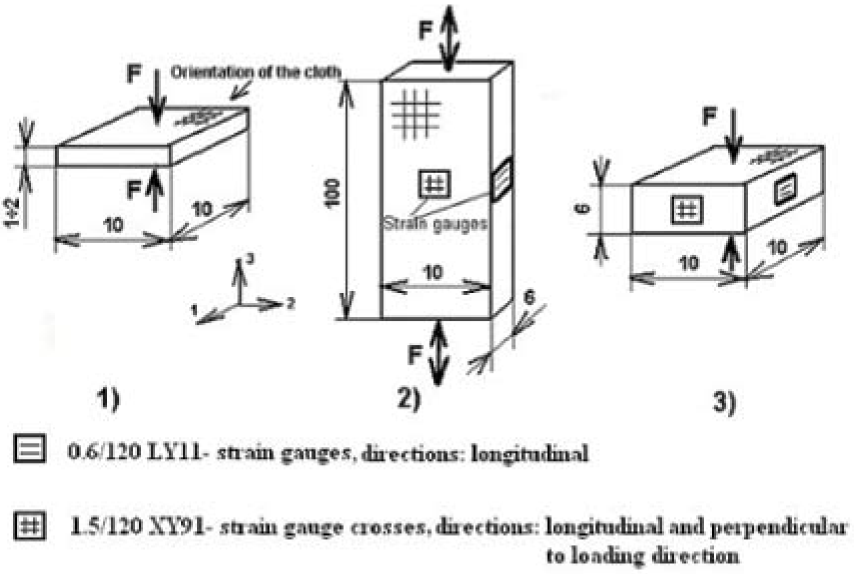


Fig. 1

Table I Batch

	A	Carbonized
1	B	Carbonized+1×impregnated
(see Fig.1)	C	Carbonized+3×impregnated
2	D	Carbonized+3×impregnated+graphited
(see Fig.1)	E	Carbonized+3×impregnated+graphited+PyC
3	F	Carbonized+3×impregnated+graphited
(see Fig.1)	G	Carbonized+3×impregnated+graphited+PyC

The **first research stage** dealt with testing of C/C-composite samples prepared as small specimens (**Fig.1** - scheme 1)). The experiments were carried out using the MTS Mini Bionix testing machine (MTS Systems Corp., USA). The testing machine jaw displacement (stroke) [mm], load [N], angle [deg] and torque [Nm] were measured in a configuration when the loading force was perpendicular to the composite laminae. It wasn‘t possible to use strain gauges, due to the small dimensions of this specimen and the mode of load application.

The **second and third research stages** have dealt with testing of samples, dimensions of which enabled to use strain gauges, **Fig.1**, while applying loading forces: in i) perpendicular - scheme **3)**, and ii) parallel - scheme **2)**, directions, respectively, to the composite laminae. More complex information about carbon-carbon composite has been obtained: E_1 and E_3, respectively, Poisson’s ratio μtp, $\mu_{31}=\mu_{32}$, and stress limit values σ 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 manufactured combined with bone cement. Based on the experiences from the first measuring stage, a **basal loading mode**, chosen **for the second experimental stage** was as follows: a lower-rate loading (1000N, repeated 3x) and, after a 60s delay, the same lower-rate loading (3×). During all the six cycles, there was a maximum compressive loading force of 1 kN applied.

The **basal loading modes** were executed to determine mechanical characteristics of C-C composite samples made by the following technologies: i) **Batch 2, 3F**; ii) **Batch 2, 3G**.

Besides the basal loading mode, a complex loading mode was proposed, consisting in: i) four steps without interruption (2,3,4,5 kN); ii) five steps without interruption (6,7,8,9,10 kN); iii) the sample destruction test.

3. RESULTS AND DISCUSION

The experiments presented in this paper have had a pilot character. They have been designed to provide mechanical characteristics of C-C composite to be applied in FEM models of intervertebral cages. The problem is complicated due to the fact that C-C composite examined has been simultaneously developed aiming to match two important properties: i) suitable mechanical characteristics, to serve as implants, on one hand, and ii) a sufficient porosity, to enable a quality bone ingrowth, on the other hand.

