IMAGE ANALYSIS OF THE FIBRE VOLUME FRACTION AND THE POROSITY OF CARBON-CARBON COMPOSITE MATERIALS

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INTRODUCTION

The properties of carbon-carbon composite materials (C-C composites) are depending on various factors like type and amount of fibre, pore size and distribution, density and structure of the matrix, the adhesion between fibres and matrix and so on.

The relative quantity of fibres usually expressed as the volume fraction of fibres is a very important parameter of C-C composites, affecting significantly their mechanical properties C-C composites are candidate material for biomedical application and overcome many of the common problems associated with metal implants in the biomedical application. They exhibit a relatively sufficient value of strength and low modulus of elasticity, which is comparable with that of human bones. Carbon-carbon composites with pores about 50μm in diameter can be also favourable for tissue and bone ingrowth.

Measurements of the fibre volume fraction of 1D C-C composites and the porosity of 2D C-C composites using image analysis method LUCIA are described.

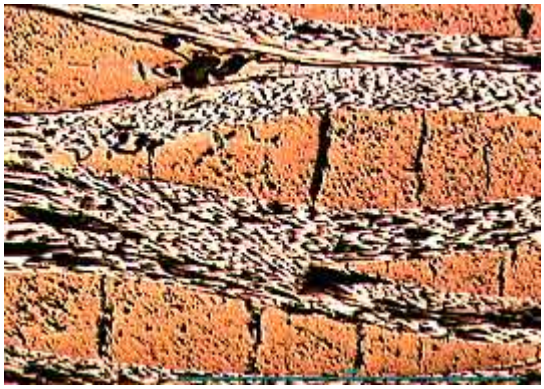


Fig 1. 2D C-C composite cross-section.
The image superposed from 20 image fields using Grab Large Image command of the system Lucia G version 4.60 and the detailed image from the same source.

MATERIALS

Measurements of the fiber volume fraction were carried out on the series of unidirectional (1D) C-C composites reinforced with carbon fibres by the wet winding (prepreg) technology as the matrix precursor the phenolic resin was used. Samples were cured under the pressure (C-P composite), then three times reimpregnated and carbonised in N2 atmosphere (C-C carbonised composite), and then graphitised in Ar atmosphere (C-C graphitised composite).

The preparation of two-directional (2D) samples for the porosity measurement was basically the same; instead of carbon fibres the plain-woven cloth from carbon fibres was used.

For the measurement, polished sections were prepared. Sample was embedded in epoxy resin and the exposed surface first smoothed, and then polished to a scratch- and relief-free finish.

SOFTWARE AND HARDWARE

The fibre volume fraction and porosity were determined by the image analysis method in the system LUCIA G version 4.60, Laboratory Imaging Ltd, Prague, Czech Republic, using the metallurgical microscope Nikon Optiphot 100S, maximal magnification 1000x, and the colour CCD camera Hitachi HV-C20 connected with the frame grabber FlashPoint Intigue Pro. The

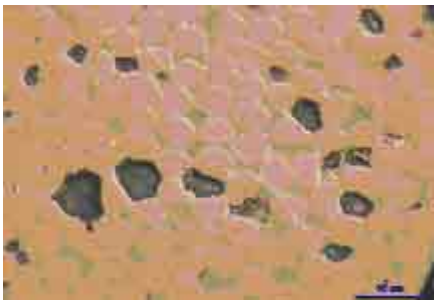
film scanner Nikon COOLSCANII and the ink jet printer EPSON Stylus COLOR and digital thermotransfer printers Mitsubishi CP-D1 and CP-800DW supplement the system.

FIBRE VOLUME FRACTION MEASUREMENT

The optical reflectance of fibres and matrix is not that distinct in C-C composites, recognition of fibres in the embedding matrix in C-C composites, especially in the graphitised sample, is quite difficult. For the maximal differentiation of fibres and matrix the initiation macroprogram was prepared that adjusted the intensity of illumination by gain setting and white balance on camera and the aperture stop of the microscope. For grabbing itself the shading correction was used. The quality of micrographs significantly affects further measurement of fibre volume fraction. Micrographs are shown at Fig 2a, 2b.

For the measurement of fibre volume fraction at magnification 1000x by the image analysis method the main macroprogram was prepared.

The function of the main macro is shown on of Fig 3.



