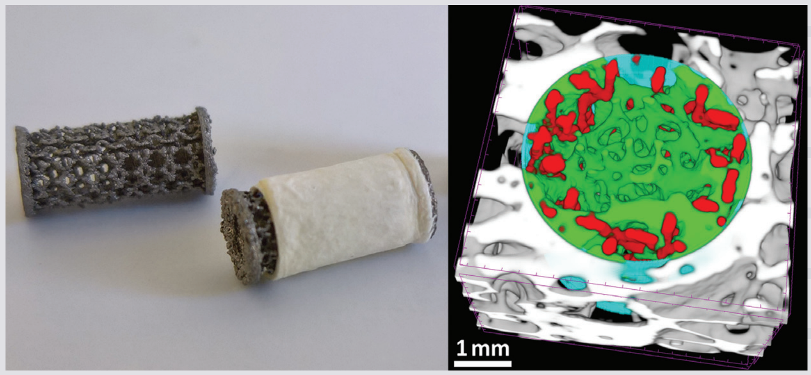
INSTITUTE OF ROCK STRUCTURE AND MECHANICS
The Czech Academy of Sciences

DEPARTMENT OF COMPOSITES AND CARBON MATERIALS

THEMATIC RESEARCH FOCUS

- **MODERN FIBROUS, PARTICULATE AND HYBRID COMPOSITE MATERIALS ON THE BASIS OF NATURAL OR SYNTHETIC MATERIALS**
- **UTILISATION OF COLLAGEN AND CALCIUM PHOSPHATES ISOLATED FROM NATURAL SOURCES**
- **COMPOSITE MATERIALS FOR MEDICAL USE**
- **HEAT-RESISTANT COMPOSITES**



3D-printed titanium implants and micro-CT image of the implanted titanium samples with collagen/hydroxyapatite electrospun layer with antibiotics (Vancomycin); the bone tissue is in white, the new bone inside the implant (red) is in green

MAIN SCOPE OF RESEARCH

- The Department of Composites and Carbon Materials focuses on advanced composite materials and their basic components, which have significant potential in two main areas, i.e. healthcare applications and applications where high temperature resistance is required.
- The team works on biomaterials as potential substitutes for both soft and hard tissues, and its aim is to be able to replace human tissues with materials that are as similar as possible to natural tissues from the viewpoint of their mechanical and structural properties and material composition. Concerning hard tissues, the team focuses on the main components of bone, namely collagen and calcium phosphates. Special attention is paid to isolating collagen and bioapatite from several natural sources (bones, skin, scales, etc.). The potential of natural materials and their applications as basic constituents of biocomposites is investigated, as well as methods for their processing. Special emphasis is placed on the utilisation of the electrospinning process for preparing sub-microfibres and nanofibres based on collagen and collagen fibres incorporating bioapatite nanoparticles or active substances as antibiotics. Several investigations are carried out, aimed at preserving the typical and advantageous unique biological properties of collagen and at imitating this major part of the extracellular matrix.
- The team also specialises in the development of unique composites, ceramic foams, ceramic matrix composites and all-ceramic sandwiches for use at elevated temperatures. Such composites are produced from a range of fibres, e.g. basalt, glass and ceramic fibres, and are fabricated using ordinary laminating technology, as used, for example, in the construction of ships and airplanes. They are then subjected to high temperatures in an inert atmosphere which significantly enhances their properties. The team has made particularly significant progress with so-called partially-pyrolyzed composites which are reinforced with basalt fibres. These materials have good mechanical properties and, unlike other fibre-based composites, they also exhibit a high degree of fire resistance.
- The team works primarily with instruments that allow it to process materials at the nanoscale. The team utilizes several devices that are able to electrostatically spin fibres from polymers. These devices are unique in that they allow the creation of fibres from natural polymers, a process which is far from easy given that natural materials rarely exhibit uniform properties. The team's most recent significant achievement concerns the application of its technology in the preparation of nanofibres from collagen, i.e. fibres with integrated nanoparticles of calcium phosphate which, in material terms, effectively imitate real bone tissue. These materials can be used, for instance, for the surface layers in hip and joint prosthetics so as to accelerate their growth into the bone tissue. Thanks to expert teams, knowledge from the fields of analytical chemistry, inorganic and organic chemistry, mechanics and biomechanics can be combined in the preparation of such materials, frequently with the involvement of the commercial sector. The team has long-standing experience of isolating collagenous materials and calcium phosphates from natural precursors (e.g. bones, skin) and their physico-chemical characterisation and monitoring of the quality of particular precursors using various techniques such as FTIR spectroscopy, X-ray diffraction, HPLC, chemical analyses, scanning electron microscopy, and energy-dispersive spectroscopy.

