Description of the main research directions investigated by the institute

The main directions of research in the Institute are fully in line with the Institute Foundation deed. Historically, the Institute was focused on metallic materials, particularly steels. Over time, the field of materials has expanded, and currently the main research is aimed at identification and understanding relationships among mechanical and other physical properties of materials under broad spectrum of external conditions and their structural and microstructural characteristics. Priority is given to the research of advanced metallic materials and metal-based composites in relation to their microstructure and preparation method. The purpose of the research is to participate in development of new materials, optimize their utility properties and predict their service life by theoretical and computational methods based on experiments.

The great advantage of the Institute is its high homogeneity. All research groups are focused on material research and complement each other very well. The research groups cooperate on the stand of joint solution of grant projects (project research teams are often composed from scientists and technicians from different research groups) and in the use of laboratories. From this point of view, the Institute has undoubtedly a considerable research potential and capacity in materials science and its position in the Czech Republic is thus unique.

Since material research is very close to engineering practice and the Czech Republic is an industrial country, it is not surprising that the Institute has been cooperating very intensively with industrial companies since its foundation. Some of the co-operation with industry has been going on for decades. Success and quality of material research in the Institute for engineering practice was recognized by the Professional jury of the Technology Agency of the Czech Republic in 2019. The co-operation and results reached in the frame of the common research project of the Institute and the company PBS Velká Bíteš, a.s. focussed on high temperature long-term failure of nickel-based superalloys was selected as a best project of the Technology Agency of the Czech Republic in Business category for year 2019. This considerable success was presented on 55th meeting of the Academy Assembly of the Czech Academy of Sciences. Effective cooperation with industry, assistance in solving material problems and research motivated by problems coming from engineering practice will play a crucial role in the coming period and will represent one of the main general directions of research.

Since many years, the *investigation of materials for high temperature applications* is one of the key research areas in the Institute. In this field, results highly appreciated by the international scientific community have been obtained and published. The respective international reputation is clearly documented by the fact that the Institute has already been twice authorised to organize one of the most important world events in this field, the International Conference on the Strength of Materials (ICSMA 11 in 1997 and ICSMA 17 in 2015). Materials for high temperature applications have always played an important role in the life of society. Ever growing need to improve efficiency of thermal cycles, reduce fuel consumption and limit environmental impacts laid a ground for intensive research in the area over last couple of decades. It is evident that these topics will stay significant also for the future development of industrial countries like the Czech Republic. Therefore, there is a clear motivation for

the Institute to foster the basic as well as applied research in this material science field. The research in the Institute builds upon a long-term tradition that has been going on for decades since the Institute foundation. In early seventies of the last century, first creep machines were designed and constructed in the group led by prof. J. Čadek. This over-decades-long investment into the laboratory infrastructure and formation of excellent team of scientists and technicians with long experience made possible to perform basic studies of creep strain and damage accumulation in pure metals, single phase solid solutions as well as precipitation hardened alloys employed in engineering applications. In spite of these early achievements, numerous truly basic questions remain still open. In particular, rather strong temperature and stress dependencies of the stationary creep rate observed in engineering alloys belong to the most serious issues. Moreover, advanced material processing technologies launch new classes of materials intended for the high temperature service. Here we mention in-situ composite materials based on the NiAl intermetallic matrix, fcc and bcc compositionally complex alloys, in particular their single phase oxide dispersion strengthened variants and modern heat resistant steels like MarBN and Sanicro 25. The thermodynamic stability and creep resistance of these new materials need to be characterized and understood and certainly will represent a research portfolio in the coming years. In addition, recent research activities also focus on the design and processing of novel oxide dispersion strengthened steels with better microstructural stability and enhanced resistance to creep and high temperature oxidation. The Institute laboratories are very well equipped with unique testing systems which enable high precision measurements concerning the mechanical properties of materials designed for high temperature applications. An adaptive setup of 35 unique creep machines can accommodate four different loading modes, in particular combinations of tensile and compression strain under conditions of either constant load or constant stress. The temperature capability reaches up to the temperature of 950 °C while tests can be performed in either a range of protective atmospheres or in air. Furthermore, two new commercial testing facilities have extended the testing temperature range up to 1400°C for experiments performed in vacuum. Another highlight concerns the testing equipment for helicoidal samples with an unprecedented high strain sensitivity at low stresses which yields results highly relevant for industrial applications. This experimental base is an important prerequisite for a world-class quality research. Based on all these facts we expect that the results obtained in the future will bring new knowledge on materials for high temperature applications which will help to accelerate the development of industry and society not only in the Czech Republic but also on the international level.

The *fatigue damage of materials and application of fracture mechanics* is another important research direction in the Institute. Similarly to the basic and applied research focused on materials for high temperature applications, thanks to the achieved results and long-term systematic research, the Institute has a very good domestic and international reputation in this field. Fatigue and consequent failure of materials and structures is a phenomenon known and systematically studied for many years; in the Institute since its founding. Occurrence of fatigue damage is related to the response of materials subjected to cyclic external forces, which is the case of practically all engineering structures. The long term and running systematic research in the Institute has up to now resulted in a great collection of useful and published data and knowledge on the influence of stress distribution, geometry of

loaded bodies, frequency of cyclic loading, environment, surface finish, temperature etc. on the fatigue resistance. Simultaneously, research of the nature of the fatigue process on a microscopic level using the latest methods of microstructural investigation has brought a great deal of understanding on the irreversible changes in material due to mechanical cycling leading to fatigue and fracture.

In the coming years, research will focus on fatigue behaviour in relation to material structure and on the development of accurate tools for prediction of fatigue lifetime. Despite advances in the knowledge of fatigue mechanisms, fatigue fractures still occur and represent very dangerous failures of industrial components. Especially with regard to new technologies of material production as additive manufacturing they represent a major challenge for this research. Owing to this, the spectrum of studied materials has rapidly increased in last decade. At present and in the future, the non-metallic materials such as polymers, polymer or ceramics based composites and advanced building materials will be investigated. Attention will be also paid to materials made with new promising technologies, e.g. to materials produced by additive manufacturing.

The cyclic loading can be applied by various ways: uniaxial or multiaxial, symmetric or with the mean stress and in condition of negligible plasticity where fatigue life is correlated to stress or in condition with significant extent of plasticity in each cycle. The former condition is a domain of the investigations of the High Cycle Fatigue Group and the latter condition is the domain of the Low Cycle Fatigue Group. Loading resulting in large extent of plasticity leads to short fatigue life, however, there are many engineering applications working in such conditions. Moreover, the physical mechanisms of damage induced by cyclic plasticity can be revealed. It appears that the similar mechanisms may operate also in the high cycle fatigue regime or in real applications. These mechanisms often include movement and rearrangement of dislocations, linear defects of crystal lattice, and their interactions with other microstructural features like grain boundaries, precipitates, twins etc. The development of modern materials cannot proceed without detailed knowledge of these mechanisms. Therefore, we try to concentrate on perspective materials under development, like Sanicro steel, high entropy alloys (HEA), TiAl based intermetallics or superalloys and describe processes of cyclic plasticity in stress and temperature ranges close to expected working conditions. A special attention is given to mechanisms of fatigue crack nucleation where the both groups dealing with fatigue have a long tradition and large number of experimental and theoretical developments, including the famous Polak's model based on combination of vacancy diffusion and dislocation slip (prof. Polak, doyen of international fatigue community is still a member of the Low Cycle Fatigue Group). Finally, the fatigue crack growth in the low cycle fatigue exhibits differences (so called short crack effect) from the long crack growing under condition of negligible plasticity around the crack tip. Propagation of long cracks in small scale yielding conditions and problems of crack propagation thresholds and crack closure will be further intensively studied in the following period.

Staying competitive in the world scale requires follow the rapid progress in experimental characterization techniques of material structure, especially advanced techniques of electron microscopy. Simultaneously, collaboration of experimenters and scientists involved in modelling of the properties of materials will be intensified, particularly strengthening of joint actions with skilled researchers investigating electrical and magnetic properties and performing ab-initio and molecular static modelling. Concerning studied materials the research will be oriented on material for

high temperature applications: Sanicro 25 and other austenitic steels, ODS steels, superalloys with coatings (this research has direct relation to cooperation with industry) and HEA alloys. The investigation of fatigue damage in the coming period will be conducted in a broad international cooperation, which is described in detail in particular Reports on the research activity of evaluated teams. The outlined main direction of research on fatigue damage of materials and application of fracture mechanics is well ensured in the Institute. There are two very experienced teams of scientists and laboratories equipped with sophisticated machines and structures operating under high cyclic stresses, high temperatures, severe environment etc. Intensive exploitation of the most advanced electron microscopy is of utmost importance for successful fulfilment of mentioned scientific topics. Here it is expected that the institutional involvement in large research centres, in cooperation with universities and in CEITEC Nano will enable exploitation advanced experimental instruments and the most progressive methods for the material characterisation.

Very living and new trend in materials research in the Institute is the investigation of *materials for health care.* The ideas come out from the successful activities and promising results of the Institute reached and published recently. The subject of this research will be (i) preparation and characterization of magnetic nanoparticles, microparticles and magnetic materials systems in general, (ii) investigation of particles released from automotive break pads, (iii) investigation of materials for implants into human body. As far as the magnetic nanoparticles in health care are concerned, those made of iron oxides are nearly ideally biocompatible and very suitable for a medical use in hyperthermia treatment of cancer. Preparation of these nanoparticles from various precursors, characterization of their structure, examination of their thermal stability as well as determination of their magnetic properties have a long tradition in the Institute and the necessary laboratory equipment and knowledge are available. Regarding the public health, the researchers of the Institute have responded in recent years to widely-publicized calls for examining automotive brakes where potentially harmful microparticles can be released during friction processes. Advanced characterization experimental techniques will be applied to probe both the chemical composition and structure of vehicle brake pads with the aim to describe and analyse this problem. The brake pad materials are in fact rather complex, their composition very sensitively depends on the actual manufacturer and they indeed contain a number of potentially harmful elements. Therefore, this represents a very consequential health and environmental topic for future studies. As far as the materials for implants into human body are concerned, we focus on a fascinating property of the near-equiatomic NiTi alloys to “remember” their shape. A reversible martensitic transformation, which establishes a base for the effect, enables the material to get back into its original shape after almost any arbitrary deformation. The reversible deformations of NiTi alloys outperform, by an order of magnitude, a reversible spring backs of classical spring steels. This unique ability of the NiTi alloys is intensively employed in medical fields like a bone and cardio surgery. However, the applications of NiTi alloys in the bio-environments are accompanied by adverse effects. The most serious is a release of toxic Ni ions which may cause an inflammatory response and allergic reactions in about 20 % of patients. Based on our expertise and recent activities in the field, we would like to start up a program on suppressing the adverse reactions. Promising directions seem to be the optimization of NiTi implant surfaces and doping the alloy by controlled doses of hydrogen. This program will be implemented in collaboration with our colleagues at the University

Hospital Brno and Department of Pathophysiology at Masaryk University. Another important direction of material research for health care will be investigation of mechanical properties of implants with complex shape produced by additive manufacturing (AM). For a reliable application of implants which are exposed to cyclic loads deeper understanding of their fatigue behaviour is urgently required. For prediction of fatigue life the knowledge of data characterizing the fatigue strength is a necessary prerequisite. Because the microstructure of materials produced by AM is substantially different from that of conventionally produced alloys the properties can differ substantially. Thus, basic and applied investigation of properties of AM materials will be performed in international collaborations and in co-operation with domestic companies in the following period.

Research and evaluation of brittle states in steels, ceramics and composites from the microstructural and fracture point of view is another topic which has long and successful tradition in the Institute. Until the nineties of the last century, the internationally recognised authority M. Holzmann formulated this basic research direction that is still up to date. His team contributed strongly to understanding and evaluation of brittle states in steels. Theory of dynamic fracture toughness based on an experimental knowledge obtained by impact tests developed in the Institute belongs to pioneering works in brittle fracture issue. Note that designers from Zwick Company, a leading producer of fracture testing systems, have been cooperating with the Institute for a long time with the aim to progress in mechanical methods of fracture tests. Arising from extensive long-term experiences and previous fruitful and internationally acknowledged activity the research is aimed to quantitative assessment and predictions of microstructure - property relationships in structural steels, ceramics, metal matrix and ceramic matrix composites. The physical nature of brittle fracture of advanced engineering materials is intensively investigated. Thanks to this basis the Institute and particularly the Brittle Fracture Group was very successful in a number of interdisciplinary research projects supported by EU funds. Among others, we contributed to biomaterials development, e.g. in field of tissue engineering with porous Bioglass® biodegradable foam with hybrid coatings or in the field of antibacterial coatings for medical implants. This research fits to the research direction of materials for health care, mentioned above. Another very promising direction of research are geopolymer based materials prepared from coal fly ash of fossil power stations and glass waste. This activity contributes not only to environmentally important waste itineration but also enabled invention of hydro-pressure cold densification process of ceramic materials. Comprehensive set of knowledge in the field of ceramic-based laminates contributed to novel layered architecture which can protect brittle piezo ceramics against cracking and harsh environments as demonstrated on protective layered system for barium titanate energy harvester. The exact knowledge of the materials response to mechanical loading including micromechanisms of failures and modelling tools played a crucial role in the mentioned examples. The research team dealing with fracture resistance including brittle states of materials is capable and has all prepositions to be successful in application of theoretical, experimental and computational approaches for materials design, preparation, processing, and characterisation aiming to enhance fracture resistance.

In the field of the experimental material research, the Institute is also involved in the development of *perspective materials for hydrogen storage*, which represent

promising possibility to store energy from clean sources. The scientific interest is and will be oriented towards better understanding of underlying processes during sorption and desorption of hydrogen, and the ways of operational temperatures improvement, which is combined with the studies of perspective additives to Mg-based alloys as well as development new materials for practical applications.

Materials research at the Institute is well balanced in terms of experimental and theoretical methods. This trend will be maintained and strengthened in the future. *Theoretical modelling of materials properties* is a research direction to which great attention will be paid in the next period. As the structure-property relations, which the research in the Institute focuses on, are inherently multi-scale, the modelling will be of the same nature. Due to the fact that comprehensive multi-scale modelling schemes are very rare up to now, we combine a number of methods designed for different length or time scales into suitable multi-methodological approaches. A particularly important type of modelling is quantum-mechanical calculation which determines the electronic structure of materials. Ab-initio modelling provides an insight into material behaviour at the length scale of individual atoms, i.e. well below the resolution of even the best high-resolution transmission electron microscopy (TEM) techniques. As a modelling-related outlook, we also plan to include newly emerging computational tools. These will include, for example, methods of artificial intelligence, such as machine learning and neural networks, when dealing with growing amount of our results and measured data. Next, as the quantum-mechanical calculations nowadays often reach computational limits of current supercomputers, we intend to focus also on performing our calculations on quantum computers which promise to offer a much bigger computational power. We already successfully performed first calculations on quantum IBM Quantum Experience computer.
Physical properties of crystalline materials are determined by the types, concentrations and spatial distributions of lattice defects and their rearrangements under the external electric, magnetic or mechanical fields. Understanding these effects is especially important in body-centered cubic (bcc) metals (V, Nb, Ta, Mo, W, Cr, alpha-Fe) whose plastic deformation is not governed by the Schmid law. IPM together with the University of Pennsylvania are the world leaders in theoretical and experimental studies of these materials. On the theoretical side, we employ the semi-empirical Bond Order Potentials developed at ICAMS Bochum and the University of Pennsylvania to describe the mixed covalent-metallic character of bonds in these materials. Molecular statics simulations using these potentials have provided crucial insight into their initial stage of plasticity, both from the point of view of isolated screw dislocations as well as from the behavior of their junctions. These data have been used to develop single-crystal yield criteria (so far for Mo, W, alpha-Fe and Cr) and thermodynamic models of slip that have already found their way into graduate-level textbooks (L. P. Kubin: Dislocations, mesoscale simulations and plastic flow, Oxford University Press). In the upcoming period, we intend to reopen the experimental program to investigate the low-temperature plasticity of these materials in combination with computer modelling.
Another class of materials that receives significant attention, and which will be investigated both by modelling and experimental ways, are III-nitride semiconductors GaN, AlN and InN. These materials have found widespread use in high electron mobility transistors, solid state lighting, and in water purification by UV radiation. Their epitaxial growth on Si substrates results in large lattice misfit that gives rise to threading dislocations. These dislocations are known to reduce the device efficiency

and thus a problem of major technological importance is to unravel, and possibly block, the mechanism by which these dislocations are nucleated. This problem is being targeted in collaboration by employing a range of experimental methods due to the close collaboration of IPM (scanning electron microscopy, measurements of electron beam induced current - EBIC), company ON Semiconductor (epitaxial growth), research infrastructure CEITEC Nano (scanning probe microscopy, X-ray photoelectron spectroscopy) and newly established company NenoVision (development of correlative probe electron microscopy in combination with a nanoEBIC technique). On the theoretical side, the progress in the field has been hampered by the lack of a theoretical model that includes the ionic component of bonding into account. The only simulations available to date were thus made using Density Functional Theory (DFT) on small computational cells. To overcome this obstacle, we aim to develop an ionic-covalent model of bonding in which the charge redistribution will be determined self-consistently with the positions of atoms.

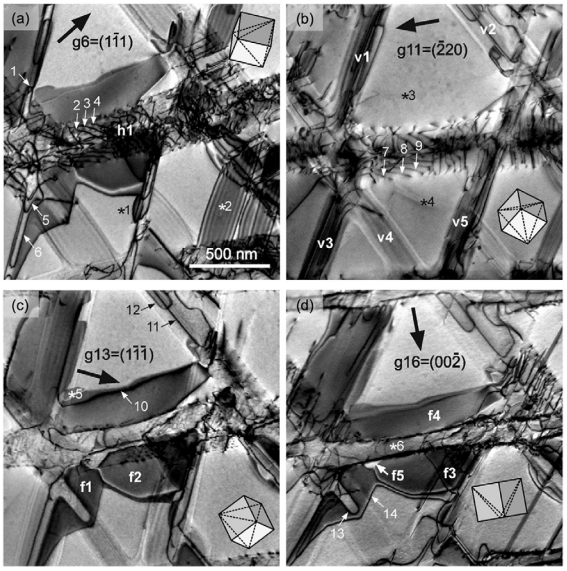
Another branch of the theoretical modelling pursued at the Institute is oriented towards development and application of the semiempirical CALPHAD method, which allows the theoretical modelling of thermodynamic properties and phase diagrams for multicomponent systems. The CALPHAD method can be very efficiently used together with above mentioned ab initio modelling, as it can supply the data required for the CALPHAD method which are not always available from experiments. The mutual synergy between both methods leads to the extended applicability of quantum-mechanical calculations in the field of applied research. The CALPHAD research at the Institute is oriented both on the further development of the method in the field of basic research and for the practical application in the new material development. The work on new types of thermodynamic databases (based on so called 3rd generation unary data) and possible extension of CALPHAD towards the application for nanomaterials belongs to the former, the development of complex thermodynamic databases for several classes of advanced materials (e.g. lead-free solders and thermoelectric materials) is crucial for the latter topics.

Research activity and characterisation of the main scientific results

Principal results achieved by the AHTM group in the evaluation period 2015-19 can be sorted out into six categories; which summarize outcomes of basic experimental and theoretical studies as well as research for industrial applications.

Category 1 - standard HT superalloys and steels

In collaboration with partners at the Ruhr University Bochum (RUB), the AHTM team provided a key input related to the nucleation of planar faults in the early stages of low temperature (750°C) and high stress (800 MPa) creep of a Ni-base single crystal superalloy (SX). Using scanning transmission electron microscope (STEM), we have shown that two families of 60° dislocations with different Burgers vectors react and form a superlattice intrinsic stacking fault in the γ' precipitate phase. Our 2D discrete dislocation model helped to fully rationalize a sequence of events which lead to the fault nucleation. These results represent microstructural evidence suggesting that the cutting of γ' precipitates by planar faults governs the early stages of creep in SXs. In particular, the described scenario fully rationalizes the double creep rate minima observed during primary creep of <001> oriented SXs. The AHTM team performed the complete STEM analysis of dislocation structures, formulated the discrete dislocation dynamics (DDD) model and provided its full numerical solution. The AHTM contribution can thus be estimated as 60% of the overall effort [C1_1, C1_2].



Advanced STEM analysis of dislocations and planar faults after 1% tensile creep strain accumulated in SX at 750°C and 800 MPa [C1_2].

Besides the Ni-base SX, the team attention has also been paid to superalloys with the Co-base matrix. Creep strain and creep damage accumulation in two cast high-chromium cobalt-based superalloys strengthened by niobium and tantalum were investigated at temperatures 900, 950 and 1000°C in a tensile stress range from 40 to 80 MPa. These alloys sustain harsh environmental conditions typical e.g. in glass production technologies. It was found that the CoNb superalloy possesses considerably longer creep life compared to the CoTa variant. The homogeneously

distributed creep damage of the CoNb superalloy is closely connected with primary carbides and is predominantly initiated either as interface decohesion between the carbide/matrix and carbide eutectics/matrix or by breakage of bulk M23C6 carbides. The final brittle fracture in the CoNb superalloy occurs via relatively fast propagation of the longest cracks after the damage accumulation reached a critical ultimate state. These results helped to understand the mechanisms of the premature creep fracture and thus serve as a basis for further optimization of the alloy composition and microstructure [C1_3].