The **first test stage** was applied on a thin samples prepared in the following modes: i) **Batch 1A** ; ii) **Batch 1B**; iii) **Batch 1C**, that were loaded perpendicularly to their carbon fabric layers, which resulted into a rather carbon matrix loading, while the composite reinforcement played a little role. After obtaining load - displacement graphs, resulting stresses and strains were computed (by using specimens dimensions) and corresponding stress-strain graphs were drawn (see **Fig.2**). Based on the linear part of the stress-strain graphs, corresponding moduli of elasticity E_3 in compression for the tested material types were assessed. Due to a lack of superfluous samples, which could have served for a proper microscope examination of polished sample sections in various loading stages, it has not been found out if the matrix has not been damaged at a certain load level. From that reason contemporary interpretation of the results obtained is as follows: The first (concave) knee of the curves can be attributed to an interface contact creation between the sample and the jaw fixations and the second (convex) knee can be explained either as yielding or destruction onset of the matrix.

Notice: Nevertheless, there can be quite different interpretation: the matrix is being damaged from the very loading onset and the curves measured are due to a gradual contact increase of the parallel carbon fabric layers. These dilemmas may be solved only after all the experiments and examinations planned have been carried out.

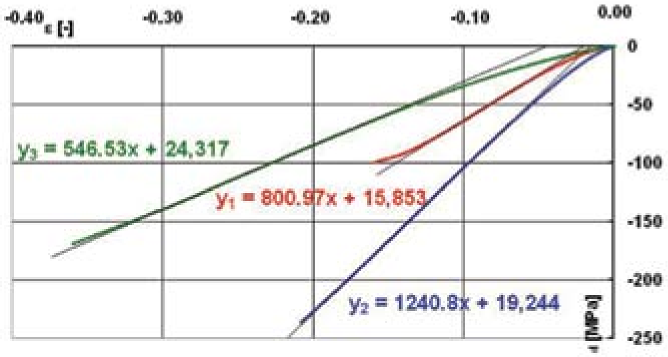


Fig. 2

Table II

Applications	Batch 1A	Batch 1B	Batch 1C
E_3 [Gpa]	0.801	0.55	1.24
$\sigma_{3\text{lim}}$ [Mpa]	-72	-139	-201

Based on the linear part of the stress-strain graphs (**Fig. 2**), corresponding moduli of elasticity E_3 in compression for the tested material types were assessed (see **Tab. II**).

In the second test stage, C-C composite mechanical tests continued with the samples shown in **Fig. 1, 3**. Three samples have been tested, they have been prepared in the following modes: two samples – **Batch 3F** and one sample –**Batch 3F**. It is evident that results obtained from these tests serve only as preliminary information about mechanical behavior of the C-C composite developed. A basal mode (6 cycles, max. load of 1kN), where the first three cycles serve only for stabilization (in accordance with the Standard of the Aircraft Research Institute in Prague), was chosen for all the samples. For the first C+I+C+I+C+I+G sample, it was necessary to repeat all the six cycles with the same rate of max. applied load (1kN) since a too little response of the testing machine was adjusted. The **Batch 3G** sample was also tested by the basal mode (6 cycles, max. load of 1kN), **Fig. 2**.

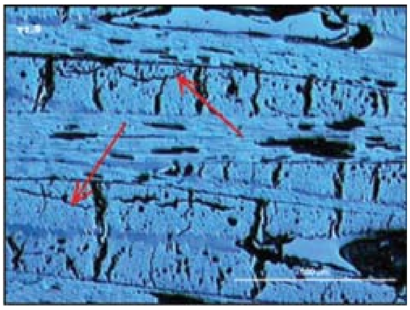


Fig. 2

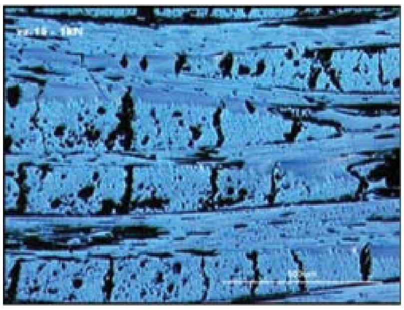


Fig. 3

These two samples were subjected to structural analyses (using the polished surface microscope scanning), **Fig. 3,4**. Meantime, the second **Batch 3F** sample was subjected to the complex lading mode.