KEY RESEARCH EQUIPMENT

- Laboratory for the production of nanofibrous layers fully equipped for electrospinning natural and synthetic polymers, electrospinning devices 4Spin (Contipro), Electrospunra ES-210 (Mikrotools), high-voltage power supplies and linear pumps

- Apreo 2 LoVac High Resolution Scanning Electron microscope (Thermo Fisher Scientific) with SE, SED, DBS, STEM 3+ and EDS – Octane Elite Plus detectors and Microtest Tensile Stage 300N
- Sample preparation workflow – Automatic Tissue Processor, Trimmer, Ultramicrotom, Sputter Coater (Leica)
- Quanta 450 Scanning Electron Microscope (FEI) with SE, BSE CI, GAD, cathodoluminescence and EDS, SDD and EDAX detectors
- Nicolet iS50 Advanced and Nicolet Protégé 460 E.S.P. Fourier transform infrared spectrometers (Thermo-Nicolet)
- Shimadzu LC10 ADvp high-performance liquid chromatography system equipped with fluorescent and UV-VIS detectors
- MarSurf TS 50/4 non-contact surface measurement (Mahr)
- Inspekt 100 universal testing machine (Hegewald & Peschke)

- Clean room equipped with laminar flow boxes, steam and UV sterilization, centrifuges, freeze driers (VirTis Benchtop 8LZL and 4KZL) and a DH CO_2 incubator (Thermo Scientific)
- TMA PT 1600 thermomechanical analyzer (Linseis)

- Image analysis laboratory equipped with a Nikon Optishot 100S (Nikon) microscope supplemented with a ProgRes colour digital microscope camera (JENOPTIK) and NIS-Element AR system (Nikon)
- HLV 5.1 hydraulic moulding press (Pracovní stroje Teplice)
- HTK 8 GR/22-1Gman high-temperature graphite furnace (GERO)
- HAAKE R600 polydrive mixer (Thermo Electron Scientific)

ACHIEVEMENTS

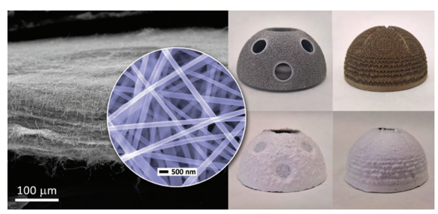
- **Collagen-calcium phosphate nanolayers with controlled elution of antibiotics for orthopaedic implants to be used particularly in the case of known prosthetic joint infections or as a preventative procedure regarding primary joint replacement at a potentially infected site**

T. Suchý, M. Šupová, F. Denk, Š. Rýglová, M. Žaloudková, Z. Sucharda, R. Ballay, L. Horný, Z. Čejka, M. Pokorný, K. Knotková, V. Velebný. A nanocomposite layer on the basis of collagen nanofibers, and a method of preparation thereof. European Patent Office. Patent EP3311854. 2020-07-09.

T. Suchý, M. Šupová, P. Sauerová, M. Hubálek Kalbáčová, E. Klapková, M. Pokorný, L. Horný, J. Závora, R. Ballay, F. Denk, M. Sojka, L. Vištejnová, *Evaluation of collagen/hydroxyapatite electrospun layers loaded with vancomycin, gentamicin and their combination: Comparison of release kinetics, antimicrobial activity and cytocompatibility*, Eur. J. Pharm. Biopharm. 140 (2019) 50–59. doi:10.1016/j.ejpb.2019.04.021.