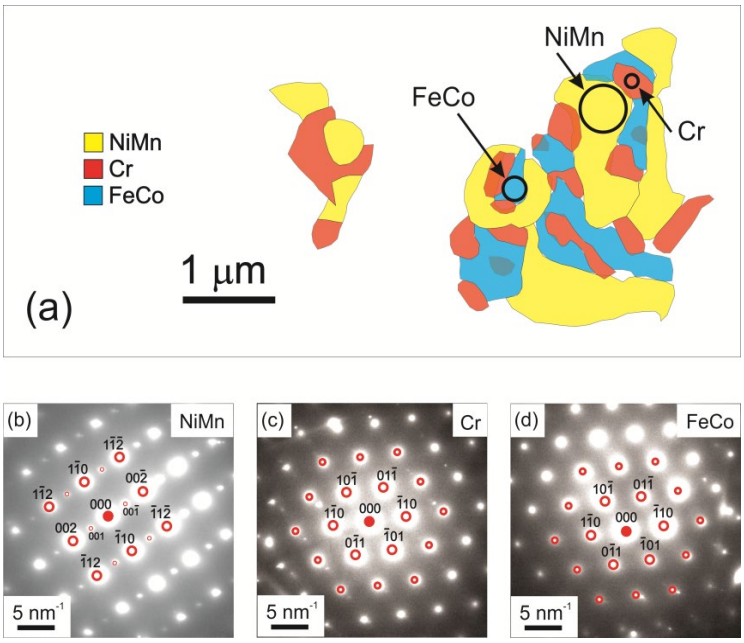
A remarkable progress has been achieved concerning a basic understanding of creep strain and creep damage accumulation in austenitic as well as tempered martensite ferritic steels, again with important implications for future development of HT steels. The low-alloyed T23 steel was subjected to creep testing and an intensive electron microscopy analysis in order to identify creep deformation mechanisms which were so far not fully understood. It was found that diffusion in the matrix is the creep-rate controlling process. A coarsening of microstructure leads to a drop of dislocation density, precipitation and solid solution hardening which, in turn, results in a significant decrement of creep strength during long-term creep exposures [C1_4]. Effects of welding operations on the creep performance of the HT steels P92 and HR3C were investigated within a framework of national (the Ministry of Industry and Trade) and European (the European Commission FP7) projects. In both cases, the creep performance of the material affected by welding heat was compared to the creep characteristics of the base metal and accounted for on the basis of the underlying microstructural changes [C1_5, C1_6].

[C1_1] Wu X. et al., Acta Materialia 112 (2016) 242-260.
[C1_2] Wu X. et al., Acta Materialia 144 (2018) 1-14.
[C1_3] Sklenička V. et al., Materials Characterization 144 (2018) 479-489.
[C1_4] Sklenička V. et al., Materials Characterization 109 (2015) 1-8.
[C1_5] Sklenička V. et al., Materials Characterization 119 (2016) 1-12.
[C1_6] Sklenička V. et al., Materials Characterization 128 (2017) 238-247.

Category 2 - advanced alloys and materials for special applications

As members of the international consortium, we have evaluated phase stability of face-centred cubic (FCC) high entropy alloy (HEA) CrMnFeCoNi after very long anneals of 500 days at 500-900°C. It is reasonable to expect that these long term treatments established thermodynamic equilibrium. Microstructural analyses were performed using complementary analysis techniques including scanning and transmission electron microscopy (SEM/TEM/STEM), energy dispersive X-ray (EDX) spectroscopy, selected area electron diffraction (SAD), and atom probe tomography (APT). We show that the alloy is a single-phase solid solution after homogenization for 2 days at 1200°C and remains in this state after a subsequent anneal at 900°C for 500 days. However, it is unstable and forms second-phase precipitates at 700 and 500°C. A Cr-rich s phase forms at 700°C, whereas three different phases ($L1_0$-NiMn, B2-FeCo and a Cr-rich body-centred cubic, BCC, phase) precipitate at 500°C. Since there is limited entropic stabilization of the solid solution state even in the extensively investigated CrMnFeCoNi alloy, the stability of other HEAs currently thought to be solid solutions should be carefully evaluated. Two AHTM team members (A. Dlouhý

and M. Kuběnová) contributed to the results (share of about 40%), namely by the exact FIB cutting of STEM samples and the full STEM diffraction and chemical analysis of the constituting phases. Respective parts of the text in the experimental methodology, results and the discussion sections were written by A. Dlouhý [C2_1].



SAD analysis of the CrMnFeCoNi alloy after long term annealing at 500°C. (a) Arrowed circles indicate the sizes and locations of the apertures used for SAD on the precipitates (NiMn, Cr, and FeCo). (b), (c) and (d) present the corresponding SAD images [C2_1].

Creep of the same CoCrFeMnNi alloy, manufactured using the powder processing route, was investigated in the temperature range 700-800°C. The alloys were prepared by milling powder blends of pure elements in a planetary ball mill and compacting by the spark plasma technique. Two variants of the alloys were prepared: (i) without and (ii) with oxide dispersions. Creep resistance was substantially improved by the presence of oxides. Lattice diffusion was suggested as a possible creep mechanism at low stresses. The effective diffusion coefficient calculated for Nabarro-Herring creep was comparable to the lattice diffusion coefficient of Ni in the same high-entropy alloy. At high stresses, the creep behaviour was characterized by the presence of a threshold stress invoked by oxide particles. Creep experiments, their analysis and TEM observations were completely done in AHTM group. Co-authors from the Brittle fracture group of the Institute of Physics of Materials (IPM) provided experimental alloys prepared in cooperation with the Institute of Plasma Physics CAS (Prague). Contribution: 70% AHTM group, 30% others [C2_2].

The attention has also been given to another compositionally complex alloy CoNiCrAlY. Creep tests of this bond coat material were conducted in the temperature range from 800 to 1200°C. The alloy was prepared from a consolidated powder with a nominal chemical composition of 34.2% Co, 28.6% Ni, 21.2% Cr, 15.6% Al, 0.4% Y (atomic percent). Results suggest that the stress exponent n decreases with the increasing temperature from 3.9 at 800°C to approximately 1 at 1200°C. Consequently, the activation energy of creep is stress dependent and ranges from 512 kJ/mol to 224 kJ/mol with increasing stress. Combined creep and constant strain rate data can be described by the Garofalo (sinh) equation. Creep resistance of the

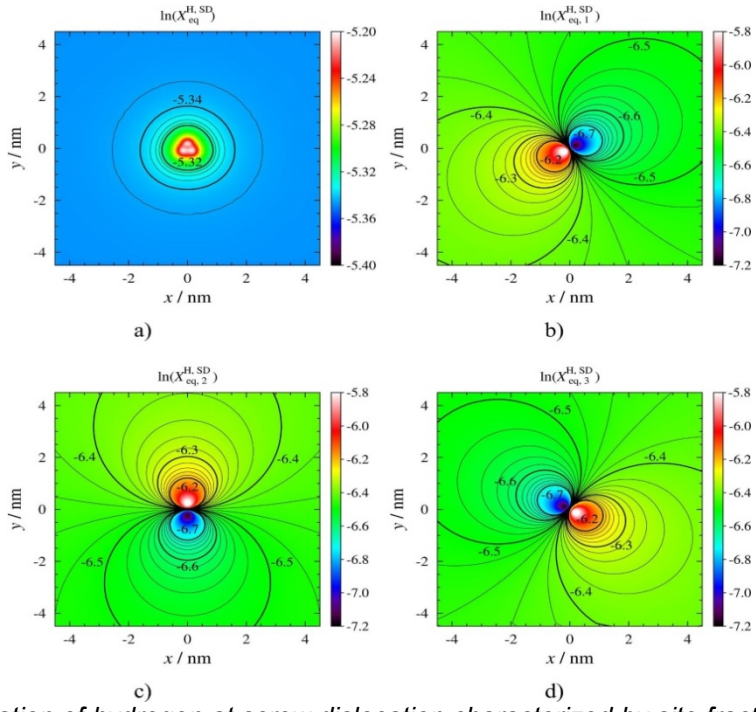
present alloy clearly overwhelms the resistance of bond coat materials reported so far. Creep experiments and their analysis were fully performed by the AHTM team. Co-authors from Yokohama National University (YNU) prepared the experimental alloy, ran the constant strain rate tests and SEM observations. The authors' contributions: 70% AHTM, 30% YNU [C2_3].

In agreement with the research plan for the period 2015-19, a part of the AHTM group research effort has been dedicated to creep in zirconium alloyed by Nb. These alloys have been extensively used as a cladding material in nuclear power stations. The alloy Zr-1 wt% Nb was tested at elevated (350°C) and high (600-850°C) temperatures, the high temperature range mimicking the loss-of-cooling-accident (LOCA) for fuel cladding tubes inside the reactor. It has been shown that Zr-1 wt% Nb alloy exhibits extraordinary creep ductility, mainly at temperatures corresponding to the LOCA conditions. Pronounce changes in the stress sensitivity of the creep rate indicate that there is a transition in creep mechanisms with decreasing applied stress. Therefore, creep properties of the alloy in the LOCA regime cannot be extrapolated from that obtained at the lower (operational) temperatures [C2_4].

[C2_1] Otto F. et al., Acta Materialia 112 (2016) 40-52. Currently 216 citations, highly cited paper according to WOS.
[C2_2] Dobeš F. et al., Materials Science & Engineering A 732 (2018) 99-104.
[C2_3] Dobeš F. et al., Materials Science & Engineering A 759 (2019) 272-277.
[C2_4] Kvapilová M et al., Acta Phys. Pol. A 128 (2015) 548-551.

Category 3 - modelling

A comprehensive study and associated overview addressing the interactions between impurity interstitial atoms (IIAs) and dislocations has been published in the highly ranked journal Progress in Materials Science. It has been shown that IIAs occupy octahedral or tetrahedral sites in bcc metals. Both types of occupation sites show only tetragonal symmetry and can be classified into three types according to their orientation with respect to the three main <100> crystallographic directions of the cubic lattice. As a result of the lower occupation site symmetry, the occupancy of the sites is different in the stress field of either screw or edge dislocation with a dislocation line oriented parallel to a particular crystallographic direction. The kinetics of segregation of interstitials at screw and edge dislocations has been modelled and simulated using the thermodynamic extremal principle (TEP). The numerical results helped understanding a high solubility of oxygen during mechanical alloying of modern ODS alloys. The model and the numerical code, on the basis of which the extensive results of simulations were obtained, were developed fully by Dr. J. Svoboda, the member of the AHTM group. The share of the effort can thus be estimated as 60% AHTM and 40% UNI and MC Leoben [C3_1].



Equilibrium segregation of hydrogen at screw dislocation characterized by site fractions (a) $X_{eq}^{H,SD}$, (b) $X_{eq,1}^{H,SD}$, (c) $X_{eq,2}^{H,SD}$, (d) $X_{eq,3}^{H,SD}$ [C3_1].

After more then seven years, the intensive development of 3D discrete dislocation dynamics (DDD) model, addressing behaviour of dislocation ensembles at high temperatures, has been successfully topped. In order to demonstrate its potential, the new model has been employed to investigate motion of general mixed dislocation segments subjected to high temperature loadings in microstructures with impenetrable particles. Our simulations focussed on migration of low angle dislocation boundaries in an array of particles while taking into account all mutual dislocation-dislocation interactions and the action of the externally applied stress. The results show, for the first time, that the migration of tilt dislocation boundaries in crystals with particles is associated with threshold stresses. The calculated thresholds are in a good agreement with experimental threshold stresses that characterize creep behaviour of precipitation hardened alloys [C3_2].

A transient grain growth represents important process which impacts microstructures of many HT materials. The distribution function f(R,t) of grain radii R, with R_c as a critical radius, has been formulated, inspired by the Hillert self-similar solution concept. Contrary to the Hillert's solution, the present approach generalizes the distribution as a product of $1/R_c^4$ and a shape function $g(R/R_c,t)$. The evolution equations for $R_c(t)$ as well as for $g(R/R_c,t)$ are obtained keeping the total volume of grains constant. The resulting integro-differential equations for $R_c(t)$ and $g(R/R_c,t)$ are solved using standard numerical tools. Remarkable advantages of this semi-analytical concept are: (i) the concept is deterministic, (ii) the numerical solution is very efficient and (iii) the shape function $g(R/R_c,t)$ remains localized in a fixed interval of R/R_c. The shape function $g(R/R_c,t)$ evolves towards the well-known Hillert self-similar distribution, which is demonstrated for two initial shape functions (one of them is triangular). The attention has also been given to "nearly" self-similar distribution functions proposed as useful approximations of experimental data. The model and the numerical code, on the basis of which the extensive results of simulations were obtained, were developed fully by Dr. J. Svoboda, the member of the AHTM group.

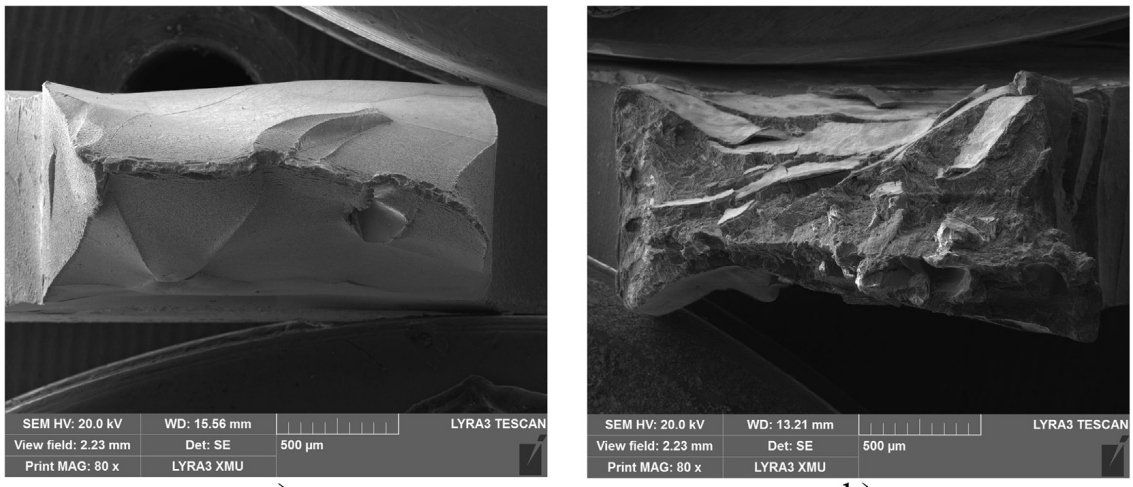
The share of the effort can thus be estimated as 70% AHTM and 30% UNI Leoben together with MPI Potsdam [C3_3].

High strength and creep resistance of modern steels and Al-alloys result from optimized precipitate structures. The understanding the role of precipitates in the high temperature deformation is thus of utmost importance. The kinetics of precipitation can be significantly influenced in cases when the precipitate-matrix misfit can be properly adjusted. The new model incorporated mechanisms controlled by the vacancy diffusion and addressed the accommodation of the misfit stress field by creep. The set of governing equations has been formulated and an efficient numerical algorithm has been provided which yielded full solution of the governing system. Steps have also been outlined towards an analytical form of the rate equation which describes the evolution of the contact pressure. This rate equation can be later implemented into models for treatment of precipitation kinetics in complex microstructures. Dr. J. Svoboda (AHTM) and prof. F.D. Fischer (UNI Leoben) joined forces and together developed the new model. Numerical results were obtained using a numerical code fully elaborated by Dr. J. Svoboda. The share of the effort can thus be estimated as 60% AHTM and 40% UNI Leoben. The model is now implemented into the software package MATCALC [C3_4].

[C3_1] Svoboda J. et al., Progress in Materials Science 101 (2019) 172-206.
[C3_2] Záležák T. et al., International Journal of Plasticity 97 (2017) 1-23.
[C3_3] Svoboda J. et al., Acta Materialia 115 (2016) 442-447.
[C3_4] Fischer F.D. et al., International Journal of Plasticity 64 (2015) 164-176.

Category 4 - special testing techniques

A miniature specimen testing technique has been used to compare creep performance of Fe-Al-base alloys subjected to either multiaxial or tensile stress states. The miniature specimen technique enables sampling of precious experimental heats where, due to a small amount of as-received material, a preparation of standard sized specimens is impossible. Small punch test and mini-tensile test were used to estimate mechanical properties of Fe-22Al and Fe-22Al-7Ti (at.%) at high temperatures. It has been shown that the force applied in the small punch test can be successfully converted into the equivalent applied stress using the empirical formula suggested by the new European standard for small punch testing. The minimum deflection rate can be converted into the minimum creep strain rate as well. The small punch testing made it possible to evaluate rupture properties including the creep rupture strength. This can effectively complement previous investigations of Fe-Al-Ti alloys performed predominantly in uniaxial compression. It has been shown that a significant increase in creep resistance is achieved by addition of titanium, which, in turn, leads to a two phase microstructure analogical to that in γ′-strengthened nickel-based superalloys. Results of deformation tests performed by the AHTM group represent the main part of the paper. The material characterization before and after high temperature testing (namely TEM) was done in cooperation with the Electrical and Magnetic group of IPM. The contribution of AHTM group is 70%, others 30% [C4_1].



a) b)

Fractographs of Fe-22Al alloy at 600 °C (a) tensile test, (b) creep test, rupture time 147 h [C4_1].

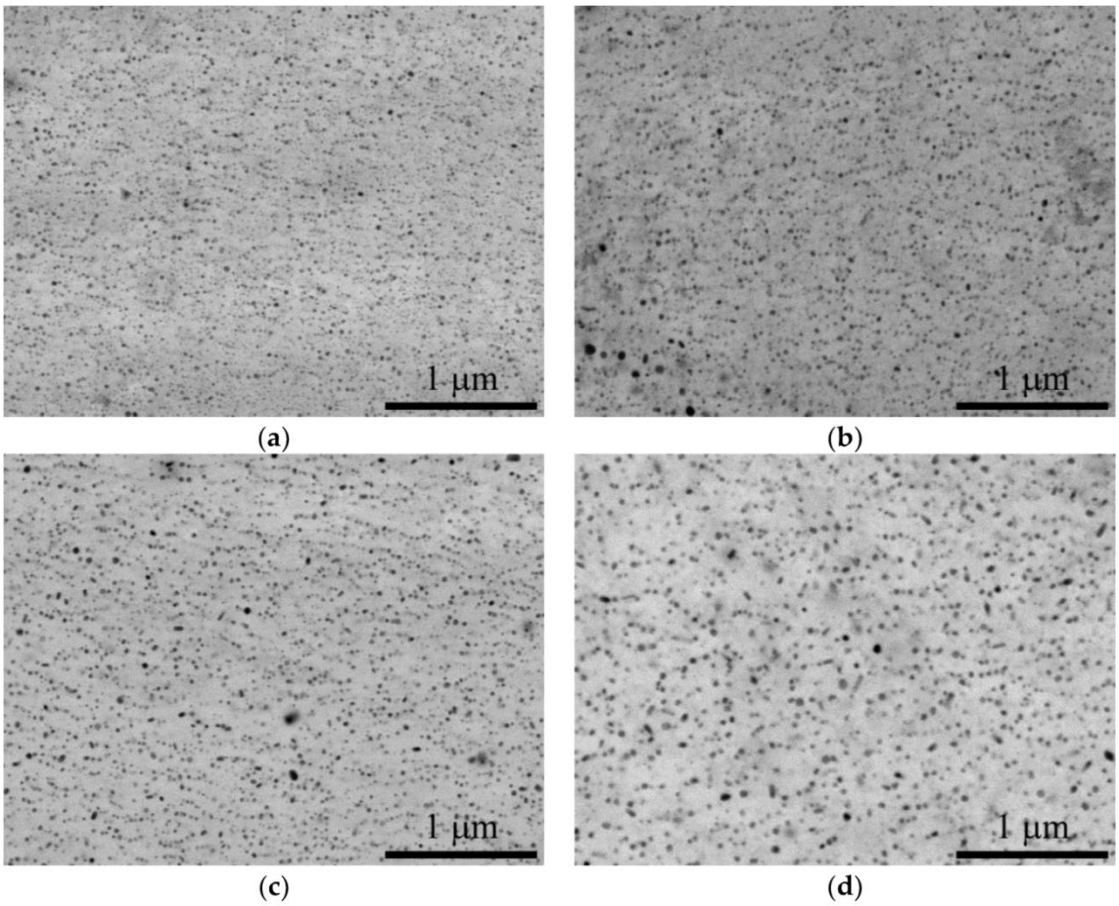
A low stress creep regime is particularly important concerning applications of HT materials in the engineering practice. Over decades, a unique experimental equipment of the AHTM group made it possible to collect rich experimental data on low stress creep in a broad range of structural materials. Based on these datasets, we have developed an internal stress model of transient low stress creep to address the effects associated with variable external conditions during the service life of HT components. The governing equations can be applied to predict creep strain accumulation kinetics of most structural materials, provided that the input parameters of the model are properly assessed. Conditions required for the implementation of the model into the 3D FEM codes have been indentified [C4_2].

The constant-deflection-rate small punch tests were also successfully employed in order to asses strength of pure aluminium and an aluminium composite processed by equal channel angular pressing (ECAP). Small punch tests were performed at the deflection rate of 0.005 mm/s at temperatures ranging from 20 to 350°C. Three main quantities have been determined: (i) yield force corresponding to the transition from elastic-bending to plastic-bending regimes, (ii) maximum load and (iii) the deflection corresponding to this maximum. The relations between these quantities and the corresponding quantities obtained in conventional tensile tests are analysed. Based on the number of ECAP passes, the observed grain size was successfully explained using a model by Starink et al. which addresses the grain refinement during severe plastic deformation. ECAP of pure aluminium, deformation experiments and their analysis were all completely performed by AHTM group of IPM. Co-author prof. Besterci provided ECAPed aluminium composite. The contributions: 70% AHTM group, 30% Slovak AS Košice [C4_3].

[C4_1] Dymáček P. et al., Theor. & App. Fracture Mechanics 99 (2019) 18-26.
[C4_2] Kloc L. et al., Mechanics of Materials 119 (2018) 49-55.
[C4_3] Dobeš F. et al., Materials Science & Engineering A626 (2015) 313-321.

Category 5 - materials design and processing

Using a new thermodynamic concept, two ODS alloys with a high-volume fraction of nano-oxides, namely the Fe-11Al-1O and Fe-17Cr-7Al-4Y2O3, have been developed. The alloys were manufactured by mechanical alloying and hot rolling. The original fine grain microstructures underwent static recrystallization at high temperatures. The kinetics of static recrystallization and coarsening of nano oxides in recrystallized grains have been determined for both systems. The difference in kinetics of coarsening of Al-based and Y-based oxides in the Fe-11Al-1O and Fe-17Cr-7Al-4Y2O3 systems is expressive and predetermines the Fe-17Cr-7Al-4Y2O3 system or similar ones to become the new leading system among creep- and oxidation-resistant materials for applications up to 1200°C. The new concept thus resulted in a radical improvement of microstructural stability as compared to similar ODS alloys reported so far in the literature [C5_1, C5_2].



SEM micrographs of oxides dispersed in grain interiors after annealing at 1200 °C for (a) 1 h, (b) 4 h, (c) 16 h, (d) 72 h in the Fe-17Cr-7Al-4Y2O3 system [C5_1].