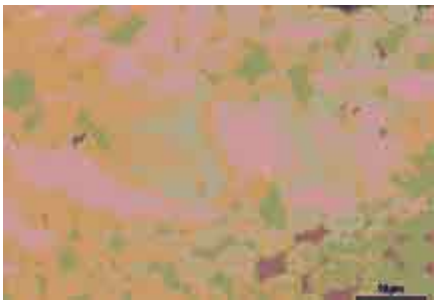
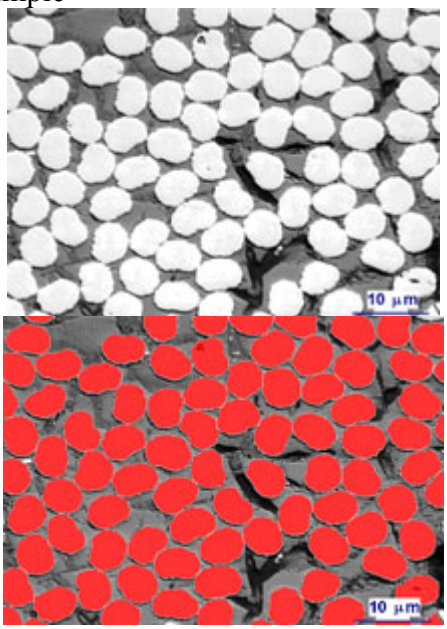


Fig 2. 1D C-C composite a) carbonised sample b) graphitised sample



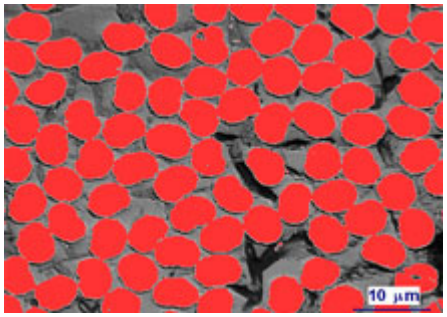


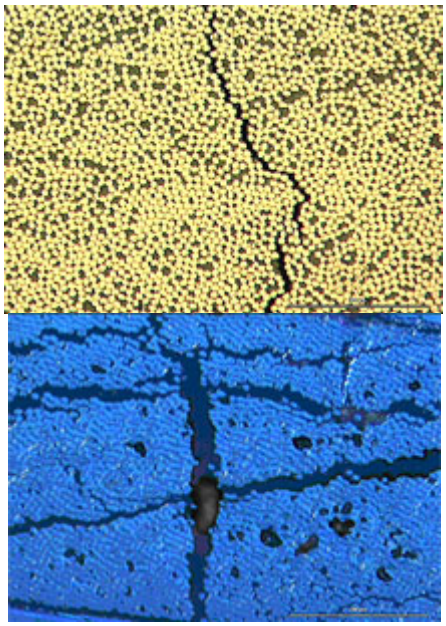
Fig 3. Main macro function
a) final micrograph grabbed after initiation converted to grey levels
b) binary image after the first automatic tresholding
c) final binary image for the fibre volume fraction measurement

Fibre volume fraction was measured on 50 fields at each sample using Area Fraction feature of the Field Measurements. Area fraction value is defined as the ratio of the segmented image area and the Measured Area. It has a strong stereological interpretation: in the case of isotropic uniform random sections it is equal to the volume fraction.

POROSITY MEASUREMENT

Porosity measurement on grabbed image is much easier due to the distinct recognition of voids (pores and cracks) in the solid material (fibres and matrix) of 2D C-C composite.

Contrast images grabbed with the initialisation macroprogram are shown on Fig 4. For each material different initialisation macro was used according to the best voids representation.



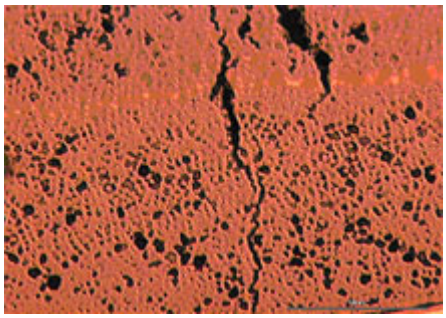


Fig 4. 2D C-C composite cross-sections, magnification 200x
a) “green” C-P composite
b) carbonised C-C composite

c) graphitised C-C composite

The colour images were processed with the main measurement macroprogram. The final image was converted into the grey scale using ExtractComponent command of Lucia Transform menu with the further contrast enhancement using the Contrast command from the same menu. Final grey scale colour image was via Define Treshold command converted into the segmented binary image suitable for the measurement. Object measurement was used, and each displayed void of the composite material was characterised by the Area, MaxFeret, MinFeret, Elongation, Circularity, and Orientation value.
The wide range of voids size had to be measured and was divided into four measurements stages using four magnifications. For each magnification the smallest measurable detail was calculated.
Interval borders and the size of the smallest measurable detail were used as a Restriction from the LUCIA Measure menu.
Porous system of 2D C-C composite is formed from pores and cracks. The pores and cracks are shown on Fig 5. Pores and cracks can be differenced using its shape character. Pores are predominantly elliptical to circular. This factor was used for Circularity and Elongation restriction from LUCIA Measure menu. Circularity in LUCIA system is a derived shape measure, calculated from the area and perimeter. Elongation is determined as a ratio of MaxFeret and MinFeret features.
Circularity = $4*p*Area/Perimeter^2$
Elongation = MaxFeret/MinFeret