T. Suchý, M. Šupová, E. Klapková, V. Adámková, J. Závora, M. Žaloudková, Š. Rýglová, R. Ballay, F. Denk, M. Pokorný, P. Sauerová, M. Hubálek Kalbáčová, L. Horný, J. Veselý, T. Voňavková, R. Průša, *The release kinetics, antimicrobial activity and cytocompatibility of differently prepared collagen/hydroxyapatite/vancomycin layers: Microstructure vs. nanostructure*, Eur. J. Pharm. Sci. 100 (2017) 219–229. doi:10.1016/j.ejps.2017.01.032.

T. Suchý, M. Šupová, E. Klapková, L. Horný, Š. Rýglová, M. Žaloudková, M. Braun, Z. Sucharda, R. Ballay, J. Veselý, H. Chlup, F. Denk, *The Sustainable Release of Vancomycin and Its Degradation Products From Nanostructured Collagen/Hydroxyapatite Composite Layers*, J. Pharm. Sci. 105 (2016) 1288–1294. doi:10.1016/S0022-3549(15)00175-6.



Collagen-calcium phosphate nanolayers with controlled elution of antibiotics can be directly deposited on the surface of orthopaedic implants (titanium or 3D printed acetabular cups)

M. Pokorný, T. Suchý, A. Kotziánová, J. Klemeš, F. Denk, M. Šupová, Z. Sucharda, R. Sedláček, L. Horný, V. Králík, V. Velebný, Z. Čejka, *Surface Treatment of Acetabular Cups with a Direct Deposition of a Composite Nanostructured Layer Using a High Electrostatic Field*, Molecules 25 (2020) 1173. doi:10.3390/molecules25051173.

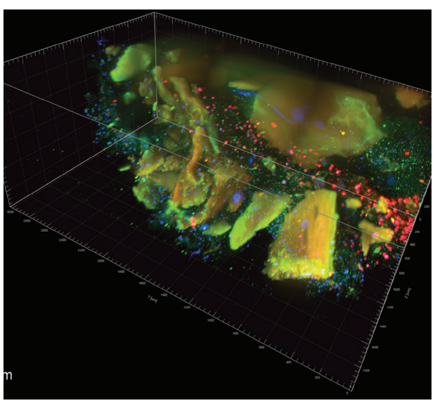
● Isolation and characterization of collagen and its potential for biomedical applications

T. Suchý, M. Šupová, M. Bartoš, R. Sedláček, M. Piola, M. Soncini, G.B. Fiore, P. Sauerová, M. Hubálek Kalbáčová, *Dry versus hydrated collagen scaffolds: are dry states representative of hydrated states?*, J. Mater. Sci. Mater. Med. 29 (2018). doi:10.1007/s10856-017-6024-2.

J. Horáková, P. Mikeš, A. Šaman, T. Švarcová, V. Jenčová, T. Suchý, B. Heczková, S. Jakubková, J. Jiroušová, R. Procházková, *Comprehensive assessment of electrospun scaffolds hemocompatibility*, Mater. Sci. Eng. C - Mater. Biol. Appl. 82 (2018). doi:10.1016/j.msec.2017.05.011.

P. Sauerová, T. Suchý, M. Šupová, M. Bartoš, J. Klíma, J. Juhásová, S. Juhás, T. Kubíková, Z. Tonar, R. Sedláček, M. Piola, G.B. Fiore, M. Soncini, M. Hubálek Kalbáčová, *Positive impact of dynamic seeding of mesenchymal stem cells on bone-like biodegradable scaffolds with increased content of calcium phosphate nanoparticles*, Mol. Biol. Rep. 46 (2019) 4483–4500. doi:10.1007/s11033-019-04903-7.

T. Suchý, M. Šupová, P. Sauerová, M. Verdánová, Z. Sucharda, Š. Rýglová, M. Žaloudková, R. Sedláček, M. Hubálek Kalbáčová, *The effects of different cross-linking conditions on collagen-based nanocomposite scaffolds – an in vitro evaluation using mesenchymal stem cells*, Biomed. Mater. 10 (2015) 65008. doi:10.1088/1748-6041/10/6/065008.