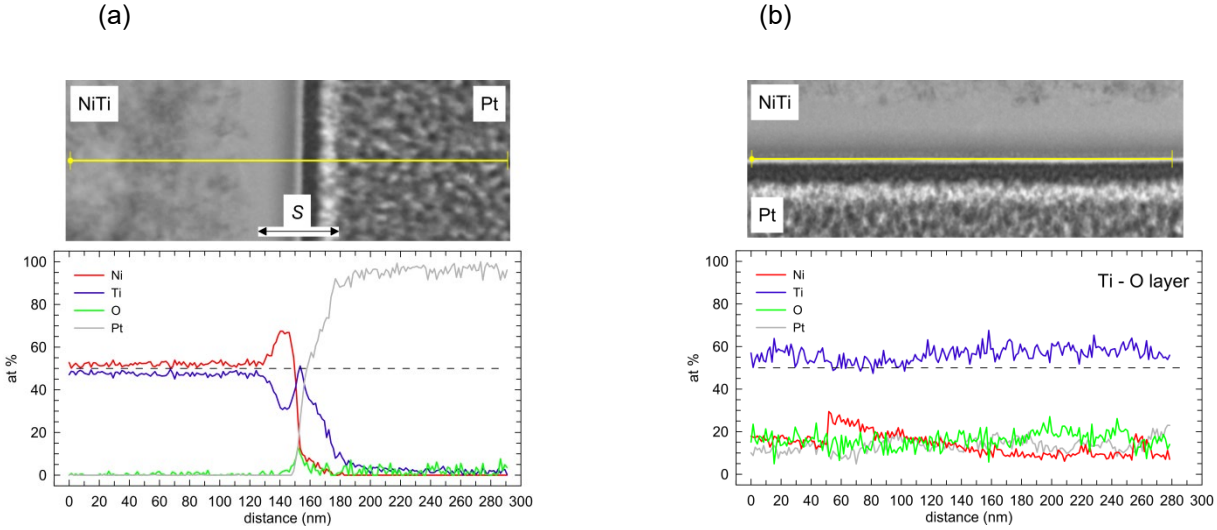
Strength and ductility of bio-compatible alloys are important parameters which may limit performance of the material in bio-environments. We have investigated the influence of processing parameters on the microstructural state of the biocompatible WE 43 Mg-based alloy. The alloy was processed via the combination of rotary swaging (RS) and friction stir processing (FSP) at three different rotational speeds of 400 RPM, 800 RPM, and 1200 RPM. The characterization of microstructure focussed primarily on the development of texture, evolution of grain size and grain boundary types. The results showed that swaging plus processing at 400 RPM and 1200 RPM

lead to substantial recrystallization and grain refinement. The grains were also the finest in the 1200 RPM sample (average grain diameter of 1.8 μm). The processed structures exhibited a slight tendency to form the {10-10} <0001> preferential fiber texture (especially the 800 RPM sample). Tensile testing showed the FSP to have positive influence on the ultimate tensile stress, as well as ductility of all the samples; the mechanical properties improved with increasing FSP rate. The overall contribution of the AHTM group members can be estimated as 80% [C5_3].

[C5_1] Svoboda J. et al., Metals 8 (2018) 1079; https://doi.org/10.3390/met8121079.
[C5_2] Dymáček P. et al., Key Eng. Mater. 8010 (2019) 113-118.
[C5_3] Kunčická L. Et al., Metals 9 (2019) 1181; https://doi.org/10.3390/met9111181.

Category 6 - shape memory alloys

In collaboration with the Department of Pathophysiology at Masaryk University Brno (DPMU), we have studied the impact of surface layers developed on the NiTi shape memory plates on the release of toxic Ni-ions into bio-environments. The NiTi alloys were exposed in the cell culture media and human vascular endothelial cell (HUVEC) culture environments. We have shown that the NiTi surface layers situated in the depth of 70 μm below a NiTi oxide scale are affected by interactions between the NiTi alloys and the bio-environments. The finding was proved with a help of inductively coupled plasma mass spectrometry and electron microscopy. As the exclusive factor controlling the Ni-ion release rates was not only thicknesses of the oxide scale, but also the passivation depth which was two orders of magnitude thicker. Our experimental data strongly suggested that some other factors, in addition to the Ni concentration in the oxide scale, admittedly hydrogen soaking deep below the oxide scale, must be taken into account in order to rationalize the concentrations of Ni-ions released into the bio-environments. The AHTM group contributed by about 60% to the research effort (alloy processing, STEM) [C6_1].



Ni, Ti, O and Pt elemental profiles (a) across and (b) along the surface layers in the Ni51Ti49 plate [C6_1].

Small-punch and nano-indentation tests were used for the first time to probe strength of 500 μm thin Ni50Ti50 shape memory platelets in their hydrogen-free and

hydrogen-doped states. Results show excellent reproducibility and suggest that hydrogen penetrates the alloy more efficiently during the cathodic charging at ambient temperatures as compared to heat treatments in a controlled hydrogen atmosphere. Hydrogen content exceeding 100 wt ppm results in a retransformation from the B19′ martensite to the R lattice and causes a systematic drop of the rupture strength. The retransformation events in thin surface lamellae were documented by TEM. All the small punch tests, cathodic hydrogen charging and the microstructural studies were performed by the AHTM team members. Dr. Jiří Čermák, a member of the Structure of Phases and Thermodynamics group (IPM) cared about the environmental hydrogen soaking experiments. Nano-indentation tests were done by Dr. Vilma Buršíková at MU, Brno. We estimate that the AHTM group contributed by about 80% to the overall research effort [C6_2].

[C6_1] Ševčíková J. et al., Applied Surface Science 427 (2018) 434-443.
[C6_2] Weiser A. et al., Scripta Materialia 162 (2019) 151-155.

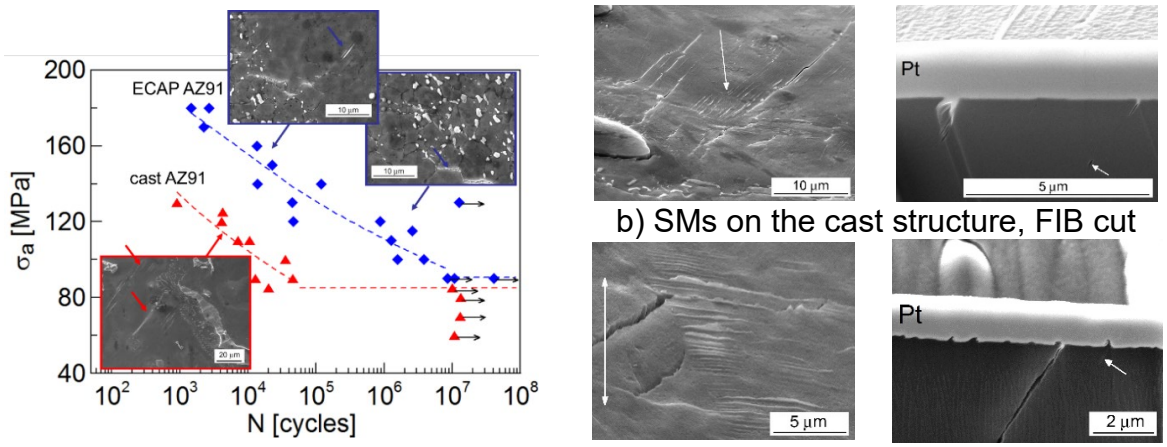
Research activity and characterisation of the main scientific results

Research activities of the group are currently focused on the following aims:

- fatigue performance of promising new materials or materials produced by advanced technological processes,
- description of fatigue cracks propagation,
- advanced numerical simulations of fatigue cracks propagation,
- fatigue and fatigue/creep damage of high temperature resistant materials,
- evaluation of long-term resistance of polymer materials,
- development of stability criteria of singular stress concentrators,
- fatigue behaviour and fracture of silica-based composites.

Fatigue performance of promising new materials or materials produced by advanced technological processes

A very promising technology, which brings new possibilities to manufacture complex and customized parts, is the Selective Laser Melting (SLM). A reliable prediction of fatigue life, however, is limited by the lack of knowledge on fatigue behaviour of specific structures of materials manufactured by SLM, which are substantially different from those produced by conventional ways. A project of Czech Science Foundation (Effect of the microstructure on fatigue of highly anisotropic stainless steels fabricated by SLM), solved in our group, is aimed at the development of new strategies for SLM production of stainless steels. To improve our expert skills in the field of additive manufacturing technologies, we have joined the H2020 project of European Commission – Structural Integrity and Reliability of Advanced Materials obtained through additive Manufacturing – focused on strengthening the knowledge on the additive manufacturing among research institutes in Eastern Europe. The project of Ministry of Industry and Trade of the Czech Rep. No. FV30219 (3D print implants to treat a damaged skeleton, especially the human pelvis) belongs to the same research area. In the frame of this project the effect of SLM technology on titanium alloys used for medical implants with respect to fatigue properties was deeply studied.
Also ultrafine-grained materials for bio-applications, namely AZ91 magnesium alloy and titanium alloys were studied intensively in the evaluated period. Thanks to grain refinement, the static mechanical properties of these materials are excellent in comparison with basic coarse-grained material. Therefore, these materials are successfully used for medical implants, where the same mechanical loading can be applied to implants with smaller dimensions. Even though the intensive grain refinement, achieved by severe plastic deformation, improves both static and cyclic properties in the case of materials with cubic lattice, different effects were observed in the case of metals with a hexagonal lattice. A positive influence of grain refinement on the static mechanical properties was reached in the case of bio-applicable AZ91 magnesium alloy (hexagonal lattice) and commercially pure Ti (grade 4). However, the fatigue endurance limit based on 10^7 cycles remained unchanged, although a significant increase of lifetime (two orders of magnitude) was observed in testing at stress amplitudes above the endurance limit, see Fig. 1a.



a) S-N curves

b) SMs on the cast structure, FIB cut

c) SMs on the UFG structure, FIB cut

Fig. 1. Fatigue behavior and crack initiation in AZ91 magnesium alloy

The small effect of the grain refinement on the fatigue endurance limit was explained by two different fatigue crack initiation mechanisms. Fatigue crack initiation related to slip markings (SMs) was shown to act in the case of the cast material and in the case of sporadic large grains (approx. 10 μm) in the ultrafine-grained (UFG) structure. The mechanism was revealed by focused ion beam (FIB) cutting (Fig. 1b-c). In addition to this mechanism, another one was observed playing a role at lower stress amplitudes in the case of the UFG structure. Grain boundary cracking was found to be responsible for the fatigue crack initiation at lower stress amplitudes in the small-grained areas. The second crack initiation mechanism decreased the fatigue endurance limit of the UFG structure to the value characterizing the coarse-grained cast material, see Fig. 1a). The results and their discussion were published in the work: *S. Fintová L. Kunz, Fatigue properties of magnesium alloy AZ91 processed by severe plastic deformation, Journal of the Mechanical Behaviour of Biomedical Materials 42 (2015) 219-228*. (this output was propounded for the evaluation). The described behaviour has to be considered in the design of medical implants, especially in the case of their long-term application.

Description of fatigue cracks propagation

An important aspect for assessment of residual fatigue lifetime or maintenance intervals of structural components is the ability to predict fatigue crack behaviour under given operative conditions. Traditional approaches usually use parameters describing a crack tip stress field, e.g. stress intensity factor. Then the fatigue crack growth rate is determined using Paris-Erdogan law or NASGRO equation. These approaches are based on phenomenological description of crack behaviour and deeper physical meaning is missing in some cases. We are focused mainly on two important areas: physically short cracks and threshold values of fatigue crack propagation. These areas are decisive for engineering applications, for damage tolerant approaches used in design of highly loaded engineering structures.

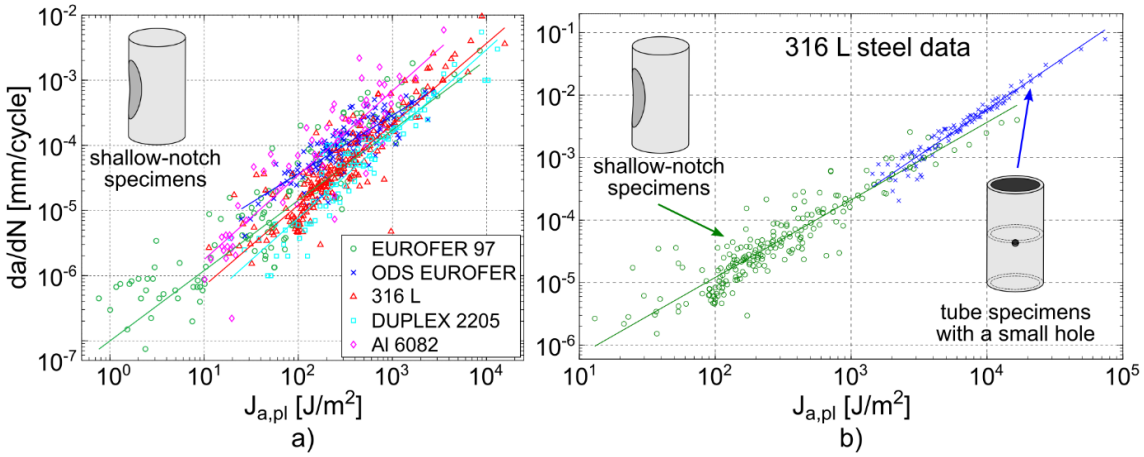


Fig. 2. Short fatigue crack propagation vs. plastic part of J-integral a) in the case of different materials b) comparison between 2 different specimens

To reach our goals we developed an original concept for short fatigue crack propagation description based on plastic part of J-integral. The proposed concept has been proved for several important engineering materials (316L steel, Eurofer 97 steel, ODS Eurofer steel, Duplex 2205 steel and aluminium alloy Al 6082), see Fig.2a. Particular experiments (performed by colleagues from Low Cycle Fatigue Group) were designed to measure short fatigue crack growth rates. The applicability of proposed concept was shown and published in *P. Hutař, J. Poduška, M. Šmíd, Ivo Kuběna, A. Chlupová, L. Náhlík, J. Polák, T. Kruml, Short fatigue crack behaviour under low cycle fatigue regime, International Journal of Fatigue 103 (2017) 207–215.* (this output was propounded for the evaluation). Experiments have shown an interesting phenomenon: the crack growth rate data of all the tested materials loaded at various loading levels plotted in log-log coordinates tend to lie on the same curve. This remarkable observation suggests that the short crack growth rate is determined by the amount of energy spent on plastic deformation, irrespective of the other material properties. Specimens with different geometries were used in the following to prove transferability of the experimental data, see Fig.2b. Recently the research team works on an extension of the proposed fatigue crack propagation description to more general conditions of mixed mode loading.

An important step during the evaluated period was done in separation and more detailed description of fatigue crack closure mechanisms. Significant influence of oxide induced crack closure on the resistance of material against fatigue crack propagation (especially in the threshold area) was found and investigated. A significant part of the threshold value corresponds to the crack tip shielding mechanisms. This part is often even higher than the natural material resistance to damage at the crack tip. Therefore, the crack tip shielding effects, mostly caused by crack closure mechanisms, represent very important topic for current research. Let us notice that metallic materials exhibit large scatter of the experimentally obtained thresholds. Various laboratories abroad have obtained different threshold values, which brought uncertainties for both engineering applications and academic research. Our group has been working on identification of mechanisms responsible for this scatter for several years. Large amount of experimental data has been acquired in our laboratory. Extensive testing capacity in the combination with long-term experience of the group members in the field of fatigue crack propagation and

together with an expertise in fractography and advanced electron microscopy led to identification of the dominant crack closure mechanism influencing the threshold value – the oxide-induced crack closure. Special experimental equipment was designed to determine the dependence of the threshold value on air humidity. Air humidity was identified as a very strong factor influencing the level of crack closure. This knowledge was for the first time published in *Pokorný P., Vojtek T., Náhlík L., Hutař P. Crack closure in near-threshold fatigue crack propagation in railway axle steel EA4T. Engineering Fracture Mechanics 185 (2017) 2-19* (this output was propounded for the evaluation).

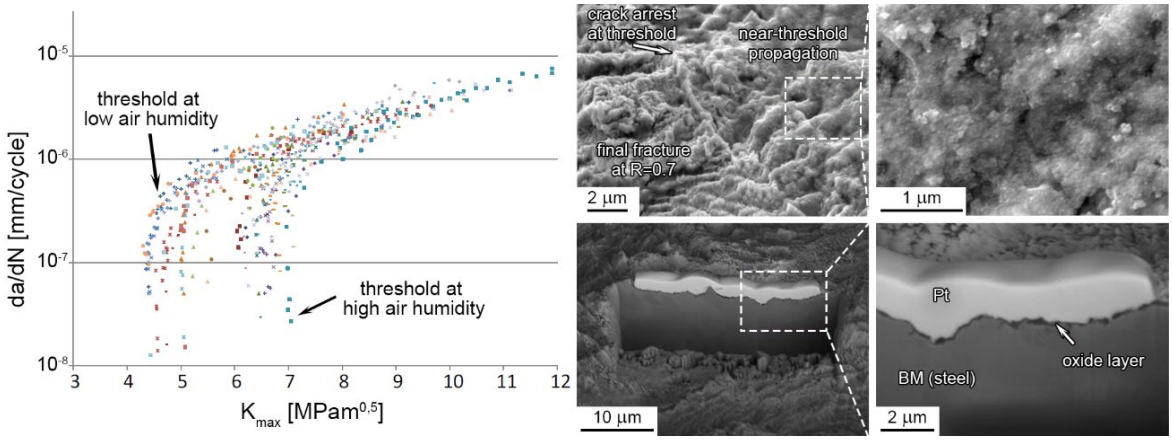


Fig. 3. Fatigue crack propagation threshold values for low and high air humidity. Scheme of the measurement of thickness of oxide layers using FIB technique.

Moreover, the absolute air humidity was identified as a parameter controlling the fatigue crack growth rate. This conclusion had not been formerly considered in the fatigue community and in the case of lifetime predictions of engineering components. The obtained knowledge has a good potential to explain differences in thresholds determined under various environmental conditions, see Fig.3. The oxide layer produced at fracture surfaces during near-threshold loading was measured in nanoscale (see photos in Fig. 3) and it revealed correlation with the air humidity and the level of crack closure. Based on this, predictions for resistance of engineering components can be improved according to environmental conditions. Our results were published in the paper *Vojtek T., Pokorný P., Kuběna I., Náhlík L., Fajkoš R., Hutař P. Quantitative dependence of oxide-induced crack closure on air humidity for railway axle steel. International Journal of Fatigue 123 (2019) 213-224* (this output was propounded for the evaluation). Presented results have increased an accuracy of the lifetime prediction of railway axles and contributed to the development of new railway axles BONAXLE® in Bonatrans Group a.s. The mentioned results were also selected and presented among the best results of the Czech Academy of Sciences at the 53rd meeting of the Academy Assembly.

Advanced numerical simulations of fatigue cracks propagation

Numerical prediction of fatigue cracks behaviour and simulation of cracks propagation in 3D structures is an important long-term research topic of our group. During the evaluated period two important aspects had been deeply studied; the

effect of free surface and the possibility of numerical modelling of plasticity induced crack closure.

Our knowledge about the effect of free surface on fatigue crack propagation was summarised in the paper *T. Oplt, P. Hutar, P. Pokorný, L. Náhlík, Z. Chlup, F. Berto, Effect of the free surface on the fatigue crack front curvature at high stress asymmetry, International Journal of Fatigue 118 (2019) 249–261* (this paper was propounded for the evaluation).

High stress asymmetry of cyclic loading eliminates the crack closure effect, which would otherwise influence the shape of the crack front. Thanks to this phenomenon, the effect of free surface could be separated and the formation of the crack front without any other influencing factor was studied. Two different methods for the determination of the crack front shape were used: a method based on a local value of the stress intensity factor and a method based on local value of the stress singularity exponent. Both can be effectively used to model the crack front shape. The real crack front was determined experimentally for two different materials (steel EA4T and aluminium alloy 7075), see Fig. 4. This work was an important step for the generalisation of linear elastic fracture mechanics to the 3D problems. It had improved accuracy of the numerical simulations of fatigue crack propagation. The next research step is going to be focused on low stress asymmetries, where the plasticity induced crack closure acts as an important factor influencing fatigue crack formation is plasticity induced crack closure.

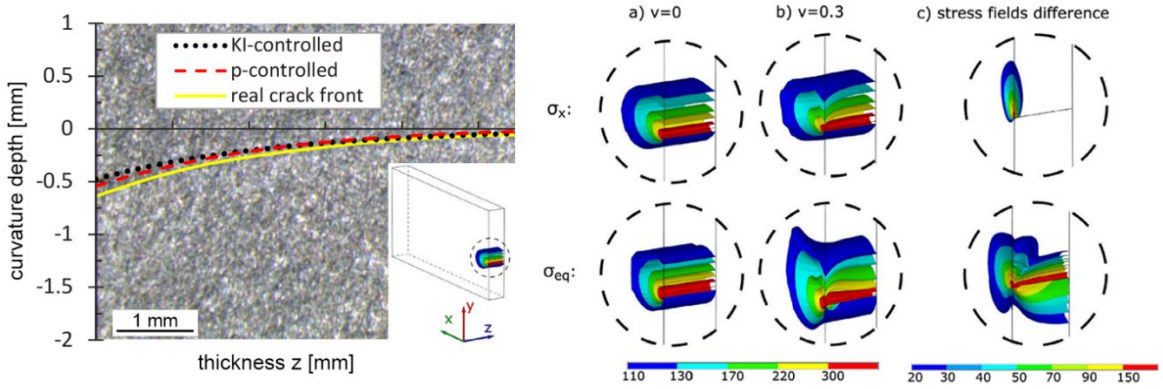


Fig. 4. Numerical predictions and real crack front shape of fatigue crack propagating in 3-point bending specimen (on the left). Numerical visualization of the stress field difference between different materials with different Poisson ratio (ν=0 and ν=0.3).

Appropriate modelling of the plasticity induced crack closure is necessary for the separation of the different closure mechanisms and can help to predict fatigue behaviour of materials (at least in the linear region of v-K curve). There is no general agreement among particular models and modelling strategies in literature. We summarized our knowledge on the plasticity induced crack closure and relevant literature data in the paper: *T. Oplt, M. Šebík, F. Berto, L. Náhlík, P. Pokorný, P. Hutař, Strategy of plasticity induced crack closure numerical evaluation, Theoretical and Applied Fracture Mechanics 102 (2019) 59–69* (this paper was propounded for the evaluation). The best modelling strategy was compared with our own experimental data (2D model was used) and a very good agreement was found. The knowledge of model preparation and perspective computational strategy gained on the 2D model was then applied to make a 3D model of the experimental specimen with a fatigue crack. The first results have been already published in the paper: *T.*

Oplt, P. Hutař, P. Pokorný, L. Náhlík, F. Berto, Numerical evaluation of plasticity induced crack closure in 3D structures, Procedia Structural Integrity 23 (2019) 101-106. The research was supported through the grant No. 18-03615S of the Czech Science Foundation and projects FV 40034 and FV 40327 of Ministry of Industry and Trade.