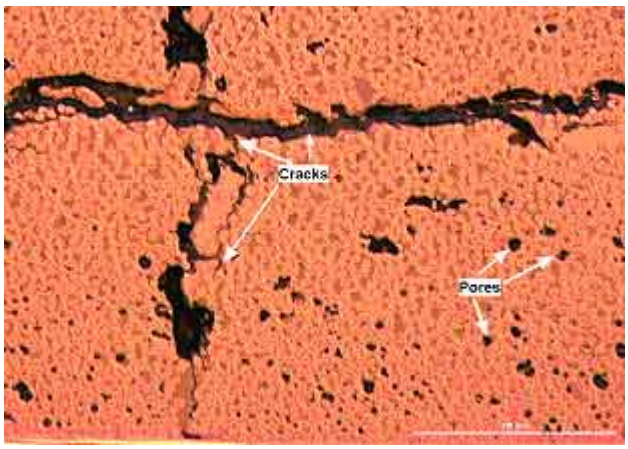


Fig 5. Porous system of 2D C-C composite

It was impossible with the lowest magnification (objective 5x) we have to see the whole cross-section and measure big holes and delamination cracks. The new feature of LUCIA G version 4.60 was used to superpose big picture of cross-section from several image fields. All cross-section of C-C composite is superposed from up to 38 image fields grabbed with objective 5x. The colour image is converted into the grey level image and segmented into binary image used for the measurement.

CONCLUSION

It may be concluded, that the main problem of image analysis method for the measurement of fibre volume fraction of 1D C-C composites, especially of graphitised samples, is to acquire sharp micrograph with the distinct recognition of fibres in the composite matrix. Measurement of coarse pores and cracks is easier due the distinct recognition of voids and the substance of composite. Initial macroprogrammes for grabbing image and main macros for measurements itself make measurements faster, and enable us to process really large amount of images, and to measure the statistically significant amount of data.

RELATED PUBLICATIONS

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Balík, K., Burešová, M., Machovič, V., Novotná, M., Pešáková, V., Sochor, M.: Biocompatibility of C-C composites covered with PyC and pHEMA, Engineering of Biomaterials, No.17 - 19, p. 9, Poland 2001, grant No. 106/00/1407.

Informace z konference

ENGINEERING OF BIOMATERIALS

POLAND 2001

BIOCOMPATIBILITY OF C-C COMPOSITES COVERED WITH PYC AND pHEMA

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The application of carbon-carbon composite materials as a biomaterial is mainly limited by its cost and brittleness of the matrix. The brittleness leads very often to the formation of microparticles in the tissue, which may cause inflammations around implants then. To prevent the releasing of carbon particles, C-C composites have been covered by different layers. In our work we have studied the biocompatibility of C-C composite surface covered with pyrolytic carbon and pHEMA (Poly-Hydroxy-Ethyl- Methyl-Acrylate), synthetic polymeric hydrogel utilized for biomedical applications.

The specimens were reinforced with the carbon plain wave fabric from Torayca T800H fibres with the phenolic resin Umaform LE as a matrix precursor. The specimens were three times impregnated, recarbonized and infiltrated and covered with pyrolytic carbon. Final carbon-carbon samples were impregnated and covered with pHEMA solution in the autoclave.

The presence of pHEMA on the surface and in inner pores of composite was indicated with optical microscope and infrared microspectroscopy. The volume fraction of pHEMA in inner pores of composite was detected from polished cross-sections using image analysis method, 57% of all open coarse pores was penetrated with pHEMA.

Embryonal human lung fibroblasts were cultured on composites. Plastic Petri dishes for tissue culture were taken as a control surface. The metabolic activity of cultured cells, and the level of some cytokines were determined. The cells cultured on C-C carbons coated with pHEMA exhibited several times higher metabolic activity in comparison with the uncoated C-C composite. The cytokines were estimated by immunoreaction in medium after the cell cultivation. The levels of both inflammatory cytokines were higher in comparison with the control surface.