Adipose tissue-derived stem cells cultivated for 7 days in collagen gel reinforced with collagen electrospun particles. Cells are stained by specific anti-alpha-actin (red) and anti-calponin (green) antibodies. Cell nuclei are stained (blue) by DAPI (E. Filová, FGU CAS).

Š. Rýglová, M. Braun, T. Suchý, *Collagen and Its Modifications-Crucial Aspects with Concern to Its Processing and Analysis*, Macromol. Mater. Eng. 302 (2017). doi:10.1002/mame.201600460.

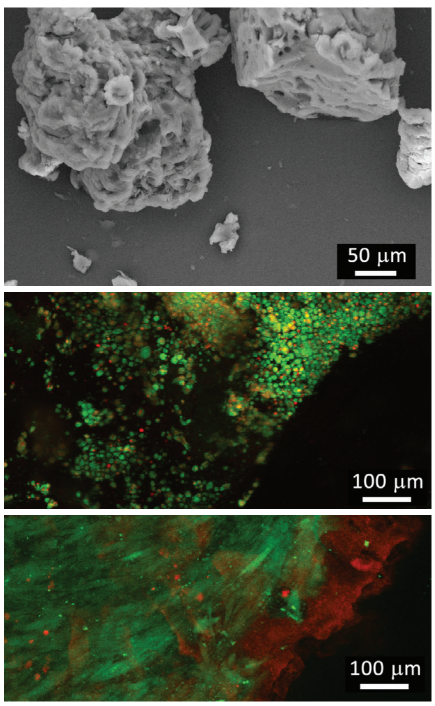
● Isolation and characterization of bone mineral and its potential for biomedical applications

M. Šupová, T. Suchý, Z. Sucharda, E. Filová, J.N.L.M. der Kinderen, M. Steinerová, L. Bačáková, G.S. Martynková, *The comprehensive in vitro evaluation of eight different calcium phosphates: Significant parameters for cell behavior*, J. Am. Ceram. Soc. 102 (2019) 2882–2904. doi:10.1111/jace.16110.

T. Šmrhová, P. Junková, S. Kučková, T. Suchý, M. Šupová, *Peptide mass mapping in bioapatites isolated from animal bones*, J. Mater. Sci. Mater. Med. 31 (2020) 32. doi:10.1007/s10856-020-06371-z.

M. Šupová. *The Significance and Utilisation of Biomimetic and Bioinspired Strategies in the Field of Biomedical Material Engineering: The Case of Calcium Phosphate-Protein Template Constructs.* Materials 13 (2020) 327.

M. Šupová, *Substituted hydroxyapatites for biomedical applications: A review*, Ceram. Int. 41 (2015) 9203–9231. doi:10.1016/j.ceramint.2015.03.316.

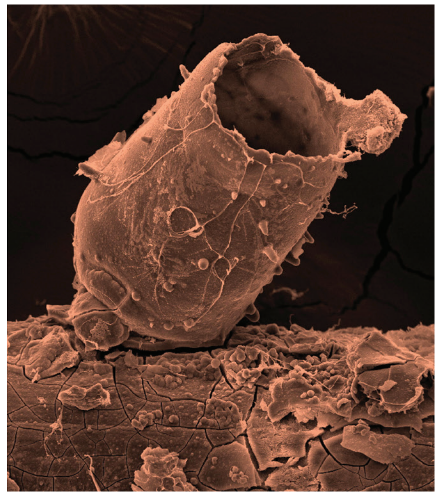


SEM image of bone mineral (bioapatite) particles isolated from human bone (left), human osteosarcoma cells (Saos2; middle) and human bone marrow-derived mesenchymal stromal cells (BM-hMSCs; right) cultivated for 14 days on polymer layers with bioapatite. Cells are stained by specific anti-alpha-actin (red) and anti-calponin (green) antibodies. Cell nuclei are stained (blue) by DAPI, (L. Wolfová IEM CAS).