Fatigue and fatigue/creep damage for high temperature resistant materials

The investigation of nickel-based superalloys subjected to cyclic loading is a progressive topic within the High Cycle Fatigue group offering a range of challenges. The main directions of the research were i) new insight into the deformation mechanism responsible for crystallographic stage I cracking, ii) the effect of porosity on high cycle fatigue performance and iii) the combined creep-fatigue loading and its influence on fatigue life. A series of fatigue tests, at temperatures from 650 to 900 °C, revealed that fatigue life strongly depends on the active deformation mechanisms which are determined by dislocation mobility at particular test temperature.

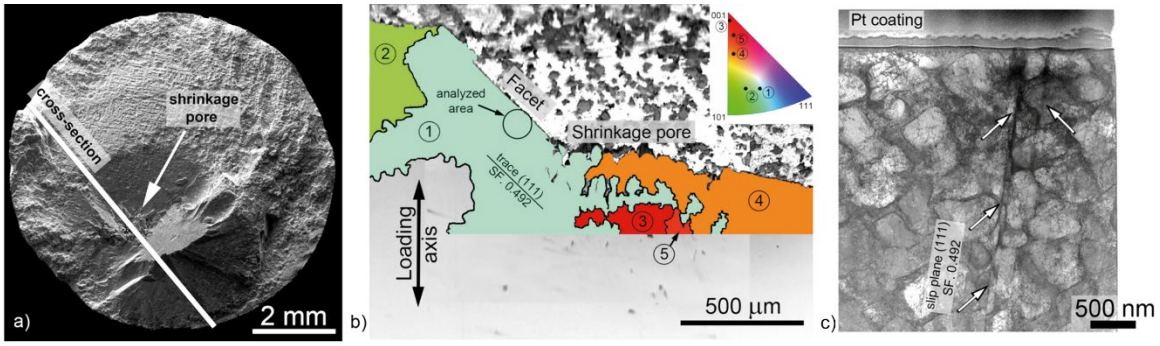


Fig. 5. Investigation of specimen cyclically loaded at 800 °C: (a) fracture surface with indicated plane of the cross-section for further analysis; (b) inverse pole figure map in vicinity of fatigue crack. Crystallographic facet of grain 1 was identified as (111) plane with high Schmid factor; (c) TEM image of extracted lamella with severe localization of plastic deformation into few slip planes.

In temperature range between 650 and 800 °C, dislocations activity is strongly restricted to suitable {111} type slip planes. This behaviour leads to fatigue crack initiation and propagation with distinct crystallographic character. With further temperature increase above 800°C dislocations mobility increases rapidly which promotes cross-slip and dislocation climb. As a consequence, dislocations can avoid precipitates more easily and the distinct crystallographic character of the crack growth is weakened. These findings were summarized in *M. Šmíd, V. Horník, P. Hutař, K. Hrbáček, L. Kunz, High cycle fatigue damage mechanisms of MAR-M 247 superalloy at high temperatures, Trans Indian Inst Met 69 (2016) 393–397* (this paper was propounded for the evaluation) and *Šmíd et al, Procedia Structural Integrity 2 (2016) 3018-3025*. Fig. 5 depicts detailed characterization of the stage I cracking. The described research activities have helped our industrial partner, PBS Velká Bíteš, to develop a precise casting procedure of selected superalloys with long-term mechanical properties optimized for a selected operational temperature range. The research activities were supported by Technology Agency of the Czech Republic and projects of Ministry of Industry and Trade. Project TA 04011525 (Research and Development of Precision Casting Technology of Radial Wheels of New Generation

Turbochargers and New Types of Gas Turbines Blades) of Technology Agency of the Czech Republic was selected as the best project of the agency in Business category for the year 2019. This result was selected and presented among best results of the Czech Academy of Sciences on the 55th meeting of the Academy Assembly.

Evaluation of long-term resistance of polymer materials

The research in this area is focused mainly on polyolefin materials for the production of polymer pipes used for gas and water distribution. Previous investigations and numerical modelling of plastic pipes damage with residual stress showed that the influence of residual stress on the lifetime of pipes is quite significant and that it is necessary to have a reasonably precise estimation of the residual stress distribution in a pipe for lifetime calculations based on fracture mechanics and finite element modelling. Using our own methodology based on measuring deformation of slit rings, the distribution of tangential residual stress (induced by the extrusion process) in a polyethylene (PE) pipe was investigated. An influence of axial residual stress component was also considered. The results were published in *J. Poduška, P. Hutař, J. Kučera, A. Frank, J. Sadílek, G. Pinter, L. Náhlík, Residual stress in polyethylene pipes, Polymer Testing 54 (2016) 288-295* (this paper was propounded for the evaluation) and provide a unique idea about the residual stress distribution in a PE pipes.

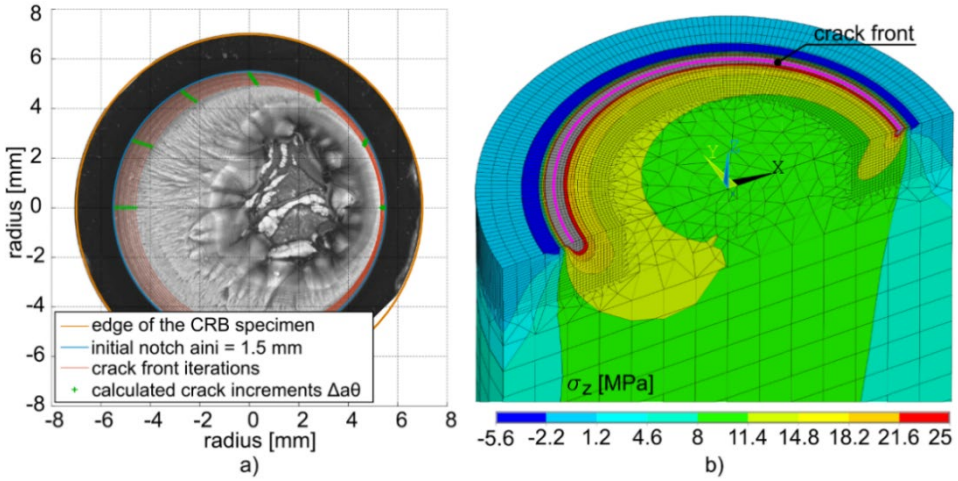


Fig. 6. a) comparison between crack fronts predicted numerically (red circles) and real fracture surface; b) detailed stress distribution in CRB specimen during simulation of the crack propagation.

Apart from the actual residual stress distributions, a simplification of the complicated experimental method was proposed and tested. The previous, more accurate method needed 13 ring specimens made of the pipe. The simplified method only needs 1 ring specimen to produce a reasonable estimation of the tangential residual stress distribution. The simplification was based on similarities observed between the residual stress distributions obtained on different pipes. This method is now being successfully applied in various companies e.g. UNIPETROL RPA, s.r.o., Borouge Pte Ltd.

LEFM-based method of lifetime calculation is largely dependent of material parameters that describe the crack growth rate in a given material. The crack round bar tests, which are often used to obtain these properties, are fast and effective, but

asymmetrical crack growth often occurs in the specimens. The influence of this asymmetry (caused by residual stresses in the specimens) on the material data was investigated based on numerical simulation of the experimental CRB test, see Fig. 6. Details were published in *J. Poduška, P. Hutař, A. Frank, J. Kučera, J. Sadílek, G. Pinter, L. Náhlík, Numerical simulations of cracked round bar test: Effect of residual stresses and crack asymmetry, Engineering Fracture Mechanics 203 (2018) 18–31* (this paper was propounded for the evaluation). These investigations showed that even though asymmetry is present in the specimens, the material parameters obtained on such specimens are not corrupted and are still applicable. This result has a significant practical impact on the applicability of CRB tests in engineering practice.
Based on our knowledge of the polymer pipe behaviour and long-term performance, our investigation has recently been focused on pipes produced from recycled material. An initial design proposition of multilayer co-extruded pipe with high resistance against slow crack propagation was published in the paper J*. Poduška, P. Dlhý, P. Hutař, A. Frank, J. Kučera, J. Sadílek, L. Náhlík, Design of plastic pipes considering content of recycled material, Procedia Structural Integrity 23 (2019) 293-298*. Our research was also awarded by an invited presentation to the 6th International Plastic Pipe Conference in China. The mentioned research activities were supported mainly by the PCCL project (founded by the COMMET program in Austria) and the MESTEC project (founded by the Technological Agency of the Czech Republic).

Development of stability criteria of singular stress concentrators

The research was focused on the toughening mechanism and crack behaviour in multi-layered ceramic composites. The alumina-zirconia laminate was used as a model composite material. Crack behaviour ahead of ATZ/AMZ (ATZ - alumina with tetragonal zirconia, AMZ - alumina with monoclinic zirconia) interface, at the interface and behind the interface was studied. The crack propagation in the compressive layer responsible for higher apparent fracture toughness of the composite was investigated by finite element modelling. A criterion based on strain energy density factor was used to estimate the crack behaviour in the layers and to evaluate the crack path through the laminate. It was shown that the crack, after passing through the first material interface between tensile and compressive layer, retards due to strong residual stresses, and is sharply deflected. An additional external load is necessary for further crack propagation at that stage. Local stress distribution allows following crack bifurcation. Further crack propagation in the compressive layer is more or less parallel to the material interface. The stages leading to the stepwise crack propagation through ceramic laminate body (see Fig. 7) were described in detail. The finite element method together with own analytical controlling routines were used for extensive numerical simulations.
The research contributed to a better understanding of the role of residual stresses and damage of strongly bonded ceramic laminates and their toughening mechanism. Main result – detailed description of failure scenario - was published in *Náhlík, L., Štegnerová, K., Máša, B., Hutař, P. A failure scenario of ceramic laminates with strong interfaces (2016) Engineering Fracture Mechanics, 167, pp. 56-67* (this paper was propounded for the evaluation).
The research result was ranked among the most significant results of IPM in 2016. Our PhD student cooperating on this research, Kateřina Štegnerová, was awarded the prize of Czech Society for Mechanics during 31st Conference Computational

Mechanics 2015 for the contribution Crack propagation study in layered alumina-zirconia ceramic composite. The research was supported through grant no. 15-09347S of the Czech Science Foundation.

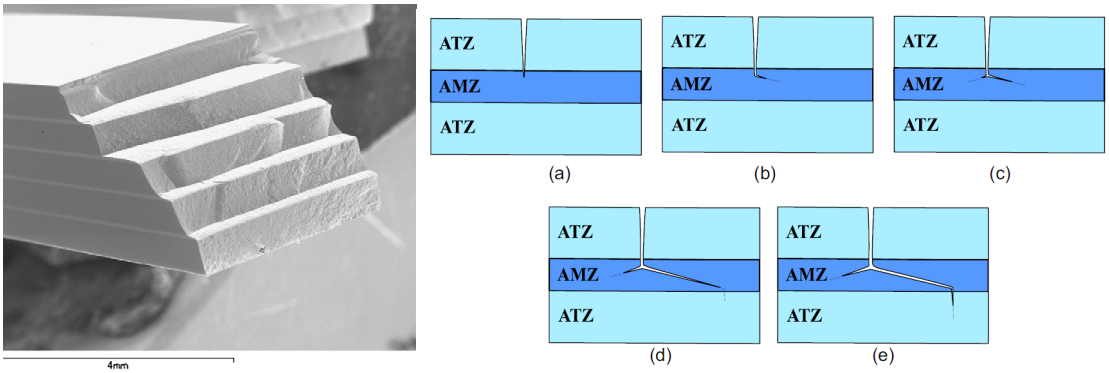


Fig. 7. Step-like fracture of an alumina–zirconia multi-layered ceramic under flexural loading and individual stages of crack propagation through laminate layers (on the right: result of numerical prediction)

Within the study of complex approaches leading to the assessment of General Stress Singular Concentrators (GSSC) we got to understand critical parameters (controlling quantities) describing the process of crack initiation. Thus, we have realised that controlling quantities can be compared directly with their critical values. This approach avoided the complex semi-analytical description of the stress distribution and also the complicated multi-parameter generalized descriptions of the failure initiation conditions. The criterion of Critical Quantity (CQ), formed on the basis of these investigations, is an easy and engineering-friendly criterion that belongs to a family of finite fracture mechanics criteria. The CQ criterion employs numerical results of common finite element models of GSSC and simple analytical relations for critical values of evaluated quantities. The CQ criterion works with complete and accurate stress distribution (gained from numerical solution), therefore the CQ criterion is very precise end easy to use, see details in *J. Klusák, O. Krepl, T. Profant, An easy and engineering stability criterion of general singular stress concentrators, Theoretical and Applied Fracture Mechanics 104 (2019) 102341* (this paper was propounded for the evaluation). The research was supported through grant no. 17-01589S of the Czech Science Foundation.

Fatigue and fracture of silica-based composites

The research was focused on fatigue and fracture performance of modern building materials. Concrete structures and their structural elements are typically subjected to the combination of bending and shear load. The Brazilian discs, a type of experimental specimen with a central notch, are usually used to investigate this type of loading in concrete. The post peak crack initiation behaviour in the Brazilian disc with central notch under mix mode loading conditions was successfully numerically predicted using concrete damaged plasticity (CDP) model, see e.g. in Fig.8. The linear elastic fracture mechanics criteria were used for comparison. A new procedure for estimation of defect initiation in concrete was suggested in the papers: *P. Miarka, S. Seitl, W. De Corte, Notch tip displacements of the concrete Brazilian disc test with central notch analysed by the concrete damaged plasticity model, Theoretical and*

Applied Fracture Mechanics 102 (2019) 122–150 and *S. Seitl, P. Miarka, V. Bílek, The mixed-mode fracture resistance of C 50/60 and its suitability for use in precast elements as determined by the Brazilian disc test and three-point bending specimens, Theoretical and Applied Fracture Mechanics 97 (2018) 108–119* (both papers were propounded for the evaluation). The results were obtained in close cooperation with precast elements manufacturer ŽPSV s.r.o., Technical University of Ostrava, University of Ghent and University of Koszalin.

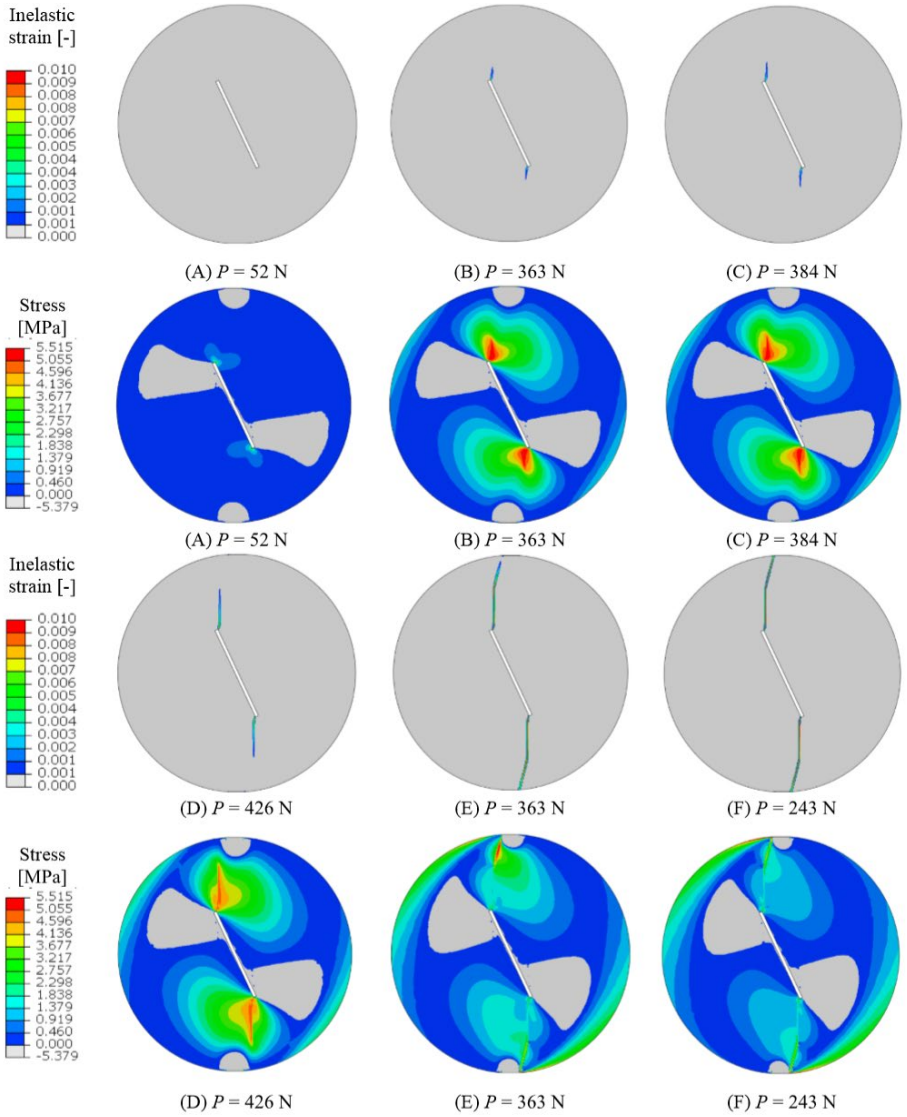


Fig. 8. Numerical simulation of the crack type damage of Brazilian disc using CDP material model.

Research activity and characterization of the main scientific results

The effort of the LCF group continually concentrates both to the basic studies and cooperation with industrially important subjects. The provision of new equipment, namely thermomechanical fatigue testing system recommended during last evaluation and the provision of biaxial testing system allowing the testing also at high temperatures enables extending our interest in new fields. The IPM succeeded in the provision of high-quality AFM microscope (producer Nenovision) which can be placed into the chamber of SEM enabling thus correlative measurements AFM vs. SEM which is useful for measurement of kinetics of cyclic slip band formation. The group continues in intense international cooperation and is active in proposing research projects. In the following the main results obtained in the evaluated period are briefly introduced.

- **Fatigue crack growth rate of short cracks**

We have carefully investigated the fatigue crack growth rate of short cracks in the condition of low cycle fatigue, i.e. in the situation where the small scale yielding assumption is not valid. It is known that in such condition cracks grow much faster than expected according to the standard approach based on the range of the stress intensity factor ΔK_a. We investigated five materials: steels and Al alloy with wide range of strength and with different crystallographic lattice (see cyclic stress-strain curves in Fig. 1). The crack growth rate in cycling with various strain amplitudes was measured. An example of crack paths in Al 6082 is shown in Fig. 2. We were searching for the parameter governing the short crack growth rate. The J-integral was calculated numerically by the finite element (FEM) simulations. These calculations were done in very close cooperation with our colleagues from the High Cycle Fatigue group. It was found out that all measured data from the five very different materials collapse on a single master curve if the plastic part of J-integral is used (Fig. 3). It means that the plastic energy introduced into the material in the vicinity of the crack tip is the decisive parameter for the crack growth of short cracks. This is an original finding.

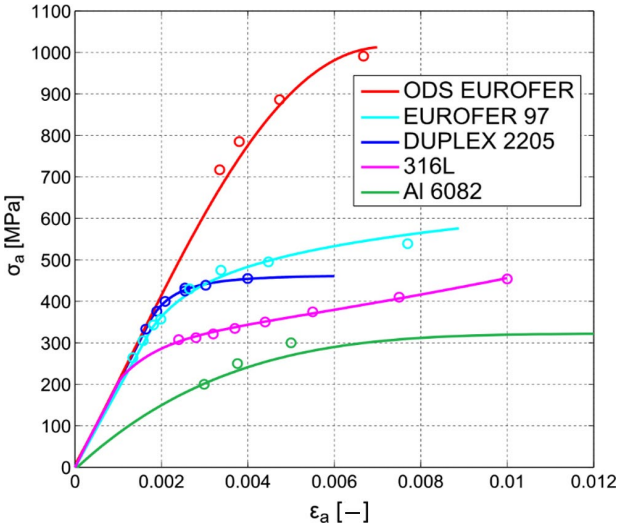


Fig. 1 Stress amplitude at the half life in strain-control cycling of five materials (see the inset).

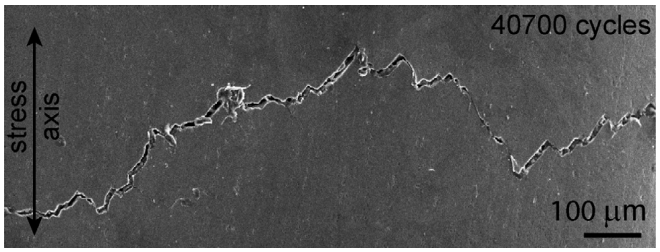


Fig. 2 Example of crack path in aluminum alloy Al 6082.

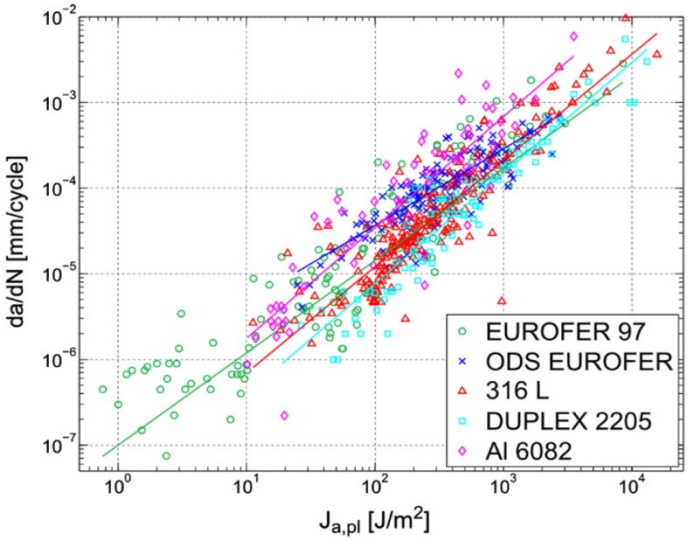


Fig. 3 Crack growth rate data measured on the five materials follow the unique master curve if plotted as a function of the amplitude of the plastic part of J-integral.