After reading the scanning results of the first two samples (tested in 6 cycles, max. loaded 1 kN), some structural changes of the sample without PyC, **Fig. 3**, were found, different from those in the sample covered with PyC, **Fig. 4**, which confirmed a better behavior of the C-C composite with PyC and its better ability to be used in the developed intervertebral 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 the pHEMA (2-hydroxyethyl metacrylate), which stimulates the bone ingrowths into the composite.

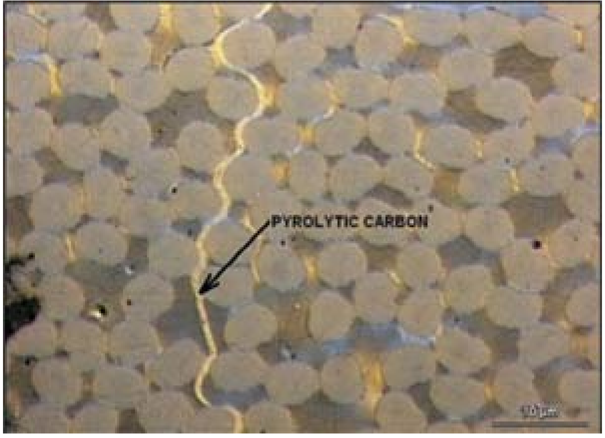


Fig. 5

Mechanical characteristics based on the second stage tests are listed in **Tab.III**. Although they cannot be used as statistically conclusive values (because of the small number of the tested samples) they basically show how different technologies (e.g., a comparison between samples covered or uncovered with PyC) influence their mechanical characteristics (see **Fig. 5**). (Based on the structural changes, the **Batch 3F** sample nonstandard mechanical properties shown in **Tab.III** can be explained). Recently tests continue with testing of samples **Batch 3 D, E** (loading force is parallel to the composite laminae, assessment of $\mathbf{E_1}$)

Table III

Applications	Batch 3G	Batch 3F
E_3 [Gpa]	3.70	12.48
µ [-]	0.13	0.32

4. CONCLUSIONS

Mechanical compression tests of C-C composite samples, made by various technologies, served for choosing an optimum procedure yielding suitable mechanical characteristics for use in human lumbar spine injuries which preliminary resulted in the C-C composite infiltrated and covered with PyC. To obtain statistic informative values a sufficient number of samples to be mechanically tested has been prepared.

References

1. Sochor M., Balik K., Tichy P., Vtipil, J., Sedlacek R.: Experiments Proposed when Developing Cages Based on Carbon-Carbon Composite. In: *Experimentalni Analyza Napeti 2001. Praha : CVUT, Fak. dopravni, 2001, p. 303-306. ISBN 80-86246-09-4.*
2. Sochor M., Balik, K., Tichy P., Spaniel M., Vtipil J.: Project of Cages Based on Carbon - Carbon Composite. In: *Proc. of the Conference. of the American Society of Biomechanics. San Diego, USA, 2001, p. 419-420.*

Acknowledgment
The project is part of the GACR-funded project: Cages (for use in spine treatment) based on the carbon-carbon composite, No. 106/00/1407; and the Ministry of Education project: “Transdisciplinary research in Biomedical Engineering”, No. J04/98:210000012.