KINETIKA KARBONIZACE PRYSKYŘICE EBOLIT FF

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Skelný uhlík lze připravit karbonizací fenolických a furfuralových pryskyřic. Při nevhodném vedení karbonizace vznikají v produktu defekty, které se projeví zhoršením jeho mechanických vlastností. Tyto defekty souvisí s migrací pyrolyzních plynů uvolňovaných při karbonizaci. Znalost pyrolyzní kinetiky je proto důležitá pro návrh optimálního teplotního režimu karbonizace. Bylo zjištěno, že stupeň karbonizace studované fenolformaldehydové pryskyřice příliš nezávisí na teplotní historii procesu, ale jen na okamžité teplotě. Závislosti stupně karbonizace na teplotě pro různé rychlosti ohřevu se prakticky překrývají. Kinetické rovnice n-tého řádu nepopisují takovýto systém s dostatečnou přesností. Na základě experimentálních dat TGA byl navržen kinetický model, který vystihuje chování fenolických pryskyřic při karbonizaci s dostatečnou přesností pro technologické aplikace (Kolář, Svítilová 2001):

$$\frac{d\alpha}{dt} = A^{*}\theta \exp\left(-\frac{E}{RT}\right)(1-\alpha) \quad 1)$$

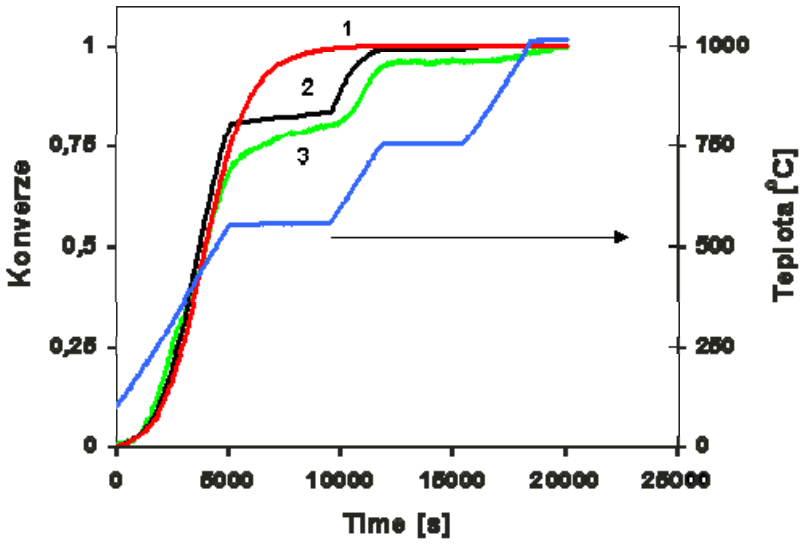
resp. 2)

$$\frac{d\alpha}{dT} = A^{*} \exp\left(-\frac{E}{RT}\right)(1-\alpha)$$

kde t je čas, T abs. teplota, R univerzální plynová konstanta, E aktivační energie, A^{*} předexponenciální faktor a Θ rychlost ohřevu, obecně nekonstantní funkce.

Jde pochopitelně jen o jisté přiblížení, model popisuje chování karbonizačních systémů, které jsou po celou dobu procesu v blízkosti rovnováhy. Platí proto tím přesněji, čím je rychlost ohřevu nižší. Jelikož skutečné rychlosti ohřevu při přípravě skelného uhlíku jsou nižší než námi studované, dá se předpokládat, že uvedený model bude dobře použitelný pro optimalizaci teplotního režimu karbonizace.

Byly provedeny experimenty, které měly za účel zjistit, jak je model schopen predikce chování studovaného polymeru při zahřívání obecnými stupňovitými cykly, kdy je lineární teplotní vzrůst střídán s úseky o konstantní teplotě. Tyto experimentální závislosti pak byly porovnány s funkcemi $\alpha(t)$ vypočtenými integrací odvozené kinetické rovnice. Typická experimentální závislosti $\alpha(t)$ je na obrázku proložena vypočtenou křivkou. Pro srovnání je i zde znázorněn průběh úbytku hmotnosti vypočtený integrací arheniovské rovnice prvního řádu.