- **Properties of Ni-based superalloy with thermal barrier coating**

LCF group focused its attention on advanced multilayered complex thermal barrier coatings (TBCs) resistant to environmental degradation and providing thermal insulation of superalloys. Fig. 4 shows an example of an as-sprayed coating consisting of a metallic CoNiCrAlY bond coat (BC) and a ceramic top coat (TC). The TC is a mixture of a near eutectic alumina-silica-zirconia (Eucor) and conventional yttria stabilized zirconia (YSZ) in the ratio 50/50. TBCs were designed and deposited on the surface of superalloys by air plasma spraying in collaboration with CEITEC, Brno University of Technology. Superalloys MAR-M247 and Inconel 713LC were supplied by the PBS Velká Bíteš, a.s. company. Low cycle fatigue tests of TBC-coated and uncoated superalloys were carried out in a symmetrical push-pull cycle in total strain control at 900 °C. Cyclic stress-strain response and fatigue life curves were obtained. Microstructural investigations revealed the effect of TBCs on fatigue damage mechanisms of superalloys. That findings help explain the differences in the fatigue behavior of coated and uncoated materials. E.g. the TBC coating consisted of a CoNiCrAlY BC and an alumina-silica-zirconia TC has a beneficial effect on the fatigue life of Inconel 713LC at 900 °C in the Coffin-Manson representation – see Fig. 5. The crack density of initiated cracks in the surface layer is up to 15 times higher in the coated material, which decreases the localization of cyclic plastic strain.

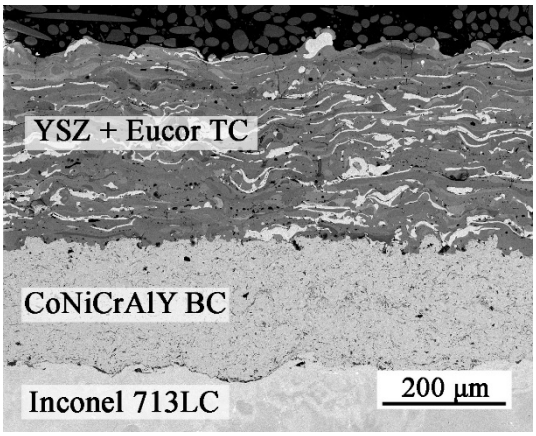


Fig. 4 BSE image of cross-section of an as-sprayed specimen. Microstructure and thicknesses of complex TBC consisted of CoNiCrAlY bond coat (BC) and Eucor/YSZ top coat (TC) deposited on superalloy Inconel 713LC.

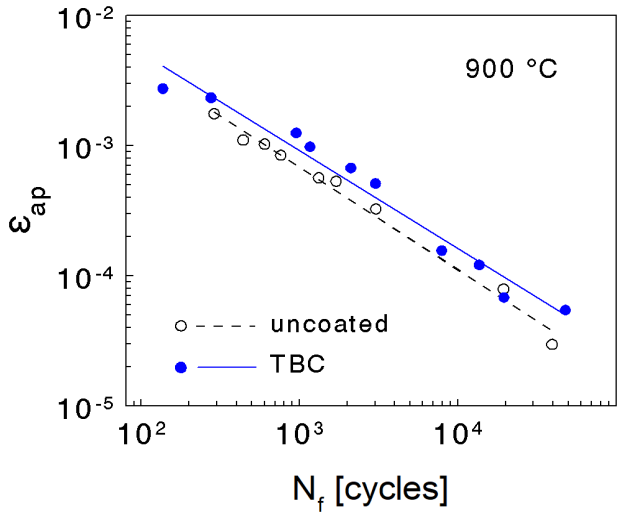


Fig. 5 Coffin-Manson diagram of the TBC coated and uncoated Inconel 713LC at 900 °C.

- **Development of TiAl alloy with increased ductility**

TiAl alloys are lightweight alloys capable to work at high temperatures. Their main limitation is the low ductility at room temperature. We designed 5 different chemical compositions with varying content of Nb, Mo and C as alloying elements. The materials were fabricated for our purposes at GfE, Nürnberg, Germany. We performed a wide range of experiments on these materials. At first, their phase compositions were detected by electron microscopy and in-situ neutron diffraction. It allowed creation of sections of phase diagram (Fig. 6). Tensile, compressive and fatigue properties were measured at RT, 750 °C and 800 °C. Microstructural changes and deformation mechanisms were studied as well. Subsequently, the specific heat treatment leading to nanolamellar microstructure of the alloys was applied (Fig. 7). Mechanical tests and microstructural characterization were done again. We showed that the nano-scale lamellar material alloyed with 7 at.% of Nb and 2 at.% of Mo shows higher strength and improved ductility both at room temperature and 750°C.

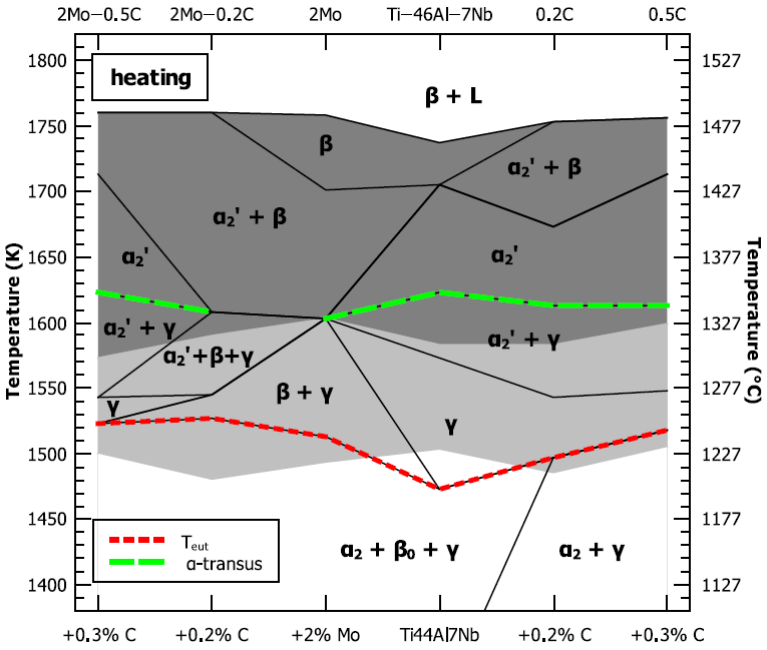


Fig. 6 Phase diagram for TiAl 7 at.% Nb alloyed with Mo and/or C.

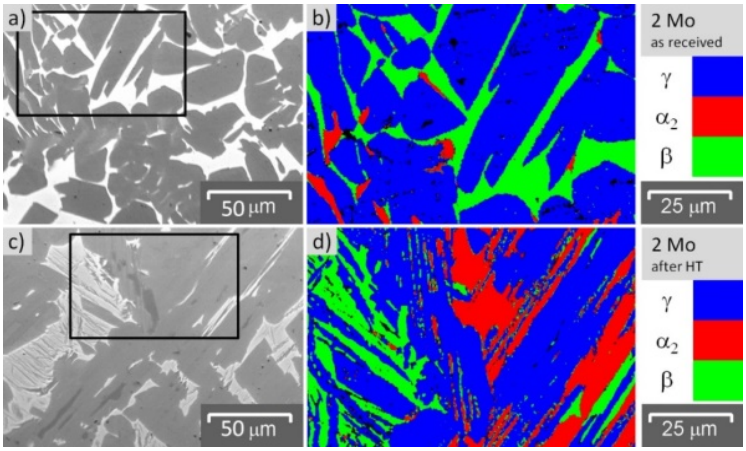


Fig. 7 Microstructure of TiAl 7Nb 2Mo alloy before (a,b) and after (c,d) the heat treatment.

- **Reason for excellent high temperature behaviour of Sanicro 25**

Sanicro 25 superaustenitic steel is one of the alloys newly developed primarily for use in next generation advanced ultra-supercritical (A-USC) coal-fired boilers. We have studied both room and high (700 °C) fatigue behavior of this steel, namely cyclic stress-strain response, internal structure and damage mechanisms. Using high resolution TEM and computer simulation it was found that cross-slip and climb, enhanced at high temperature, lead to a remarkably homogeneous dislocation structure with high dislocation density. Moreover, dense volume dispersion of coherent Cu-rich nanoparticles and rising number of incoherent NbC carbides (Fig. 8), supported also by the presence of W in solid solution result in significant retardation of dislocation motion and the cyclic strength of the steel at high temperature outperforms that at room temperature.
Damage mechanisms were followed at room temperature and in isothermal cycling and thermomechanical cycling with and without dwells. FIB cuts observed in SEM

allowed revealing the individual damage mechanisms, i.e. extrusion/intrusion source of early fatigue cracks at low temperature and oxide extrusions and oxide intrusions at grain boundaries at elevated temperature (Fig. 9).

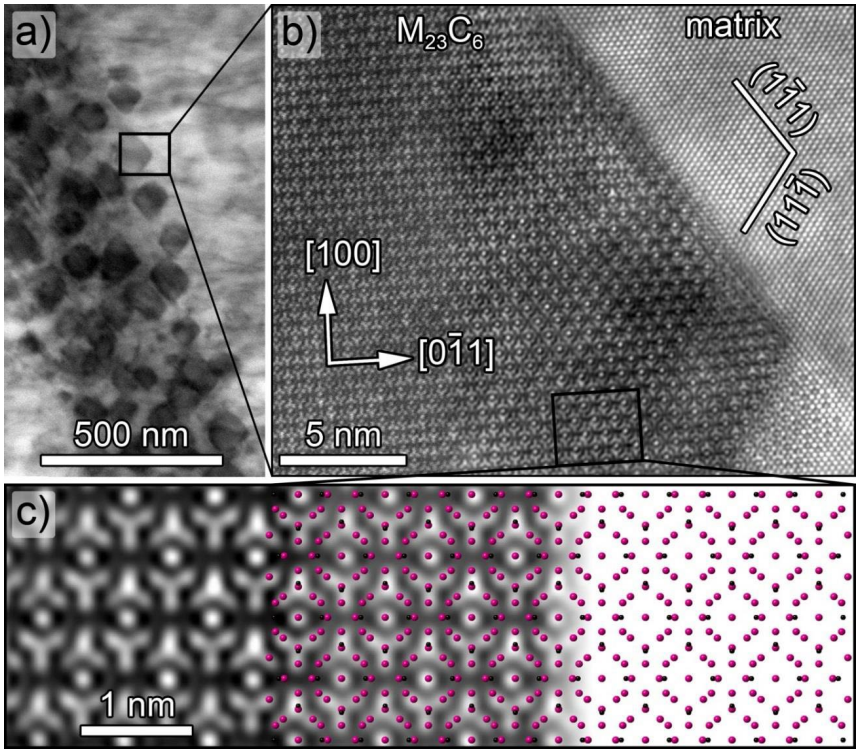


Fig. 8 High resolution TEM of strengthening carbide particles and comparison with simulated atom position with the micrograph.

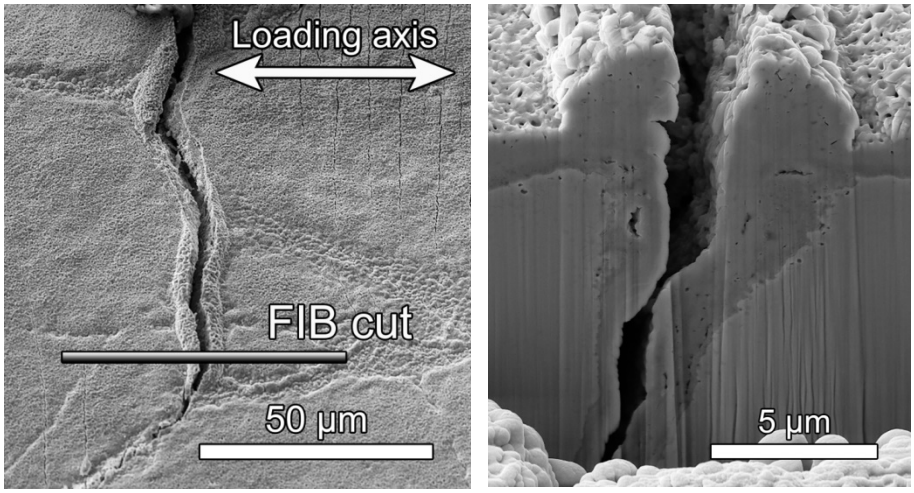


Fig. 9 Cracked grain boundary in cycling Sanicro 25 with dwells at high temperature.

- **Multiaxial fatigue of austenitic stainless steel**

The provision of biaxial MTS 809 testing system enabled us to perform cyclic tests in combined axial and torsional loading. As the first material we have chosen the 316L steel which is widely used in various industrial applications and we gathered large knowledge about its behaviour in tension/compression mode in the past. Since most of the studies concerning the multiaxial loading in the literature have been focused on the fatigue life, we have concentrated on the research of the damage mechanisms and the comparison of these mechanisms in axial, torsional, in-phase multiaxial and out-of-phase multiaxial modes. It was found and explained that formation of α'-martensite is accelerated in torsion (Fig. 10). Cyclic strengthening effects were discussed (Fig. 11).

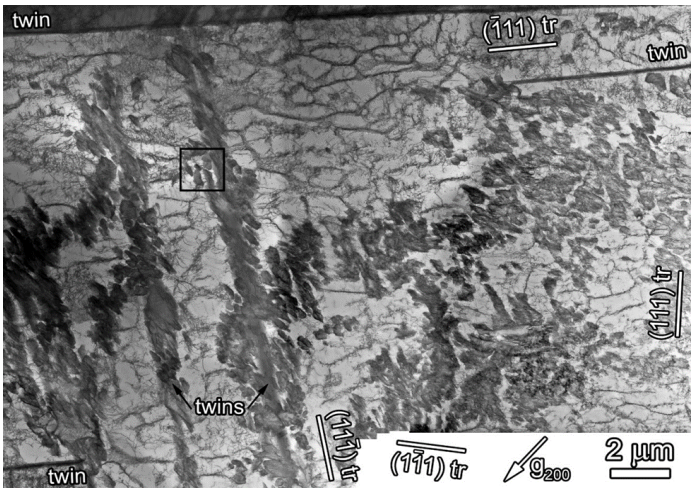


Fig. 10 Microstructure after the fatigue failure of specimen cycled in torsion with equivalent strain amplitude of 0.35 %. High number of α'-martensitic islands nucleated preferentially on deformation twins.

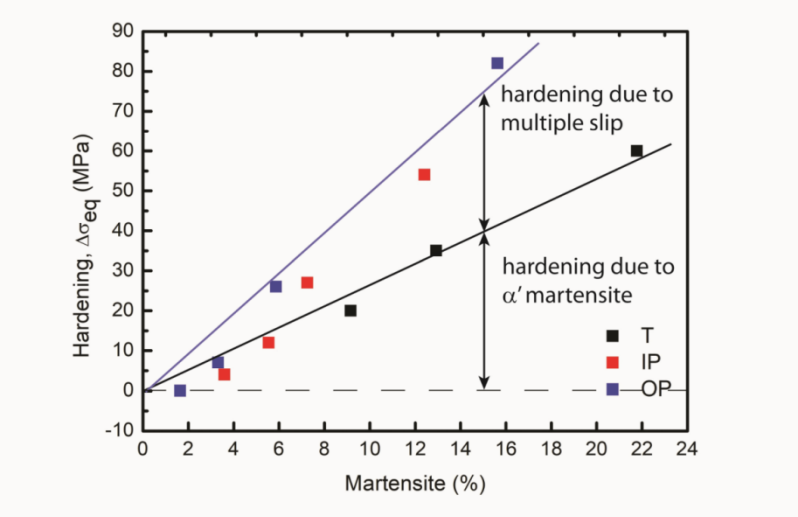


Fig. 11 Two main cyclic strengthening mechanisms were identified and quantitatively assessed: strengthening due to multiple slip of dislocations and strengthening due to martensite formation.

- **Mechanical properties of new UFG 301LN austenitic stainless steel**

Austenitic stainless steels are widely used in many industrial applications due to excellent corrosion resistance, weldability and good mechanical properties. Their main drawback is however relatively low yield stress and strength. An alternative and very promising technique of grain refinement and thus strengthening in case metastable stainless AISI 301LN steel represents the so called reversion annealing after prior cold deformation. Introduction of two different annealing conditions during special thermomechanical treatment resulted in considerable grain refinement down to 1.4 μm (Fig. 12) and thus increase of tensile characteristics while ductility was preserved (Fig. 13). A prosperous effect of grain refinement on fatigue life is also clear – see Fig. 13. We performed detail study on the effect of grain refinement on cyclic plasticity and relevant damage mechanisms. Microstructural changes and destabilization of originally fully austenitic structure were characterized in detail by magnetic measurements and various microscopic techniques (HR TEM, EBSD and ECCI). Special color etching technique was adopted to reveal distribution of deformation induced martensite across the full thickness of fatigued sheets (Fig. 14). This enabled to explain the specific shape of hysteresis loops (hysteresis loop constriction) and characteristic variations in mean stresses during early stage of cyclic straining.

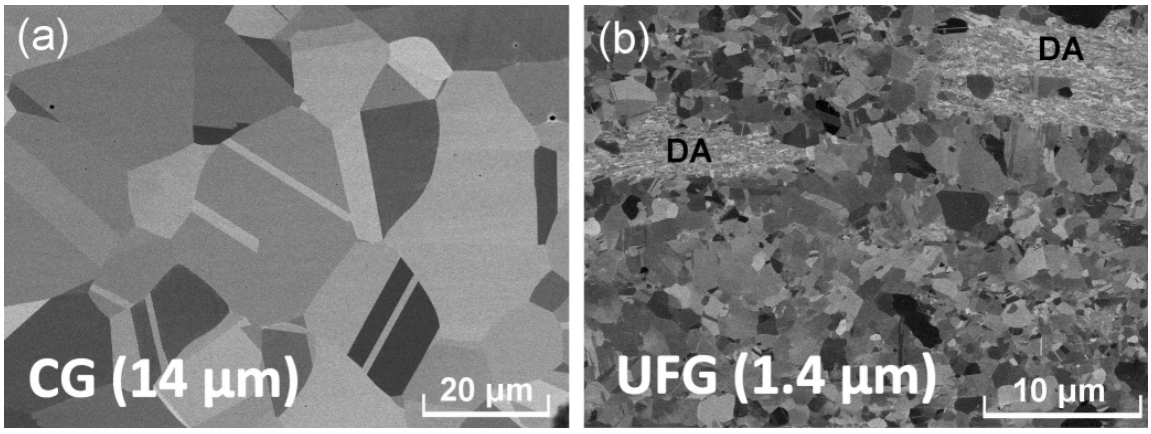


Fig. 12 Austenitic AISI 301LN stainless steel with different grain size as obtained by reversion annealing after prior cold deformation.

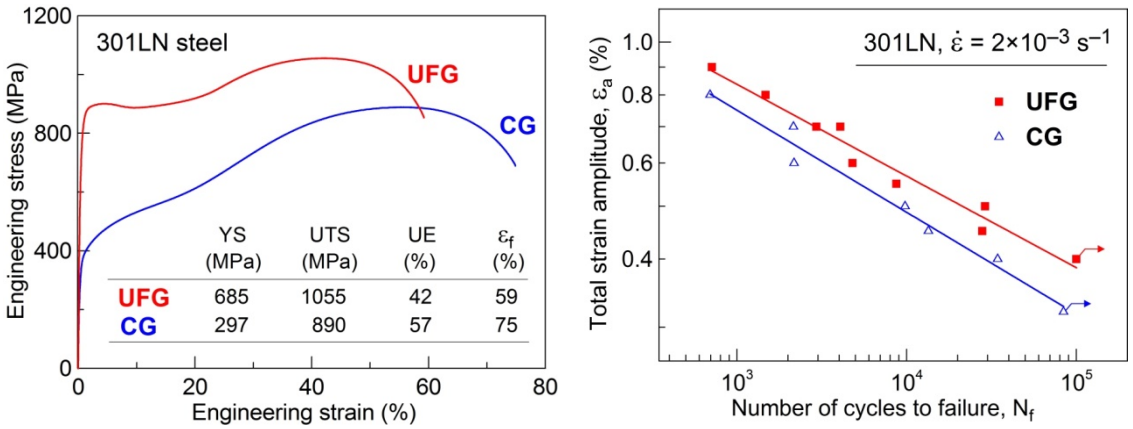


Fig. 13 Effect of grain refinement on tensile characteristics and low-cycle fatigue life.

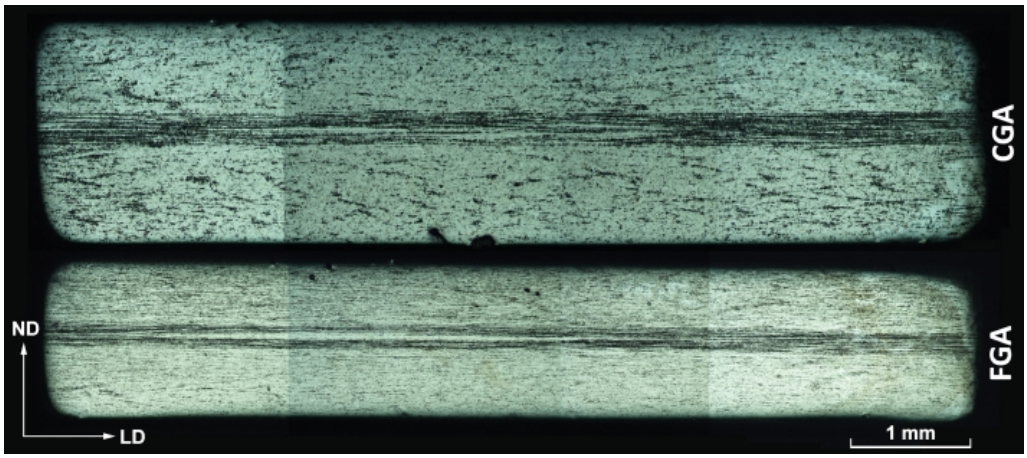


Fig. 14 Distribution of deformation induced α'-martensite (dark features) across the sheet full-thickness of CG and FG/UFG 301LN steel as revealed by color etching.

Research activity and characterisation of the main scientific results

In the field of Mechanical Behaviour and Fracture Resistance Evaluation of Ceramics and Composites Including their Design and Processing.

(i) $Al_2O_3/ZrO_2/BaTiO_3$ laminates - design, preparation and fracture assessment.

Comprehensive set of knowledge in the field of ceramic-based laminates prepared by various methods was achieved by the Group during the last years. The Group utilised this knowledge base to design novel layered architecture which can protect brittle piezo ceramics against cracking and harsh environments. The leadership of our team can be seen mainly in the understanding processes during the preparation of the laminates by electrophoretic deposition (EPD) and in understanding to fracture behaviour of the layered structures with internal stresses. Also, knowledge of the EPD kinetics allowed to prepare theoretically well founded design of individual layers as demonstrated on protective layered system for $BaTiO_3$ energy harvester in **Fig. 1**.