PRVNÍ VÝSLEDKY VYSOKOTEPLOTNÍCH MĚŘENÍ NA UNIVERZÁLNÍ TESTOVACÍ STROJ INSPEKT

Nový univerzální testovací stroj Inspekt (instalovaný v laboratoři uhlíkových materiálů ÚSMH AVČR na konci roku 2001) byl na počátku roku 2002 testován v různých měřících konfiguracích. Keramická zatěžovací sada byla doplněna alternativní sadou vyrobenou z žáropevné oceli. Tím se podařilo rozšířit rozsah teplot měření na 20°C – 1 500°C. Pro kontrolu teploty v peci v průběhu měření byl nezávislý měřící systém, který přiřazuje měřenou hodnotu tepoty do datových souborů měření v reálném čase. To je důležité pro plánované experimenty, které mají poskytnout obraz reologického chování materiálů s tvarovou pamětí. Pro tento účel je zamýšlen experiment se strunou z tohoto materiálu upnutou mimo pec do čelistí. Deformace struny v pracovní části pece bude sledována bočním vysokoteplotním extensometrem.
Přístroj Inspekt je již několik měsíců úspěšně využíván pro standardně užívané mechanické testy, především pro měření pevnosti C-C kompozitů v tříbodovém ohybu. Dále byly prováděny experimenty při vysokých teplotách na kompozitních materiálech s keramickými vlákny a sklokeramickou matricí. Při teplotách 1000°C až 1300 °C byl měřen Youngův modul pružnosti v konfiguraci čtyřbodového ohybu. Tento experiment byl prováděn při různých rychlostech deformace. Výsledky tohoto experimentu ukazují, že zvolená rychlost deformace má zřetelný vliv na měřený modul pružnosti.. Je třeba poznamenat, že měřící systém Maytec, detekující deformaci čtyřbodového ohybu ve středním poli nosníku, dává velmi spolehlivé výsledky. To bylo prokázáno srovnávacími měřeními s metodou rezonančních frekvencí a měřením materiálů se známými vlastnostmi při pokojových teplotách. Dalším vysokoteplotním experimentem, při kterém byl využit přístroj Inspekt bylo předběžné měření pevnosti v ohybu již výše zmíněných kompozitů s keramickými vlákny a sklokeramickou matricí. Na základě výsledků těchto měření bude možno plánovat optimální parametry objektivního experimentu, především počet vzorků a geometrii měřených těles.

Informace o semináři

ÚSTAV STRUKTURY A MECHANIKY HORNIN AVČR,
ČESKÁ SPOLEČNOST PRO UHLÍKOVÉ MATERIÁLY
a
UNIVERSITY OF MINING AND METALLURGY,
DEPARTMENT OF SPECIAL CERAMICS, KRAKOW
pořádali
ve dnech 18. – 19. dubna 2002 česko-polský seminář na téma
„Carbon and Carbon Based Composites“

PROGRAM SEMINÁŘE:

- *B. Szaraniec, J. Chlopeka and M. Blazewicz (AGH Krakow)*: Physiological fluid influence on properties of hydroxyapatite modified carbon-carbon composites.
- *T. Suchý, M. Sochor, J. Vtípil, K. Balík and H. Hulejová* (ČVUT, ÚSMH): C-C composites applied in the spine surgery.
- *S. Blazewicz, B. Konieczna, C. Paluszkiewicz and Z. Robak* (AGH Krakow): Pitches-based carbon/carbon composites.
- *P. Polívka* (ÚSMH): The possibility of impregnating C-C composites with pitch using microwave energy.
- *O. Frank* (PřFUK): Fullerenes in natural environment.
- *J. Hlavatý* (ÚFCHJH): Carbonaceous polymers from alkynes and hexatriynides.
- *T. Gumula and S. Blazewicz* (AGH Krakow): Mechanical properties of carbon-fibers based composites with silicon-containing polymer matrix.
- *M. Černý* (ÚSMH): Measurement of elastic constants of orthotropic C-C composites by resonant frequency method.

Příspěvky *S. Blazewicz, B. Konieczna, C. Paluszkiewicz and Z. Robak*: Pitches-based carbon/carbon composites a *T. Gumula and S. Blazewicz* : Mechanical properties of carbon-fibers based composites with silicon-containing polymer matrix byly publikovány v časopise **Acta Montana B 12(126), 2002**.

ÚSTAV STRUKTURY A MECHANIKY HORNIN AVČR,
ČESKÁ SPOLEČNOST PRO UHLÍKOVÉ MATERIÁLY
a
UNIVERSITY OF MINING AND METALLURGY,
DEPARTMENT OF SPECIAL CERAMICS, KRAKOW

připravují česko-polský seminář
"Biotesty kompozitních materiálů“
v měsíci **dubnu 2003**
v posluchárně ÚSMH, V Holešovičkách 41, Praha 8 – Libeň

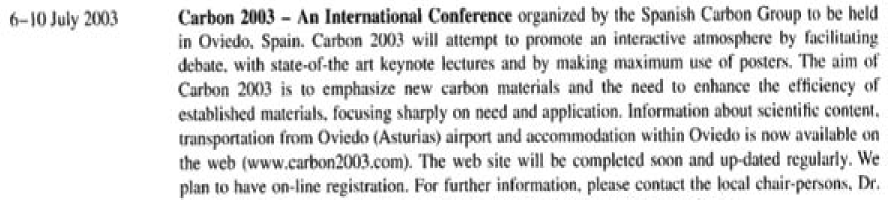
Spojení MHD: tramvaj 10, 24, 25, bus 102, 144, 152, 156, 175, 187
zastávka Vychovatelna

1. oznámení

Těšíme se na Vaši účast.