Závislost stupně konverze na čase při stupňovitém karbonizačním cyklu. Experimentální body jsou zde proloženy funkcí $\alpha(t)$ vypočtenou integrací rovnice (1) pro stupňovitý teplotní režim:

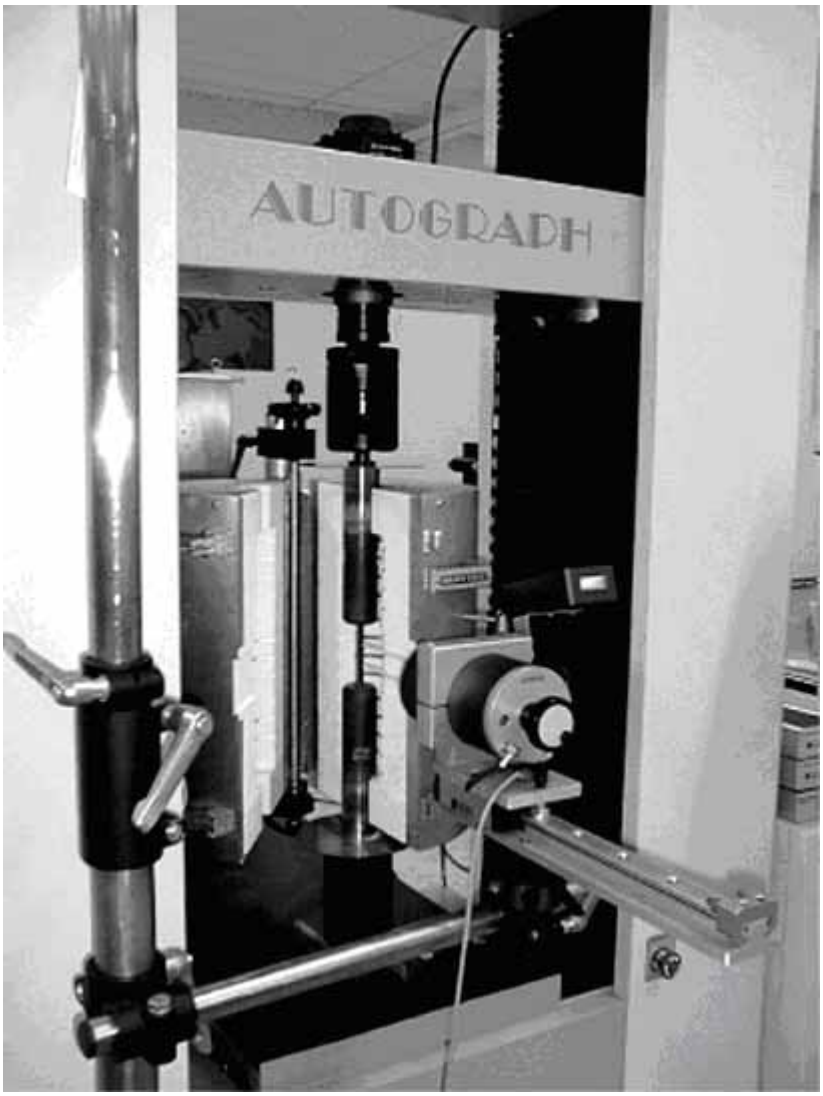
$$100\,^{\circ}C \xrightarrow{5\,K/min} 550\,^{\circ}C\,(75\,min) \xrightarrow{5\,K/min} 755\,^{\circ}C\,(60\,min) \xrightarrow{5\,K/min} 1010\,^{\circ}C\,(25\,min)$$

1- rovnice 1. řádu, 2 – kinetická rovnice (1), 3 - exp. - experimentální body

Literatura: Kolář, F., Svítilová, J.: Acta Montana B 11 (120), 2001

Univerzální testovací stroj Inspekt 50kN

byl instalován v odd. uhlíkových materiálů ÚSMH AVČR. Umožňuje provádět tahové a ohybové zkoušky vzorků studovaných materiálů při zatížení do 50 kN. V peci do 1500°C lze uskutečňovat ohybové a tlakové zkoušky tepelně odolných materiálů na vzduchu nebo v proudu dusíku při zatížení do 2 kN. Pomocí vysokoteplotních extenzometrů je možno měřit deformaci ohybově nebo tahově namáhaných vzorků, např. vláknových kompozitů nebo drátů ze speciálních slitin, umístěných v peci.



Na fotografii je měřicí rám a příprava pokusu v tahovém uspořádání (pootevřená pec s válcovým pracovním prostorem, v popředí vpravo držák vysokoteplotního extenzometru Maytek zasahujícího při měření do pece). Přístroj byl pořízen v rámci projektu P2046110 „Analýza chování komplexních systémů“, zařazeného do Programu podpory rozvoje přístrojového vybavení progresivních vědních oborů AVČR. Bude využíván při řešení projektů zaměřených na výzkum teplotně stálých kompozitů a jiných materiálů.