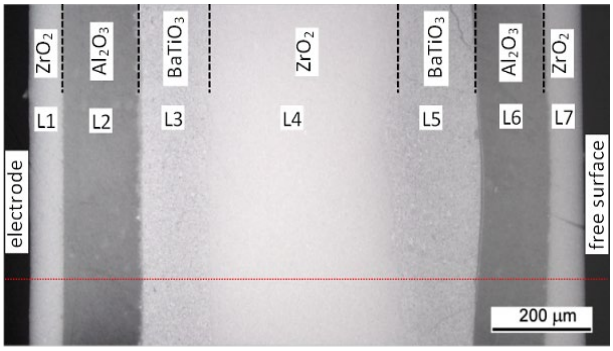


Fig. 1: *Demonstration of layered architecture for the protection of $BaTiO_3$.*

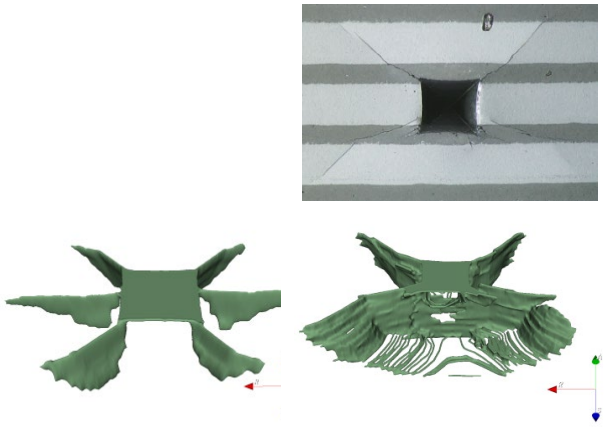


Fig. 2: *Examples of reconstructed crack patterns for different layer indented.*

The results obtained cover all issues from the design and modelling of the layered structures trough their precise preparation exploiting knowledge of processing steps up to the stresses development description in the context of final fracture behaviour and resulting piezoelectric characteristics.

Understanding to fundamental aspects of multicomponent layered structures with strongly bonded layers including internal stresses allows optimising the individual layers design to achieve enhanced properties. The key task was to transfer knowledge from the two-component system to multi-component one. The crack trajectory in real 3D layered structures was key input in this case; reconstructed indentation cracks patterns can be seen in **Fig. 2**. The effort was also targeted to the sintering behaviour; reactivity between layers can affect the interphase bonding strength and the development of desired internal stresses. Controlled reaction can be expected to form in-situ strongly bonded layers possessing additional functionality, e.g. serving as an electrode for example. The findings were obtained thanks to co-operations, in sintering issues with the Brno University of Technology, in modelling and fracture behaviour analyses with the Montanuniversität Leoben and local behaviour evaluation with the Institute of Materials Research SAS Kosice.

References:

[1] Tofel P., Machů Z., Chlup Z., Hadraba H. et al.: Novel layered architecture based on Al2O3/ZrO2/BaTiO3 for SMART piezo ceramic electromechanical converters. The European Physical Journal Special Topics 228 (2019) 1575-1588, IF = 1.837.

[2] Chlup Z., Drdlík D., Fides M. et al.: Properties of BaTiO3/Al2O3 Laminate Structure by Nanoindentation. Procedia Structural Integrity 23 (2019) 499-504.

[3] Hadraba H., Chlup Z., Drdlík D., Šiška F.: Mechanical properties and fracture behaviour of Al2O3/ZrO2/BaTiO3 laminate, J. Eur. Ceram. Soc., IF = 4.029.

Related projects:

"**The development and characterisation of layered structures**", 17-08153S, Czech Science Foundation (2017-2019).

"GrInHy: **Green Industrial Hydrogen**", H2020 FCH-02.4-2015, grant agreement No. 700300 (2016-2019).

(ii) Fibre-reinforced composites: fundamentals, development and assessment.

The development and characterisation of reliable ceramic materials is one of the strong topics of the Group. The research effort was concentrated onto understanding of fundamental processes taking place during pyrolysis as an important processing step in hybrid composites preparation. The first significant set of composites under investigation was materials based on polysiloxane precursors with the focus on solvent-free resins where reinforcement was kept the same. Recently, the temperature stability of the optimised composite was determined.

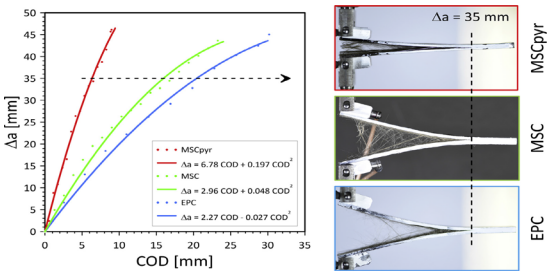


Fig. 3: *Comparison of fracture – crack development for various composites.*

The fracture response in the in-plane direction is crucial for composite stiffness. Therefore the effects of matrix type and pyrolysis level on interfacial composite chemistry and properties were extensively investigated. **Fig. 3** demonstrates the crack interaction with the basalt fibre to matrix interface, as example.

The same polysiloxane matrix reinforced by other types of fibres was evaluated. It was noticed that basalt fibres interaction with polysiloxane matrix is unique possessing good application potential independently of unexpected formation of iron precipitates inside the basalt fibres, **Fig. 4**.

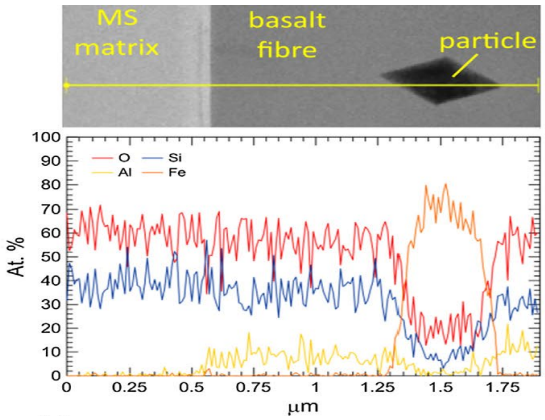


Fig. 4: *Example of iron precipitate in basalt fibre formed at 650°C.*

The activities in this field are example of an attempt to prepare economically attractive and maximally green high temperature resistant material with outstanding fracture resistance in the wide range of temperatures. The materials preparation was enabled by cooperation with other CAS institutes (IRSM and IMC).

References:

[4] Černý M., Chlup Z., Strachota A., Halasová M. et al.: 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) 6237-47, IF = 3.45.

[5] Halasová M., Černý M., Strachota A., Chlup Z., Dlouhý I.: Fracture response of SiOC-based, composites on dynamic loading. J. Composite Mater. 50 (2016) 1547-1554, IF = 1.865.

[6] Halasová M., Kuběna I., Roupcová P., Černý M., Strachota A., Chlup Z.: Iron precipitation in basalt fibres embedded in partially pyrolysed matrix. Composites Part A 123 (2019) 286-292, IF = 6.282.

[7] Chlup Z., Černý M., Kácha P., Hadraba H., Strachota A.: Fracture resistance of partially pyrolysed polysiloxane preceramic polymer matrix composites reinforced by unidirectional basalt fibres. J. Eur. Ceram. Soc., corrected, proof in press, IF = 4.029.

Related projects:

"**Fundamental aspects of partial pyrolysis of hybrid composites with polysiloxane matrix precursors**", 17-12546S, Czech Sci. Foundation, (2017-2019).

(iii) Deconstruction of cellulosic fibers for advanced composites materials.

Cellulose nanocrystals (CNCs) are rod-like units consisting of individual cellulose molecules stabilized by a strong and complex network of hydrogen bonds. When derived from plant sources such as cotton and wood pulp, CNCs typically exhibit lengths varying between 100 and 350 nm, widths of about 10–20 nm and thicknesses usually about 3–5 nm. Cellulose-based materials have gained increasing attention due to their outstanding mechanical and functional properties such as nontoxicity, biodegradability, large surface area, hydrophilicity and remarkably high elastic modulus. All these properties predestine CNCs as reinforcing phase in composites.

A process which leads to the decomposition of microfibrillated cellulose (MFC) into its elementary nanosized constituents - CNCs was developed. It consisted of treating of MFC in highly concentrated, low-silica sodium silicate solutions. This endeavour represents the first attempt of CNCs extraction in alkaline environment. The alignment of CNCs is regarded as the key how to fully exploit the outstanding mechanical and functional properties of CNCs. The sustainable, low-energy and green character of the process and the materials involved has to be also underlined.

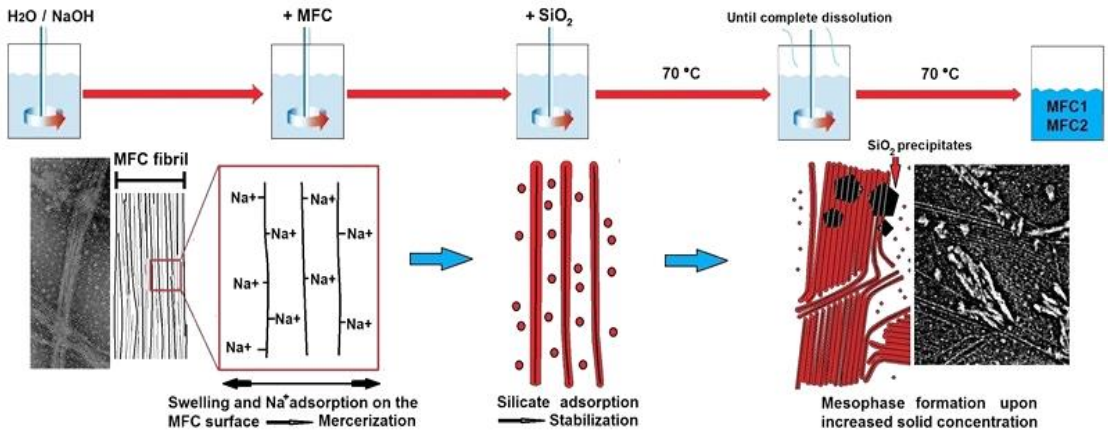


Fig. 5: *Characterisation of processing steps leading to cellulose nanofibers preparation.*

The sample preparation and SEM observations were done in IPM. Microscopic work (DIC and TEM) and ^{29}Si NMR analysis were carried out at CEITEC. In ARCCHIP CentreTelč, FTIR and XRD analysis were performed.

References:

[8] Bertolla L., Dlouhý I., Bartoničková E. et al.: Deconstruction of microfibrillated cellulose into nanocrystalline cellulose rods and mesogenic phase formation in concentrated sodium silicate solutions. Cellulose 26(7) (2018) 4325-4344, IF = 4.379.

Related projects:

"CoACH: **Advanced glasses, Composites and Ceramics for High growth Industries**", MSCA-ITN-2014-ETN, grant agreement No. 642557 (2015-2019).

"GlaCERCo: **Glass and Ceramic Composites for High Technology Applications**" – FP7-PEOPLE-2010-ITN, grant agreement No. 264526 (2011-2015).

(iv) Room temperature hydro-pressure densification.

An innovative method [9] of powder densification, inspired by the bio-silicification in diatoms, was suggested and verified for the first time by pressing a suspension of amorphous silica microbeads/nanoparticles in water and caustic solution, though the technique itself is extendable to many types of materials. The suspension is pressed in uniaxial hydraulic press by means of a die for pellets production. After only 5 minutes of pressing and half hour of drying, a densified and translucent sample is obtained. Densification of the powder is achieved thanks to dissolution enhanced by the application of pressure, and second-phase precipitation among the compacted particles (**Fig. 5**). The last stage induces a hardening over time and water evaporation. Sample is mechanically stable; relative density always higher than 99%.

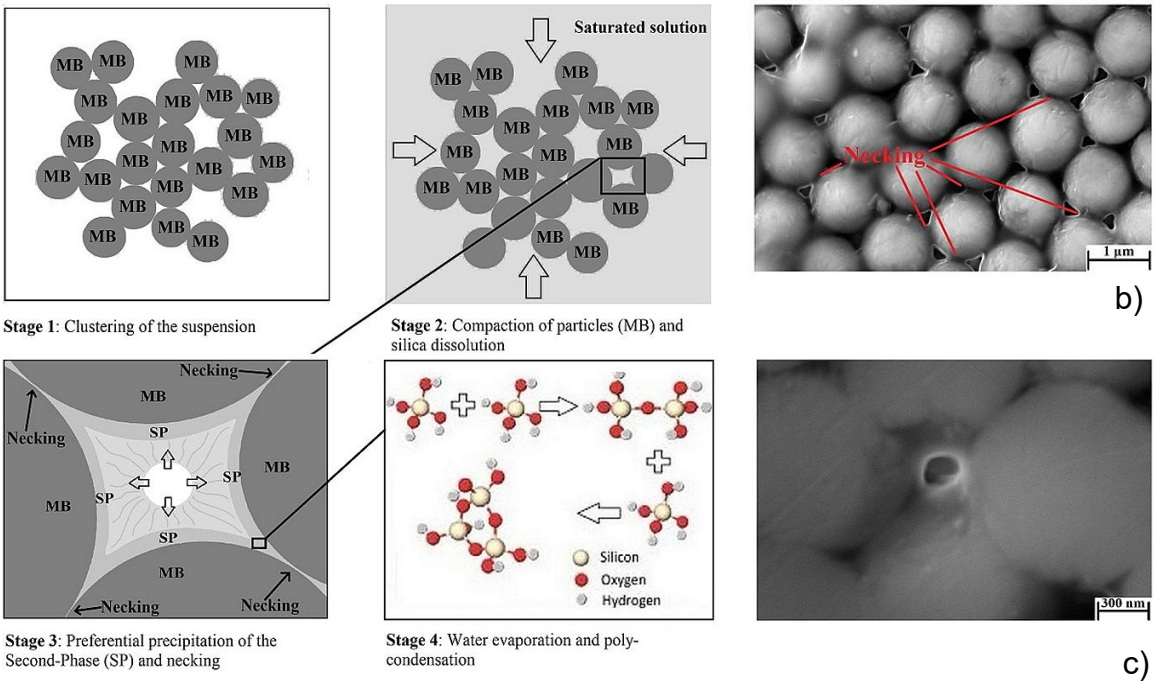


Fig. 5: *Model of HP densification a) Model description: (1) Clustering of micro beads (MB), (2) Compaction and dissolution, (3) Preferential precipitation of the second-phase (SP), (4) Poly-condensation. b) SEM image of a consolidated by hydro-pressure sample, necking formation. c) A close-up of a pore, by which the model was built.*

The hydro-pressure (HP) densification process was also applied to nanosilica, showing translucency and mechanical properties comparable to sintered silica

ceramics and biosintered products. It was demonstrated that HP method is effective for other materials densification; applications can be extended also for production of thermal-insulating material and solid state battery electrodes and electrolytes.

References:

[9] Taveri G., Dlouhy I., Grasso S.: Densification process of inorganic powders by hydrostatic pressure and rig to this, Patent application, PV 2018-420, 2018.

[10] Taveri G., Grasso S., Gucci F., Dlouhy I.: Bio-Inspired Hydro-Pressure Consolidation of Silica. Advanced Functional Mater. 28 (2018) 1805794, IF = 15.621.

Related projects:

”CoACH: **Advanced glasses, Composites and Ceramics for High growth Industries**”, EU Training Network, MSCA-ITN-2014-ETN, No. 642557 (2015-2019).

In the field of Mechanical Behaviour and Fracture Resistance of Metallic Materials Related to their Design and Processing.

(v) Low-activation steels – new types of strengthening phase.

Oxide dispersion strengthening (ODS) method is based on incorporation of small amount (0.25 wt.%) of homogeneously distributed temperature stable Y-Ti oxides to the microstructure serving as obstacles for dislocations and pinning grain boundaries. First, we developed novel method utilising internal oxidation of oxidizable elements contained in the steel solid solution. This method was used for fabrication of Y-Ti oxide strengthened ODS steels with mechanical properties comparable to those prepared by direct Y_2O_3 and Ti incorporation during mechanical alloying. Also, ODS steels strengthened by Al_2O_3 nano-oxides were prepared; the Al_2O_3 particles cannot be added to the steel composition in the form of pure oxides because their high hardness disallows erosion of the oxide particles during mechanical alloying and preparation of fine oxide dispersion inside the steel [11].

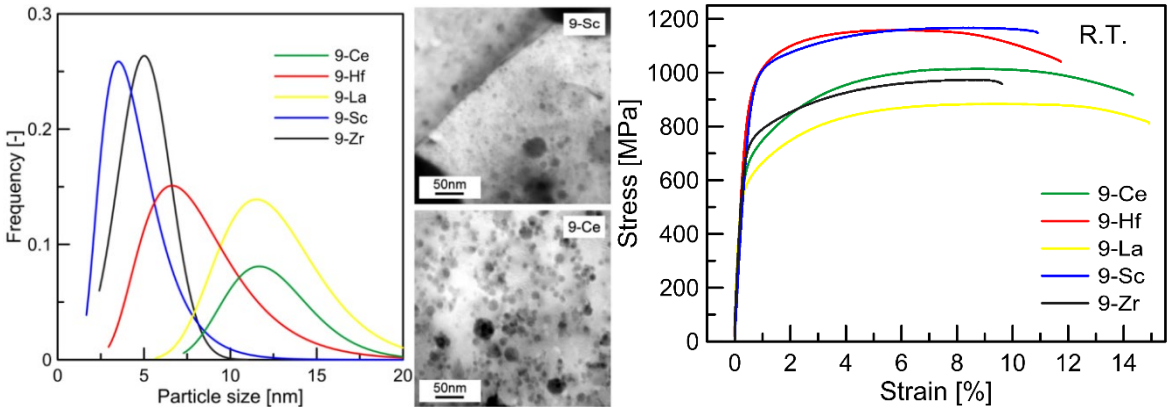


Fig. 6: *Particle size distribution functions of 9Cr ODS steels with different types of oxide dispersoids, microstructure with fine dispersion (9-Sc) and coarse dispersion (9-Ce) of oxide particles and influence of oxide size on final mechanical properties of the ODS steels.*

Next, the internal oxidation method was utilized for preparation of new ODS steels strengthened by complex Y-X oxides, where X stands for group IIIB elements (La, Sc) and group IVB elements (Ce, Hf, Zr). The complex oxide dispersion showed superior thermal stability compared to Y-Ti oxides usually used for ODS steels preparation. Furthermore, the complex oxide dispersion leads to development of denser distribution of finer oxide particles and further improvement of the dispersion strengthening effect. By analysis of dispersion contribution to steel strengthening the

key role of oxide interparticle spacing was found. By chemical composition variation, the oxide size and average grain size of steels were altered in the ranges of 5-13 nm and 0.6-1.7 μm, respectively. The yield strength of the prepared steels varied between 550 MPa and 950 MPa depending on the doping element [12] in this way.

Finally, the usability of prepared ODS ferritic steels for first circuit parts of lead cooled fast fission reactors was proven. The high-chromium ODS steels with self-developing oxide scale ability were pre-strained and subjected to melted Pb-Bi. Developing and rigidity of oxide scale as well as embrittlement of the steels subjected to liquid metal were evaluated. High resistance against liquid metal embrittlement was proved [13].

References:

[11] Hadraba H., Husák R., Stratil L., Šiška F., Chlup Z., Puchý V., Michalička J.: Survey of oxide candidate for advanced 9%, 14% and 17%Cr ODS steels for fusion applications. Fusion Engineering and Design 124 (2017) 1028-1032, IF = 1.457.

[12] Husák R., Hadraba H., Chlup Z., Heczko M., Kruml T., Puchý V.: ODS EUROFER Steel Strengthened by Y-(Ce, Hf, La, Sc, and Zr) Complex Oxides. Metals 9 (2019) 1148, IF = 2.371.

[13] Hojná A., Hadraba H., Di Gabriele F., Husák R.: Behaviour of pre-stressed T91 and ODS steels exposed to liquid lead-bismuth eutectic. Corrosion Science 131 (2018) 264-277, IF = 6.355.

Related projects:

Czech Science Foundation project No. 14-25246S "**Advanced ODS steels for applications in heavy liquid metals environment**s", 2014-2016.

Czech Science Foundation project No. 20-20873S "**Development of liquid metal resistant ODS steels for exploitation in new systems of nuclear fission and fusion**" (2020-2022).

(vi) High entropy alloys – strengthening by oxide dispersion.

High entropy alloys (HEA) represents new concept of alloys based on multiple principal elements in roughly equal proportions. Thanks to their excellent mechanical properties at both low and high temperatures, the HEAs represent a promising alternative material for vacuum vessel and for the plasma-facing components of fusion reactors. The major limitation for current state-of-the-art, i.e. ferritic-martensitic steels, is the reduction in strength at higher temperatures. Incorporation of nano-sized oxide particles to the HEA microstructure can overcome the yield strength and ultimate tensile strength drop at high temperatures and enhance creep resistance of the HEAs. We have developed new oxide dispersion strengthened one-phase CoCrFeMnNi HEA through mechanical alloying. The incorporation of nano-oxides into the HEA microstructure resulted in the effective grain boundary and dislocation pinning effect. The grain refinement of the HEA and presence of nano-oxides had a positive effect on strength and creep resistance of the HEA. The results gives first outcomes of HEA microstructural engineering through grain size influencing [14].

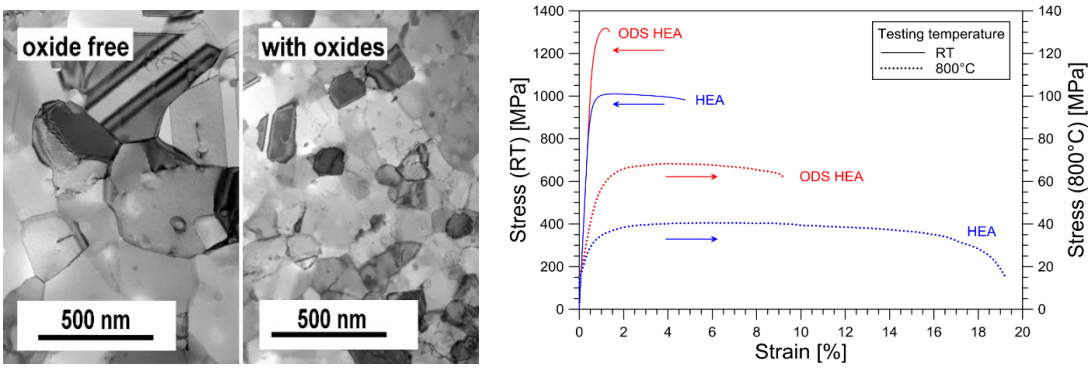


Fig. 7: *Microstructure of HEA with and without oxide dispersion and influence of oxide dispersion on final mechanical properties of the CoCrFeMnNi HEA.*

Flexural static and fatigue tests of the CoCrFeNiMn HEA prepared by powder metallurgy route with incorporating oxide dispersion demonstrated higher fatigue endurance limit of HEA with smaller grain size. Microcracks formed in large grains of the microstructure due to the twins acted as the fatigue crack initiation sites for both material states. The effective grain boundary pinning by oxide dispersion represents a key factor influencing fatigue response of the HEA [15].