PERGAMON

CARBON



6–10 July 2003

Carbon 2003 – An International Conference organized by the Spanish Carbon Group to be held in Oviedo, Spain. Carbon 2003 will attempt to promote an interactive atmosphere by facilitating debate, with state-of-the art keynote lectures and by making maximum use of posters. The aim of Carbon 2003 is to emphasize new carbon materials and the need to enhance the efficiency of established materials, focusing sharply on need and application. Information about scientific content, transportation from Oviedo (Asturias) airport and accommodation within Oviedo is now available on the web (www.carbon2003.com). The web site will be completed soon and up-dated regularly. We plan to have on-line registration. For further information, please contact the local chair-persons, Dr. J.J. Pis (jjpis@incar.csic.es) or Dr. Rosa Menéndez, (rosmenen@incar.csic.es)

Informace pro publikování v Časopise Acta Montana

Acta Montana je multidisciplinární časopis s náplní:

- Vybrané aspekty geodynamiky (řada A)
- Paliva, uhlíkové materiály a úpravnictví (řada B)
- Disertace, výsledky grantových projektů vypracovaných v ÚSMH (řada AB)

Základní informace naleznete na internetové adrese: http://www.irsm.cas.cz/
Každá řada časopisu Acta Montana vychází 3 až 4 krát ročně. Cena jednoho výtisku je 100Kč + poštovné.

Bankovní spojení:
Česká národní banka, Praha 1, Na Příkopě 28, 115 03, č. účtu 635-081/0710, Variabilní symbol 468888

Adresa redakce:
J. Netrestová
IRSM AS CR
V Holešovičkách 41
182 09 Prague 8, Czech Republic
E-mail: irsm@irsm.cas.cz
Tel. +420 266 009 318

INSTRUKCE PRO ČESKÉ AUTORY

Časopis Acta Montana přijímá původní příspěvky splňující podmínky výše zmíněných disciplin. Rukopis zašlete e-mailem, resp. 3.5" disketě, ZIP nebo CD-ROM (MS Word libovolné verze), příp. spolu s dalšími soubory (obrázky, tabulky apod.). Dále je třeba dodat vytištěný příspěvek, ve třech vyhotoveních. Redakčbí rada rozhodne o přijetí příspěvku na základě recenze dvou oponentů. První autor obdrží jeden výtisk Acta Montana a 20 reprintů svých článků zdarma.

Článek musí obsahovat:
Název, celá jména autorů s adresami, včetně telefonních čísel, faxu, příp. e-mailu, abstrakt, klíčová slova, vlastní sdělení (vše v angličtině).

Dále může obsahovat:
Tabulky max. velikosti 24x16 cm a min. velikosti písma 9 b., v textu nebo na zvláštních stránkách. Tabulky musí být psány jako skutečné tabulky – ve sloupcích, nikoli jako text v řádcích. Záhlaví všech tabulek musí být na zvláštní stránce. Ilustrace: Přijímají se černobílé fotografie, kresby a mapy v dobré kvalitě (min. 600 dpi). Grafy se přijímají černobílé, velikosti bodů, tloušťka čar a popisy os musí být dostatečně velké kvůli možnému zmenšení. U všech grafů v článku je třeba dodržet stejný styl popisu. Ilustrace mohou být umístěny v textu nebo v samostatných souborech. Přípustné formáty těchto souborů jsou (*.doc, *.xls, *.ptt, *.bmp, *.pcx, *.tif, *.jpg, *.gif). Ilustrace jsou číslovány v pořadí tak, jak je o nich referováno. Popis nesmí být umístěn přímo v obrázku, soupis všech popisů musí být na samostatné stránce. U popisu obrázku je třeba uvést jeho požadovanou velikost v textu. Redakce přijímá rovněž barevné ilustrace (min.

1200 dpi, max. velikost vytištěného obrázku je 24x16 cm). Barevné ilustrace jsou tištěny jednostranně a umístěny na konci článku jako příloha. Náklady na tisk těchto barevných příloh hradí autor. (2000 Kč za stranu). Proměnné, konstanty aj. symboly v matematických rovnicích a odkazy na ně v textu je třeba psát výhradně v editorech rovnic MS Equation Editor nebo MathType. Všechny použité symboly musí být vysvětleny v textu nebo v seznamu symbolů.