● Biodegradable Mg wires covered in biodegradable polymers for biomedical applications

K. Tesař, K. Balík, *Nucleation of corrosion products on H2 bubbles: A problem for biodegradable magnesium implants?*, Mater. Today 35 (2020) 195–196. doi:10.1016/j.mattod.2020.04.001.

K. Tesař, K. Balík, Z. Sucharda, A. Jager, *Direct extrusion of thin Mg wires for biomedical applications*, Trans. Nonferrous Met. Soc. China 30 (2019) 373–381. doi:10.1016/S1003-6326(20)65219-0.



SEM image of a growing calcium orthophosphate tube on top of the commercially pure magnesium wire with a diameter of 250µm. This particular wire degraded for 48 hours in the cell cultivation media (αMEM).

● Advanced ceramic foams from pyrolysed polymer precursors

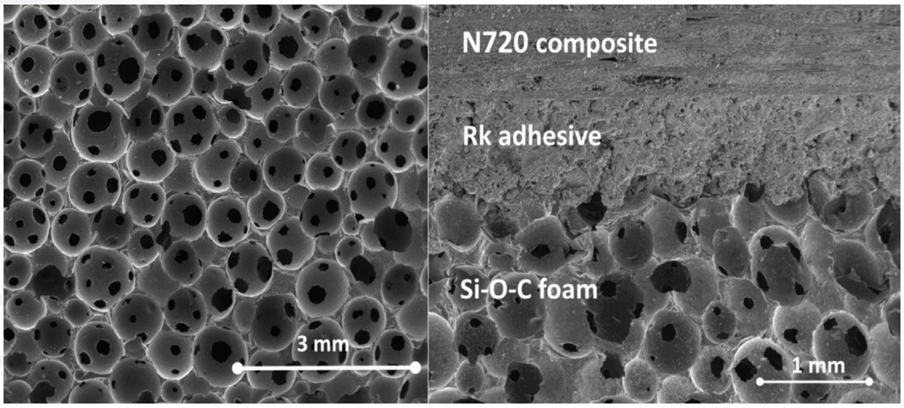
M. Černý, Z. Chlup, A. Strachota, J. Schweigstillová, J. Svítilová, M. Halasová, *Rheological behaviour and thermal dilation effects of alumino-silicate adhesives intended for joining of high-temperature resistant sandwich structures*, J. Eur. Ceram. Soc. 37 (2017) 2209–2218. doi:10.1016/j.jeurceramsoc.2016.12.046.

M. Černý, Z. Chlup, A. Strachota, M. Halasová, S. Rýglová, J. Schweigstillová, J. Svítilová, M. Havelcová, *Changes in structure and in mechanical properties during the pyrolysis conversion of crosslinked polymethylsiloxane and polymethylphenylsiloxane resins to silicon oxycarbide glass*, Ceram. Int. 41 (2015) 6237–6247. doi:10.1016/j.ceramint.2015.01.034.

M. Černý, Z. Chlup, A. Strachota, J. Svítilová, J. Schweigstillová, M. Halasová, Š. Rýglová, *SiOC ceramic foams derived from polymethylphenylsiloxane precursor with starch as foaming agent*, J. Eur. Ceram. Soc. 35 (2015) 3427–3436. doi:https://doi.org/10.1016/j.jeurceramsoc.2015.04.032.

A. Strachota, M. Černý, Z. Chlup, K. Depa, M. Šlouf, Z. Sucharda, *Foaming of polysiloxane resins with ethanol: A new route to pyrolytic macrocellular SiOC foams*, Ceram. Int. 41 (2015) 13561–13571. doi:10.1016/j.ceramint.2015.07.151.

M. Havelcová, A. Strachota, M. Černý, Z. Sucharda, M. Šlouf, *Effect of the dimethylsilyloxy co-monomer "D" on the chemistry of polysiloxane pyrolysis to SiOC*, J. Anal. Appl. Pyrolysis. 117 (2016) 30–45. doi:10.1016/j.jaap.2015.12.018.