The CoCrFeNiMn alloy develops porous structure underneath the oxide scale during high temperature oxidation due to outward diffusion of Mn which serves as main surface oxide-forming element. Incorporation of the Y-Ti oxides decreases thickness of the porous layer in the bulk through limitation of the Mn diffusivity [16].

References:

[14] Hadraba H., Chlup Z., Dlouhý A., Dobeš F., Roupcová P., Vilemová M., Matějíček J.: Oxide dispersion strengthened CoCrFeNiMn high-entropy alloy. Materials Science and Engineering A 689 (2017) 252-256, IF = 4.081.

[15] Chlup Z., Fintová S., Hadraba H., Kuběna I., Vilémová M., Matějíček J.: Fatigue behaviour and crack initiation in CoCrFeNiMn high-entropy alloy processed by powder metallurgy. Metals 9 (2019) 1110, IF = 2.371.

[16] Vilémová M., Illková K., Csáki Š., Lukáč F., Hadraba H., Matějíček J., Chlup Z., Klečka J.: Thermal and oxidation behaviour of CoCrFeMnNi alloy with and without yttrium oxide particle dispersion. Journal of Materials Engineering and Performance 28 (2019) 5850-5859, IF = 1.67.

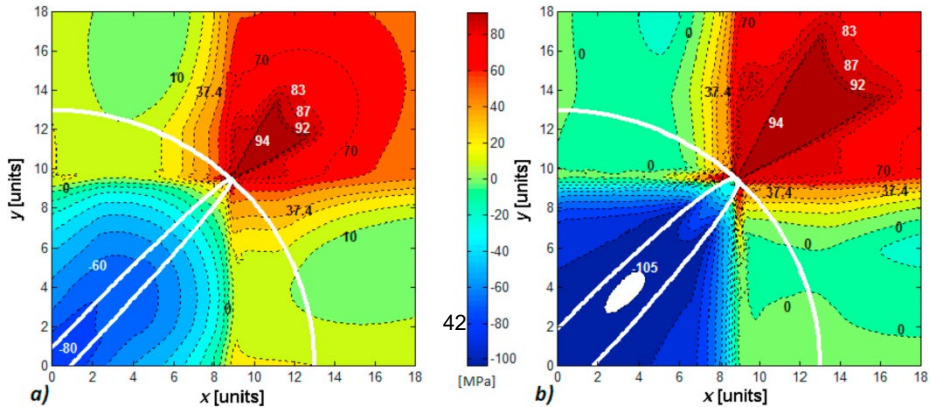
Projects:

Czech Science Foundation project No. 17-23964S “**Dispersion strengthened high entropy alloys for extreme conditions**” (2017-2019).

Czech Science Foundation project No. 20-20873S “**Development of Liquid Metal Resistant ODS Steels for exploitation in new systems of nuclear fission and fusion**” (2020-2022).

(vii) Twin-microstructure interactions in HCP materials

Twinning is a deformation mechanism that is based on the instant collective shuffling of atoms. It is important in hexagonal close-packed (HCP) metals due to the lack of



slip systems at certain crystallographic orientations. Activation of twinning causes anisotropy in macroscopic behaviour of the metal which worsens its use in applications. The twinning process has three distinct stages: initiation, propagation and growth. Therefore, it appears more complex than dislocation slip and not yet fully understood. Twin initiation is a stochastic problem that can be explained on atomistic level, while propagation and growth can be analysed within framework of continuum mechanics. Propagation and growth are driven by external stress relaxation by the stress-induced during twinning. The level of induced stress depends on the twin dimensions and elasto-plastic behaviour of the surrounding material. The elasto-plastic properties of a metal/alloy can be changed by solute atoms and/or presence of second phase precipitates which can also restrain twin dimensions.

Fig. 8: *Contours of shear stress in the twin plane for 55 μm grain size and the applied load of 37.4 MPa (as experimentally observed). Two aspect ratios are shown a) 0.05; b) 0.1.*

The effects of elasto-plastic properties and precipitates on twinning are studied in our Group using numerical simulations based on the finite element method combined with crystal plasticity. The numerical analysis shows that twin acts like an inclusion inside microstructure and induces extensive plastic slip in its vicinity. Therefore, an increase of the critical resolved shear stress (CRSS) for slip suppresses extension of plasticity and consequently also twin propagation and growth. Mutual interaction of twins has also a significant influence on their behaviour. They induce stress concentration in front of their tips which helps to initiate, propagate and grow other twins. Therefore, twins very often create band-like structures. Precipitates play two roles with respect to twinning. They increase slip CRSS which affects twinning and they create obstacles for twin propagation and growth. The initial size of the twin is limited by the precipitate spacing and the stress field in front of the twin is shielded by precipitates. Therefore, thick precipitates with close spacing suppress twinning.

The experiments were performed in collaboration with the Faculty of Mathematics and Physics, Charles University and the Deakin University in Australia.

References:

[16] Šiška F., Stratil L., Čížek J., Ghaderi A., Barnett M.: Numerical analysis of twin thickening process in magnesium alloys. Acta Materialia 124 (2017) 9-16, IF = 7.293.

[17] Guo T., Šiška F., Barnett M.R.: Distinguishing between slip and twinning during nanoindentation of magnesium alloy AZ31. Scripta Mat. 110(2016) 10-13, IF = 4.559.

Related projects:

"**Multiscale analysis of twin-microstructure interactions in HCP metals and alloys**", Czech Science Foundation, project Nr. 18-07140S (2018-2020).

Project proposal to Czech Science Foundation **„Controlled twinning in HCP alloys"** (2021-2023).

(viii) Plasticity of metallic architectured materials.

The architectured materials create new class of materials that are characteristic by their specifically ordered internal structure. This ordering creates an extra degree of freedom that is used for an improvement of functionality such as tuning properties for a specific application or creating a completely new function. The typical examples of the architectured materials are truss structures that combine solid material and free space. Truss structures provide a high ratio between stiffness/strength and density which is an order of magnitude higher than the one for foam structures. However, this is true only for perfect undamaged geometries. Our research has been focused on the stiffness and strength of the pyramidal truss core composites with damaged

trusses. Stiffness of these structures deteriorates rapidly even for small damage because the compromised trusses become loaded in bending rather than compression. Bending stiffness is an order of magnitude lower which results in the decrease of mechanical properties of the damaged truss core towards the foam structures after only 8% of plastic deformation [19].

The second type of architectured materials combines two or more solid materials that can be ordered in geometrical patterns that create a synergic effect and the combination show better properties than its constituents. This was numerically verified in the analysis of seven different 2D geometries containing two elastic-perfect plastic materials. The first effect of these geometries is that they modify Poisson's ratio which can be useful in applications like sheet forming. Another effect connected with plastic behaviour is called geometrically induced strain hardening. Some geometries are able to produce hardening even if both constituents are perfect plastic materials. This property can be useful for the absorption of strain energy. The highest induced hardening is provided for geometries that combine stretch dominated members with cantilever beam-like members loaded in bending. Specifically ordered material combination can be used also to mimic properties of other monolithic material. A combination of aluminium with 30% of iron has a similar density and elastic properties as titanium [20].

This research was performed in collaboration with the Faculty of Mechanical Engineering, Brno University of Technology, the Institute of Thermomechanics CAS and the Deakin University in Australia, respectively.

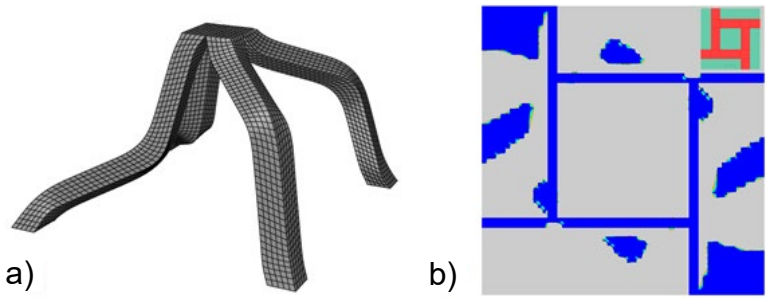


Fig. 9: *a) Buckled shape of truss core with relative density of 3.2% damaged to deformation of 15%. b) Distribution of the plastic strain during uniaxial tension of the 2D geometry consisting of two perfectly plastic materials (matrix: E = 69 GPa, yield = 200 MPa, geometry: E = 210 GPa, yield = 800 MPa). The macroscopic strain is 0.42%. The grey areas are in plastic state while blue ones are elastic.*

References:

[19] Siska F.,Tawfeeq A.F.,Dlouhy I, Barnett M.R.: Stiffness and strength degradation of damaged truss core composites. Comp. Struct. 125 (2015), 287-294, IF = 4.975.

[20] Siska F., Cizek J., Seiner H.,Dlouhy I.: Numerical analysis of geometrically induced hardening in architectured materials, Comp. Struct. 233 (2019), IF = 4.975.

Related projects:

"**Architectured metallic materials for cold spray kinetization**", Czech Science Foundation, project Nr. 17-13573 (2017-2019).

"**Architectured materials designed for additive manufacturing**", ESIF, EU Operational Programme Research, Development and Education (2018-2022).

(ix) Small specimen test technique, transferability of fracture toughness data.

Small specimen test technique (SSTT) uses subsized test specimens to evaluate mechanical and fracture properties. This approach is significant in the cases of

limited amount of test material during a process of material development in laboratory scale, local material characterization and/or assessment of in-service components. The methodology of test results interpretation and correction for specimen size and crack tip constraint loss effect caused by test specimen miniaturization are developed by our Group. SSTT and transferability of fracture toughness parameters from laboratory sample to real component require both the experimental evaluation and the numerical analysis of crack tip stress – strain state by finite element method.

The SSTT and scale up approaches were applied for assessment of mechanical and fracture behaviour in several case studies. (i) Fracture toughness of A533B Cl.1 steel which is a reference material of IAEA for the studies of reactor pressure vessels steel embrittlement was evaluated. It was shown that miniature three-point-bend specimens (TBP) of 0.12T size, **Fig. 10a**, were able to provide close estimation of the reference temperature of the master curve as specimens of larger sizes 0.2 – 0.5T [21]. (ii) Miniature tensile and fracture toughness specimens were used for assessment of mechanical and fracture behaviour of ODS steels with different composition of strengthening phase produced by the Group at laboratory scale. It was shown that the strengthening phase of fine oxides decreased fracture toughness proportionally to hardening of the ODS steels [22]. (iii) In the field of SSTT methodology and size effect/constraint loss correction a simplified mechanical model enabling correction of crack resistance curves of TPB specimens was developed, **Fig. 10b**. This semi-analytical model together with simple experimental input of specimen's fracture energies proposes fast and effective solution for compensation of the size and constraint loss effect using only minimum amount of test material. (iv) More precise/advanced solution of transferability proposes micromechanical approach based on numerical modelling of the particular fracture micromechanism. This approach was developed and used for modelling of ductile fracture of pipeline steel material by micromechanical GTN model in collaboration with Centre des Matériaux, Mines ParisTech in France [23].

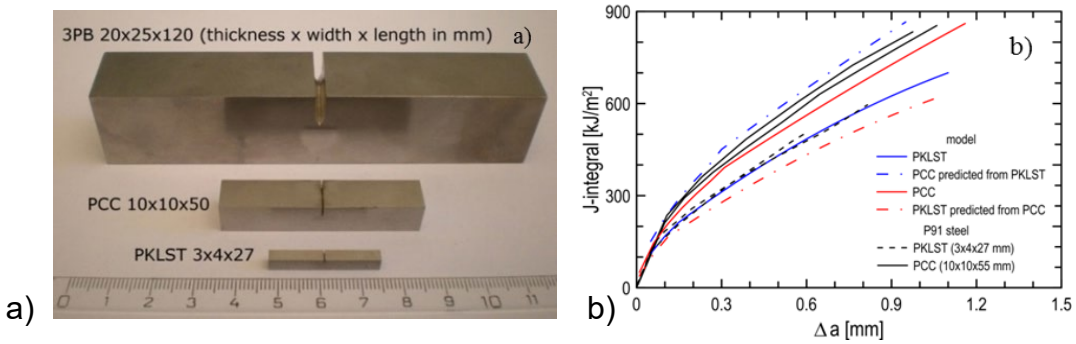


Fig 10: *a) Miniaturization of three-point-bend test specimen for fracture toughness determination; b) Prediction of J-R curve based on simplified mechanical model.*

References:

[21] Stratil L., Šiška F., Dlouhý I., Serrano M., The Application of Miniaturized Three-Point-Bend Specimens for Determination of the Reference Temperature of A533 Cl.1 Steel. J. Press. Vess. Tech. 139 (2017) 041410. IF = 0.924.

[22] Stratil L., Šiška F., Hadraba H. et al.: Fracture behaviour of the ODS steels prepared by internal oxidation. Fus. Eng. Des. 124 (2017) 1108-1111. IF = 1.457.

[23] Stratil L., Dlouhý I., Madi Y., Besson J.: Experiments and modelling of ductile fracture behaviour and fracture toughness of low-alloy C-Mn steel. 22nd European Conf. on Fracture, Belgrade, 2018.

Related projects:

”**Transferability issues in ductile to brittle transition and ductile regime**”, The Czech Academy of Sciences project No. MSM100411601” (2016-2018).

″INNOFAT: **Innovative approach to improve fatigue performance of automotive components aiming at CO2 emissions reduction**″, RFCS-02-2016 -747266 (2017-2020).

Research activity and characterisation of the main scientific results

As was mentioned above, the research interests of the SPT group cover mainly three fields of the material science devoted to the development of new materials and improvement of their properties by complex studies of mutual relations between thermodynamic and diffusion properties of multicomponent systems and their structure. The researchers in the group used their experience to propose projects which suitably combine at least two of above mentioned fields.

Phase Diagram Modelling and Thermodynamic Database Development

The experimental and theoretical phase diagram modelling and development of thermodynamic databases is one of intensively studied topics by the SPT team. This is one of key programs of the group and both existing studies and new programs were carried out in the years 2015-2019.

The combination of the *CALPHAD* method and ab initio calculations was used for the modelling of the particle size influence on the thermodynamic properties and phase equilibria of more complex systems. Nanoalloys are subject of intensive interest among researchers and the extent of experimental and theoretical studies of nanoalloys is growing fast in many fields, from theory and simulation to synthesis, analysis, property measurements and applications. Similarly as in the case of bulk materials, the knowledge of the thermodynamic properties and phase equilibria in dependence on particle size is crucial for the ability to predict the nanomaterials properties and to tailor them for a given purpose.

The *CALPHAD* semiempirical method represents approach to the modelling of size-dependent phase diagrams and thermodynamic properties of nanosystems, working on the scale from thousands of atoms to the “classical” bulk systems. The crucial information, necessary for such modelling, is the value of surface stress (tension) which allows us to calculate the surface energy contributions to the overall Gibbs energy. The availability of experimental data is important for calculation of such contributions by the *CALPHAD* method and there is considerable lack of such data. Experimentally accessible data are usually the values of surface tension for elemental or binary liquids and the surface tension for binary alloys for liquid and solid solutions can be deduced from the experimental values. Therefore, the previous development in this field concentrated on the modelling of size dependent thermodynamic properties for simple binary systems with complete solubility between pure elements or with simple invariant reaction.

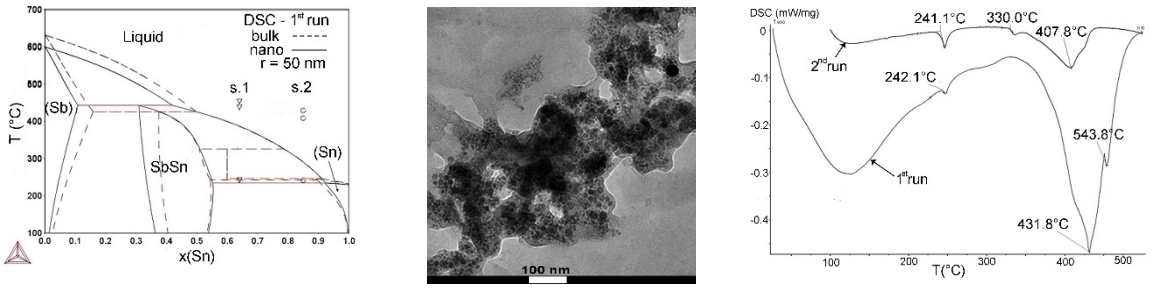
The possibility to model phase diagrams of more complex binary or even ternary systems is of course very important. The critical lack of experimental surface stress data cannot be solved by experiments especially for the intermetallic phases, but the members of the research team proposed the use of ab initio methods for the calculation of surface energy and surface stress of intermetallic phases. The original method for the modelling of the surface stress of nonstoichiometric phases was also presented. The basic principles of this method, combining in a novel way the *CALPHAD* and ab initio methods were presented at important international conferences and published in leading scientific journals e.g. [1,2].

The calculations describing the influence of the particle size on the thermodynamic properties and invariant reactions were compared with the experimental results in the scope of joint project with prof. J. Pinkas from Masaryk University. Our team carried out all theoretical modelling, the work on the characterization of nanoparticles by means of HRTEM and the DSC measurements of samples.

Several systems, namely binary Ni-Sn, Sb-Sn, Ag-Ni, and ternary system Ni-Sb-Sn were published and/or presented at the conferences in the years 2015-2019. The program still continues and further systems are currently studied. The experimental and theoretical description of Ag-Ni system was already published in 2020, the experimental programs for Bi-Ni and Bi-Sn systems is still running.

The most interesting result stemming from the theoretical modelling is the prediction about possible destabilization of some phases with decreasing diameter of particles. This phenomenon was observed for the Sb-Sn and Bi-Ni systems. It was also experimentally observed for the former system, but as it is not easy to prepare reliable nanoparticles with requested composition and size, we cannot claim full confirmation of the predictions. The experimental program for the Bi-Ni system is not yet finished, so question of the prediction reliability is not yet resolved.

Some results are shown in Fig. 1. It is clear from the phase diagram Fig. 1a, that the Sb_2Sn_3 intermetallic phase does not exist at least below 50 nm radius of nanoparticle and the DSC signals for the 1st run indicates no presence of the Sb_2Sn_3 phase (no signal around 330 °C). The DSC curves for the 1st and 2nd run are in the Fig. 1c and it is clear that after the 1st heating cycle, the nanoparticles coagulated and the 2nd heating cycle exhibits again the signals corresponding to the equilibrium phase diagram.

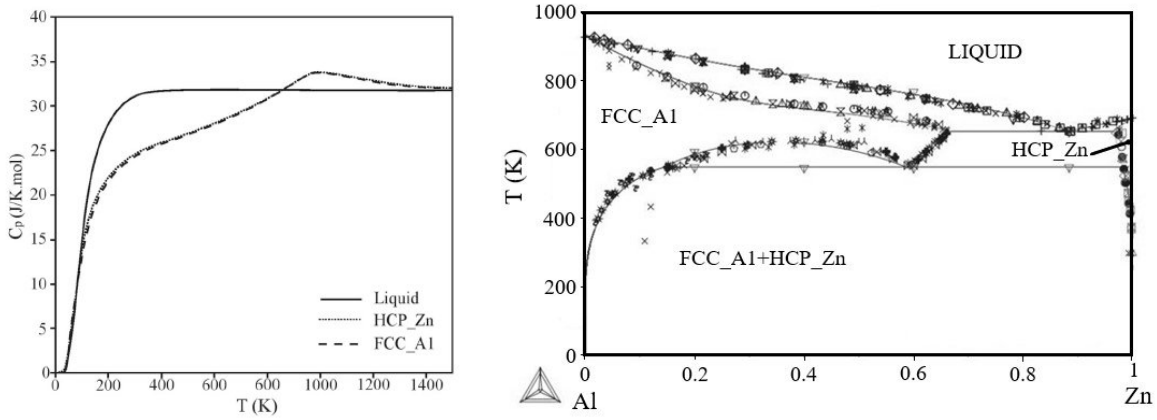


a) b) c)

Fig. 1: The comparison of theoretical and experimental results for the Sb-Sn system: a) the comparison of the equilibrium Sb-Sn phase diagram for the bulk (dashed line) and phase diagram calculated for the particle radius 50 nm (full line). b) The nanoparticles in the sample s1 (TEM image) c) The DSC curves of the sample s1. The upper curve corresponds to the 2nd run, the lower curve to the 1st run of the measurement.

Second program, which started in 2018 is devoted to the work on the 3rd generation unary thermodynamic database and its application for the modelling of systems of practical interest. The development of new database which covers the temperature dependent Gibbs energy descriptions for pure elements in stable and metastable structures is driven by the need to improve some shortcomings existing in currently used unary thermodynamic databases based on the data of SGTE consortium (Scientific Group Thermodata Europe is a consortium of scientists establishing and putting forward **computational thermochemistry** in science, engineering, and industry). The main aim of this effort is to extend the validity of the data below room temperature close to 0 K using Einstein model for heat capacity and improve the description of heat capacities of pure elements at melting point. Also the new model for the liquid phase is proposed for the new generation of data. Our team joints this effort in cooperation with prof. A. Dinsdale from Hampton Thermodynamics Ltd. and prof. A. Khvan from NUST MISIS, Moscow. We participated in the modelling of the Al-Zn-X systems using the new generation unary data. Until now, the unary data have not been extended in a comprehensive way to model the thermodynamic

properties of binary, ternary and multicomponent systems. In paper [3] (published in 2020) the way in which the parameters underlying these physical models vary with composition was explored. This includes a method to define the Einstein temperature for metastable phases of the elements and its relation to the so-called lattice stabilities used in the past, and the variation of the Einstein temperature with composition to account for the composition dependence of the excess entropy. The results were demonstrated for the Al-Zn system, including relatively simple way for parameter conversion between the old and new datasets, and they are presented in Fig. 2. Fig. 2a shows modelled heat capacity of Al for the hcp, fcc and liquid phases, Fig. 2b shows the calculated phase diagram for the Al-Zn system using the concentration dependent coefficients derived in paper [3] with the experimental data superimposed.