Odkazy na literaturu v textu musí být ve formě (autor, rok), např., (Balik, 2001), (Rudajev et al., 2002). Soupis všech odkazů v abecedním pořadí musí být na samotné stránce ve tvaru:
Příjmení, iniciály prvního autora, (příjmení a iniciály dalších autorů, kromě posledního) and (příjmení a iniciály posledního autora): rok, název článku, název časopisu, číslo, stránky od-do

např. Mierzejewski, M., Korzak, F. and Kaczalek, M.: 2002, Geodynamic research of recent movements in the Karkonosze Mts, Acta Montana ser.A., 15(126), 56-78

Příspěvek musí být zaslán v konečné verzi. V autorském náhledu je možno opravovat pouze překlepy, jiné zásahy do textu jsou nepřípustné.

Acta Montana is a multidisciplinary journal concerned with:

- Selected aspects of the geodynamics (series A)
- Fuel, carbon and mineral processing (series B)
- Extended essays, PhD dissertations or results of grant projects elaborated in IRSM (series AB)

Topical information on internet address:
http://www.irsm.cas.cz/
Acta Montana issues periodically 4 times per annum.
Aim: The main purpose of Acta Montana enables a possibility to publish the results of scientific works and contribute to information exchange among scientists, engineers and others professionals.
Issues, price list: Acta Montana appears about three times annually for each series. The price of one issue is 12 EUR plus carriage charges. Back issues can be obtaining on request.

Bank connection:
Czech National Bank, Prague 1, Na Příkopě street 28, 115 03, account no. 635-081/0710 Variable Symbol 468888

Subscription office:
J. Netrestová
IRSM AS CR
V Holešovičkách 41
182 09 Prague 8, Czech Republic
E-mail: irsm@irsm.cas.cz
Tel. +420 266 009 318

INSTRUCTIONS FOR AUTHORS

Submissions:

Acta Montana accepts original papers in English concerning all aspects of mentioned disciplines. Authors should submit three hard copies of their contribution and identical text file in MS Word (any version) and in case of need other files (figures, tables etc.) by e-mail, on 3.5" floppy disc, ZIP or CD-ROM. The Editorial Board on the basis of reviews of at least two referees makes the decision upon their publication. Author first named will receive one volume of Acta Montana and twenty reprints free of charge.

The manuscript must contain:

Title, full names of all authors, their affiliations and addresses including phone number, fax number and e-mail address, abstract, keywords and main body of paper (all in English).

It can be included:

Tables at max. size 24x16 cm and min. font size 9 pt, in text or on separate pages. Tables must be written really as tables (in columns), not as text (in rows). Captions of all tables must be on separate page. Figures: Black and white photographs, drawing or maps in good quality (min. 600 dpi) are acceptable. Charts and diagrams must be in black and white, description of axes must be at sufficient size in due to possibility of reducing. Figures may be placed in text or as separate files. Only permitted formats of these files are (*.doc, *.xls, *.ptt, *.bmp, *.pcx, *.tif, *.jpg, *.gif). Figures are to be referred as Fig. Arabic numeral and should be numbered consecutively, according to their sequence in the text. Caption must no be in placed in the figure area. List of captions of all figures must be on the separate page. Required size of the pictures in the text must be denoted at the list of captions. Color illustrations are tolerable (min. 1200 dpi), but at author's expense 75 EUR per 1 page A4). These pages, (pressed on one side) will be placed on end of the paper as appendix. Variables, constants and other symbols in mathematical functions and also in the text are accepted written exclusively at MS Equation Editor or MS MathType. All used symbols must be explained in text or in the List of symbols.

References quoted in the text must be in form (author, year), e.g., (Balik, 2001), (Rudajev et al., 2002). All references should be listed together at the end of the paper in alphabetic order as:
First name, signatures of surnames, (names of other authors except last) and (First name, signatures of surnames of last author): year, title, journal, number, pages

e.g. Mierzejewski, M., Korzak, F. and Kaczalek, M.: 2002, Geodynamic research of recent movements in the Karkonosze Mts, Acta Montana ser.A., 15(126), 56-78

Paper must be supplied as final version. At press-proof it is possible to correct only typing errors, other changes of text are not acceptable.