Si-O-C ceramic foam prepared from preceramic polymer (left); application of Si-O-C foam as a lightweight core of all-ceramic sandwich with increased temperature resistance (right)

- **Hybrid composites made by partial pyrolysis**

M. Černý, M. Halasová, J. Schweigstillová, A. Strachota, Š. Rýglová, *Mechanical properties of partially pyrolysed composites with plain weave basalt fibre reinforcement*, Ceram. Int., 40 (2014) 7507–7521. doi: 10.1016/j.ceramint.2013.12.102

M. Černý, Z. Chlup, A. Strachota, J. Svítilová, *Potential of glass, basalt or carbon fibres for reinforcement of partially pyrolyzed composites with improved temperature and fire resistance*, Ceram. – Silik. 64 (2020) 64, doi: 10.13168/CS.2019.0056

Z. Chlup, M. Černý, A. Strachota, H. Hadraba, P. Kácha, M. Halasová, *Effect of the exposition temperature on the behaviour of partially pyrolysed hybrid basalt fibre composites*, Compos. Part B Eng. 147 (2018) 122–127. doi:10.1016/j.compositesb.2018.04.021.

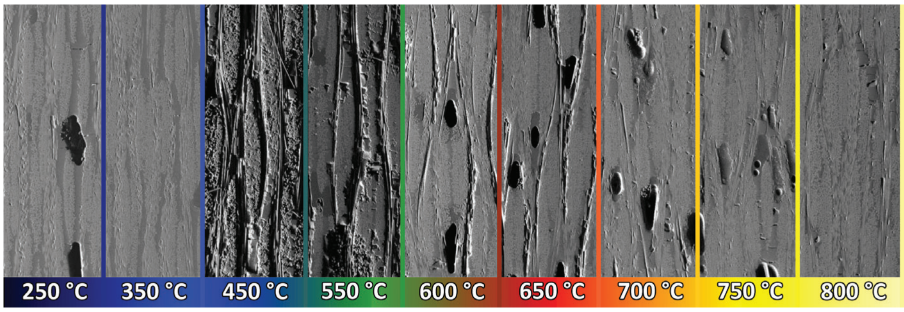
M. Halasová, M. Černý, A. Strachota, Z. Chlup, I. Dlouhý, *Fracture response of SiOC-based composites on dynamic loading*, J. Compos. Mater. 50 (2015) 1547–1554. doi:10.1177/0021998315594682.

A. Strachota, M. Černý, Z. Chlup, M. Šlouf, J. Brus, J. Plestil, Z. Sucharda, M. Havelcová, M. Halasová, *Preparation of silicon oxynitrocarbide (SiONC) and of its ceramic-fibre-composites via hydrosilylation/radical polymerization/pyrolysis*, J. Non. Cryst. Solids. 423 (2015) 9–17. doi:10.1016/j.jnoncrysol.2015.05.019.

A. Strachota, M. Černý, Z. Chlup, K. Rodzen, K. Depa, M. Halasová, M. Šlouf, J. Schweigstillová, *Preparation of finely macroporous SiOC foams with high mechanical properties and with hierarchical porosity via pyrolysis of a siloxane/epoxide composite*, Ceram. Int. 41 (2015) 8402–8410. doi:10.1016/j.ceramint.2015.03.037.

Z. Chlup, M. Černý, A. Strachota, J. Svítilová, M. Halasová, *Effect of ageing at 1200 degrees C in oxidative environment on the mechanical response of SiOC foams*, Ceram. Int. 41 (2015) 9030–9034. doi:10.1016/j.ceramint.2015.03.273.

Composite reinforced with basalt fibers and with a matrix in the hybrid state of polymer/ceramic conversion. The individual materials are indicated by the pyrolysis temperature with which they were prepared.



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- Technical University of Liberec (Liberec, CZ)
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- Devro, s.r.o. (Jilemnice, CZ)
- PrimeCell Advanced Therapy, a.s. (Prague, CZ)
- MEDIN, a.s. (Nové Město na Moravě, CZ)
- LA Composite, s.r.o. (Prague, CZ)
- LATECOERE Czech Republic s.r.o. (Prague, CZ)
- National Radiation Protection Institute, (Řež, CZ)

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