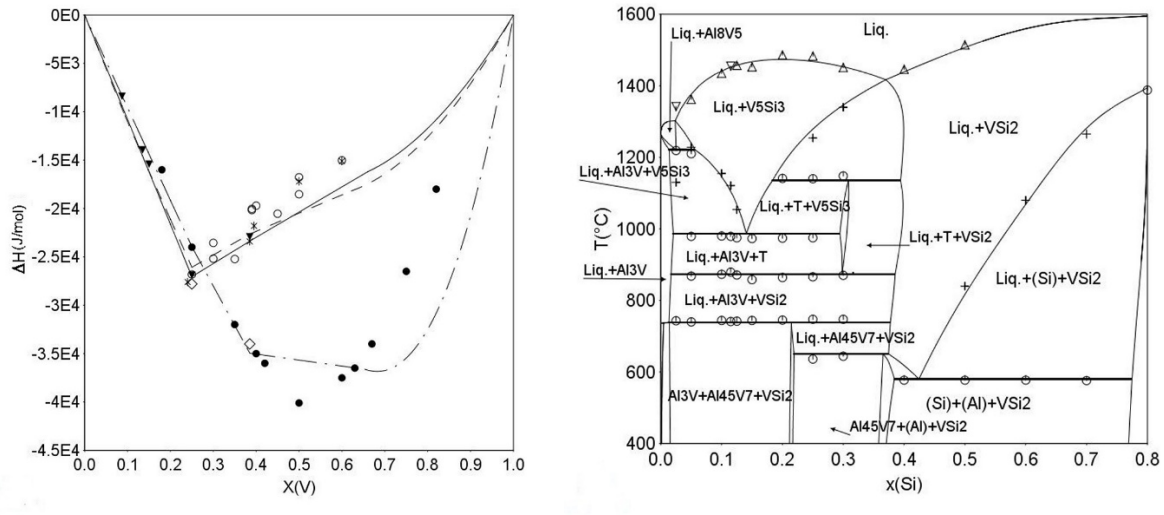


a) b)

Fig. 2: a) Heat capacity of Al and b) phase diagram of Al-Zn system

The theoretical assessments of binary and ternary systems and the thermodynamic database developments using the approach based on “classical” SGTE unary data was carried out also in recent years. The new approach based on 3rd generation data is still far from being applicable for applied research and therefore the development of databases for practical applications continues apace. Our team is working in cooperation with prof. S.-W. Chen from National Tsing-hua University, Taiwan on the development of the Ag-Pb-Se-Sn-Te database which will serve as core database for the development of new thermoelectric materials. This project started in 2018 only and currently first assessments of relevant subsystems (e.g. Ag-Pb-Sn. Pb-Se-Sn, Pb-Se-Te, Ag-Sn-Te) are either sent for publication or in final stages of preparation. In the scope of international cooperation the work is carried out e.g. on the theoretical assessments of the Fe-B-X systems (with Dr Homolova from IMR SAS Košice, Slovakia) and Al-Si-V system (with prof. K. Richter from University of Vienna, Austria) [4] (Fig. 3). Here the errors in the binary Al-V assessment were discovered. Despite of good agreement of the modelled Al-V phase diagram with experiments, there was strong disagreement between some experimental and calculated thermodynamic data. Fig. 3a shows the comparison of experimentally measured enthalpy of formation for the Al-V system at 298 K with assessed values from original literature source (dot-dashed line) and our new reassessment (full line). Complete reassessment of the Al-V system was therefore necessary before completing the ternary system. Our final assessment of the ternary Al-Si-V system shows excellent agreement between theoretical results and

experimental DSC data measured by our co-authors from University of Vienna (Fig. 3b).



a) b)

Fig. 3: a) Enthalpy of formation for the Al-V system at 298 K; b) the isopleth of the Al-Si-V system for 20 a.% V

The ab initio theoretical modelling was not exploited only in combination with the *CALPHAD* method for the modelling of the phase diagram and thermodynamic properties. In cooperation with respected scientist both from the Czech Republic and abroad interesting results were also obtained in modelling of various materials properties e.g. magnetism, thin films properties and plasticity. The results were published in leading scientific journals [5-7].

The epitaxial relationship and stresses of Pd and Rh thin films grown on various substrates were calculated and compared with experiments [5]. In addition to the calculation of total energy of the resulting Pd and Rh structures and the stress in the thin film, also the variation of the total magnetic moment with respect to the uniaxial and biaxial deformation has been studied. It was found that the ferromagnetic ordering in hcp Pd is slightly distorted ratio of hexagonal lattice parameters *c/a* and also for the body-centered cubic (bcc) structure of Rh. Our results allowed to explain ferromagnetic ordering experimentally observed in twinned Pd nanoparticle.

Another interesting results were obtained in purely theoretical work where ab initio calculations were applied in the field of macroscopic plasticity in magnesium [6]. Using a small model of a four-atomic unit cell, we have studied possible mechanism in changing the hexagonal close-packed (hcp) structure into its twinned counterpart. Although the model did not contain the twin boundary, we have been able to explain the trajectories of individual atoms during the twinning process and set back recent arguments in literature about the negligible role of macroscopic shear during twinning.

Ab initio modelling was also able to suggest new materials that can be regarded as natural nanocomposites [7]. In the model, iron nanowires have been embedded in the copper, palladium or platinum face-centered cubic (fcc) matrix. It has been found that the Fe nanowires change the resulting structure into tetragonal with the tetragonal *c/a* ratio close to unity. Then, a simulation of uniaxial tensile tests for the

fcc matrix with the Fe nanowires inside has enabled one to study the deformation behaviour as a variation on the iron content in the nanocomposites. While Fe nanowires rather softened the Cu matrix, the Pd and Pt matrices, on the contrary, were strengthened by the presence of Fe nanowires. We have also studied ground state magnetic ordering of the nanocomposites.

AEM Studies on Relations between Structure and Properties of Materials

The AEM methods were applied by members of the team for the study of the local microstructural changes induced by static and dynamic indentation in nanostructured and nanolaminate coatings (CSF project, 2015-2017, in co-operation with Masaryk University). The main subject of the project was preparation of novel nanostructured and nanolaminate coatings using magnetron sputtering, and complex analysis of coating response to local static and dynamic indentation tests. The team's share on the results was detailed microstructural characterization by means of AEM (Fig. 4). Original data were obtained from indentation tests in a broad range of applied loads; microstructure of deformed volume was studied by advanced methods of electron microscopy (SEM including 3D mapping using FIB, TEM including high resolution), deformation mechanisms and failure onsets were assigned and various effects detected on the indentation curves (such as pop-ins and slope discontinuities) were interpreted in terms of observed microstructural changes and results of computer modelling of indentation response. The results were applied for optimization of deposition conditions and novel nanolaminate coatings with superior mechanical properties were obtained. Results were published in 5 papers in impacted international journals (see e.g. [8,9]).

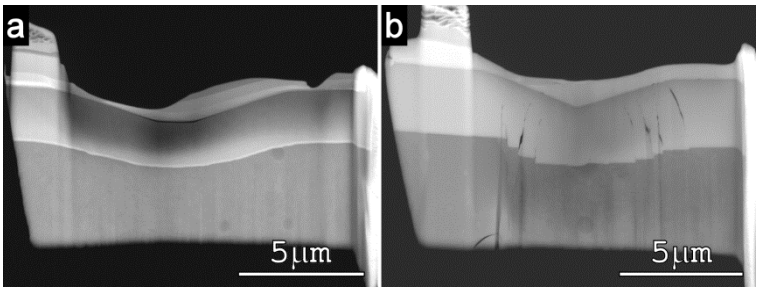


Fig. 4: SEM micrographs, signal of BSE. Thin lamellas across indentation prints prepared on two Mo-B-C samples produced with different deposition conditions, illustrating the resistance of the coatings against indentation with maximum load of 1 N.

TEM expertise of the members of the team was also used for collaboration with team of prof. Peter Rogl at the University of Vienna and with team of prof. Pavel Brož at Masaryk University in the CSF project (2017-2019) "Thermal and phase stability of advanced thermoelectric materials". Our share on the results was microstructural characterization by means of AEM (Fig. 5), DTA experiments and thermodynamic modelling. The research in the field of new non-toxic thermoelectrics is of great importance for materials engineering in automotive and aerospace industry, electronics and others. Improvement of thermoelectrics involves improvement of their physical properties as well as their long term stability. Skutterudites and half-Heusler alloys are the most promising materials. However, both groups contain constituents, which are volatile at device operation temperatures so that progressing degradation impairs the thermoelectric properties. The innovative aspect of the project was in determination of the vaporization characteristics of volatile components by means of Knudsen effusion mass spectrometry and in study of kinetics of respective

processes. These data, together with those on phase equilibria and microstructure provided a physically based understanding of degradation processes of macrocrystalline and nano-structured high performance thermoelectrics and a detailed knowledge on their long term stability. Results were published in 7 papers in impacted international journals (see e.g. [10,11]).

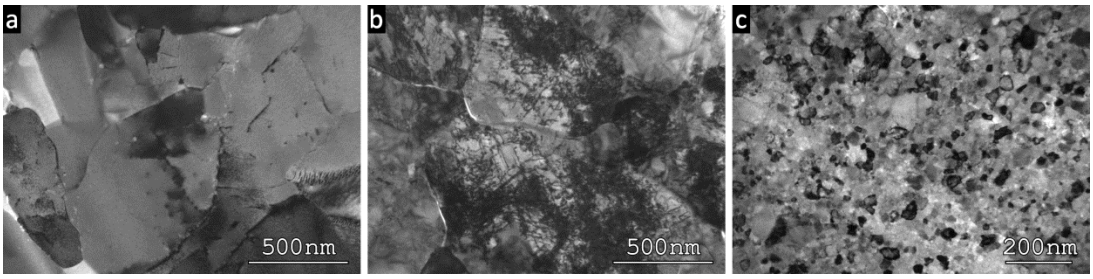


Fig. 5: Microstructure (TEM) of $Ti_{0.15}Nb_{0.85}FeSb$ Half-Heusler alloy after hot pressing (a), after additional high pressure torsion (b) and after annealing (c).

Our research of thermoelectric materials was supported also by two Mobility projects of MEYS: “Spinodal decomposition in half-Heusler alloys: A nanostructuring route towards high efficiency thermoelectric materials“ in 2015-2016 and “High entropy half-Heusler thermoelectric materials with high efficiency“ in 2019-2020. The projects were focussed on nanostructured thermoelectric materials in {Ti,Zr,Hf}NiSn (half-Heusler) alloys with the aim to optimize thermal treatment and improve thermoelectric properties. The combination of know-how, expertise and equipment from both co-operating institutions (i.e. the Institute of Physics of Materials, CAS in Brno and the Institut für Festkörperphysik, TU Wien) was a perfect symbiosis with good synergies to tackle the proposed goals of the project. The state-of-the-art experiments provided reliable knowledge on the temperature dependent solubility limits and on the mechanical properties. Together with the results of microstructural studies they were used for the optimization of thermoelectric properties via nanostructuring.

Long lasting cooperation with the creep experts from the Advanced High-Temperature Materials Group on microstructure investigation by electron microscopy also continued during the period under review. The results were published in six papers in international journals. The microstructural processes occurring at different creep loads have been studied in metal matrix composites [12], low-alloy steel 2.25%Cr1.6%W, austenitic steel HR3C, and 9 %Cr martensitic steel [13,14] and nickel and cobalt-based superalloys [15].

Members of the SPT Group contributed also to the results in other topics solved in research groups of the Institute in the scope of scientific projects. Mechanisms of plastic deformation and twinning interfaces in hexagonal metals, microstructure of Fe-Al based alloys and new composite materials for environmental applications were among them. The Team’s share on these projects was microstructural characterization by means of AEM.

The Studies of the Diffusion in Ternary Systems

The simple numerical algorithm was proposed that enables to estimate ternary interdiffusion coefficients D in systems with not too strongly interacting components. D’s are obtained from measurement with a single model diffusion couple [16]. They can be used to predict concentration curves $C_i(x)$ (i = 1, 2, 3) in arbitrary time with numerical errors that are comparable with experimental errors of measurement of C_i itself and comply with theoretical constraints following from the linear irreversible

thermodynamics. The algorithm does not need any external thermodynamic data and external measurements (e.g., it is not necessary to measure the shift of inert markers in the interface). It can be applied using commonly accessible SW, and it is not time-consuming. Values of D are close to those obtained with the help of much more laborious experimental – also approximate – methods.

The Development of Advanced Materials for the Hydrogen Storage

We continued our investigation of new prospective hydrogen storage (HS) alloys, based on alloy base which was covered by CZ patent (No. 302464 in 2011). Detailed study of HS behaviour of the alloy during its one-year ageing was recorded and a model was proposed relating the structure changes of the material and its HS characteristics. This deepened the understanding of this class of HS alloys.

It is known that certain additives facilitate the preparation of HS alloys. The mechanism of their participation in HS process was, however, not fully understood. We studied hydrogen sorption separately in several most frequently used additives (carbon allotropes and talc). The best results were found with amorphous carbon that acted as anti-sticking agent and, at the same time, it also beneficially took part in HS process itself.

We paid extensive attention to improving the HS performance via changing sorption reaction ways. This means the introduction of elements/compounds that provide intermediate thermodynamic steps between the hydrogen-free and fully hydrogen-charged states of studied alloys. Significant improvement was found for Mg-based HS composition doped with Sb that forms stable intermetallic with Mg [17].

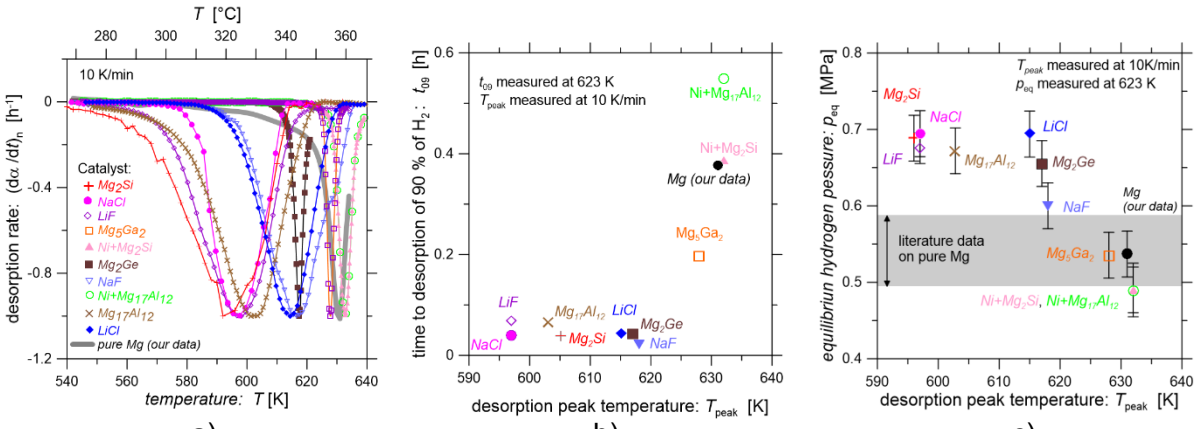
The team also took part in experiments with NiTi shape-memory alloys. Changes in phase composition and in shape-memory parameters in dependence on hydrogen doping were investigated. These research activities were performed in close co-operation with Advanced High-temperature Materials Group of our Institute. Investigation of structure of pure Mg, which makes the basis of our Mg-based HS alloys, was also done in co-operation with Electrical and Magnetic Properties Group of our Institute.

We made also attempts to design a HS Mg-based alloy with as low operating temperature as possible. Results achieved with a complex Mg-Al-Ti-Zr-C ball-milled alloy were summarized and published in paper [18].

A significant fraction of capacity of the team was devoted to investigation of additives with catalytic effect upon the HS process in Mg-based alloys. We achieved noticeable results that place our team to the top position among the renowned research teams in the field. We studied easy-gate-effect that showed fine particles of chosen intermetallics ($Mg_{17}Al_{12}$ and Mg_2Si) located at the surface of Mg grains [19]. It was found that this morphology type of HS alloy shows beneficial effect upon the HS kinetics.

Introduction of additives directly inside the Mg grains is another way how to influence the HS catalysis. We performed an extensive study of a series of additives in Mg basis [20] and found a data set that showed features of modified alloys.

Considerable decrease was reached in desorption temperature (Fig. 6a), improvement of desorption kinetics (Fig. 6b) and also in lowering of the thermodynamic stability of hydride phase (Fig. 6c). All these achievements are desirable from the point of view of practical applications.



a) b) c)

Fig. 6: a) Results summarizing experiments on temperature programmed hydrogen desorption (a), kinetics (b) and dynamics hydrogen desorption experiments [C5].

The study was indicated by selection committee of Advances in Engineering (AiE - see web page https://advancesENG.com) as a key scientific article contributing to research excellence. AiE is a prestigious international body that highlights papers of exceptional scientific importance to a broad science and engineering audience and mediates the extended visibility of the results. It disseminates globally the selected studies of research excellence across academia and in the wider community and to inspire new research directions.

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Research activity and characterisation of the main scientific results

The main research areas of the Electrical and Magnetic Properties (EMP) group are related to defects in (nano-)materials, mostly magnetic ones, and their impact on materials properties, in particular electric and magnetic characteristics. The plasticity of materials is included, too, in order to solve the situation when potentially promising materials are so brittle that they are unfortunately useless for any practical application. Selected results are represented by 31 publications (mostly ordered from the oldest to the latest) that are mentioned at the end of this subsection and are referred to as [EMP_01] - [EMP_31]. Below we summarize major research projects together with contributions of individual scientists.

The most important research project in the group in the evaluated period was entitled “Theory-guided design of novel Fe-Al-based superalloys” and was supported by the Czech Science Foundation. The head of the EMP group, Dr. Martin Friák, was the principal investigator and Assoc. Prof. Vilma Buršíková from Masaryk University was the co-investigator. Members of the EMP group contributed with a synergic combination of theoretical calculations and experimental measurements while Assoc. Prof. Buršíková provided nano-scale measurements of elastic and plastic properties.

The Fe-Al-based superalloys are two-phase magnetic nanocomposites (see Fig. 1) with very high number of defects. These include point defects (off-stoichiometric anti-site atoms or vacancies), different levels of chemical (dis-)order, as well as extended defects such as antiphase boundaries and internal interfaces. It was found that (i) magnetic, thermodynamic, structural and elastic properties very sensitively depend on the distribution of atoms and (ii) the overall magnetic properties of these nano-scale composites depend in a very complex way on the properties of individual phases.

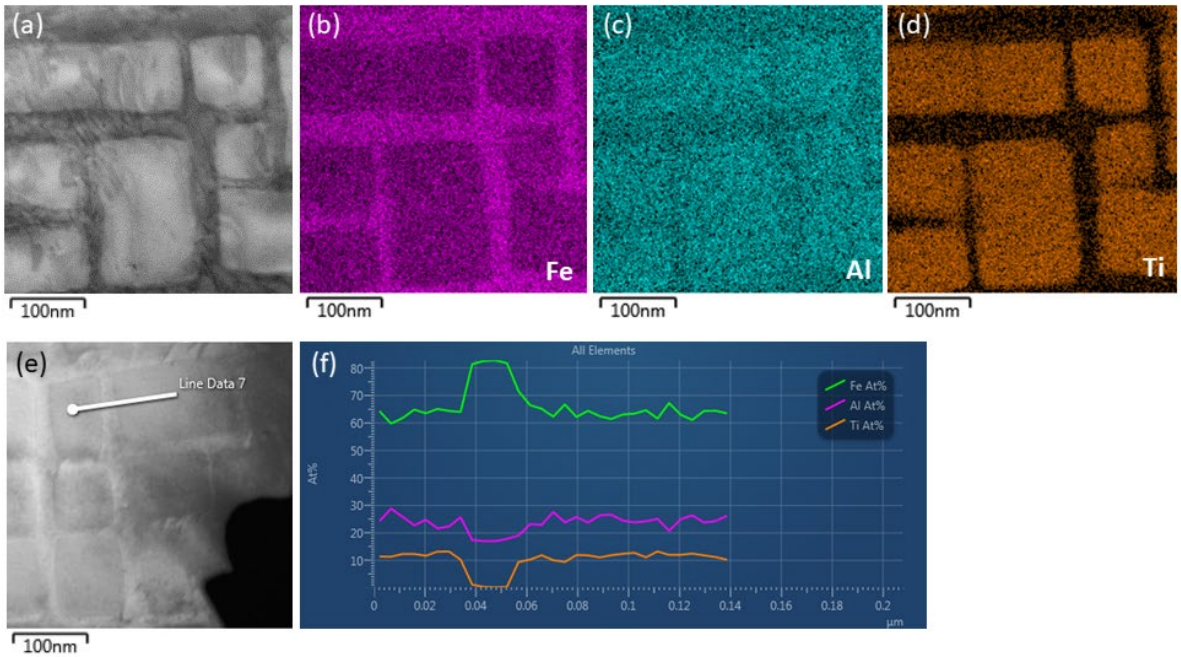

Figure 1 *A transmission electron microscope (TEM) micrograph (a) showing the superalloy nano-structure in our $Fe_{71}Al_{22}Ti_7$ sample. The corresponding chemical composition (b–d) as detected by the Energy-Dispersive X-ray (EDS) spectroscopy in*

the same spot of our TEM lamellae. The analysis is accompanied by one of our line-scans (e) together with the corresponding chemical profile (f).

The research within the project resulted in findings which were often reported for the first time. For example, intricate structure-property relations were revealed between vacancies and magnetic properties in the Fe-Al system [EMP_25]. Quite unexpected connections were found between the local distributions of atoms and tensorial elastic properties in Fe-Al(-Ti) phases [EMP_23]. A years-long unexplained discrepancy between theoretical results and experimental data related to the low magnetic moment of Fe_2AlTi was resolved for the first time [EMP_18]. Further, a new structural model (i.e., a distribution of atoms) in Fe_2AlCo was proposed in [EMP_16], which, in contrast to previously published models, agrees with experiments. Further, impact of internal interfaces in Fe-Al nanocomposites on their magnetic properties was theoretically analysed in detail for the first time, either in case of interfaces between the phases [EMP_20] or internal extended defects, antiphase boundaries [EMP_23]. The antiphase boundaries were found particularly interesting because the lowering of chemical order, which they induce in $Fe_{71}Al_{22}Ti_7$, can increase the total magnetic moment by 140 % with respect to the thermodynamically most preferred state (see Fig. 2). As the study [EMP_23] identified this surprising impact of antiphase boundaries (APBs) on magnetism, we studied them also in Fe_3Al with and without Cr additions [EMP_30] and found their properties to be very sensitive to many factors, such as the density of APBs as well as the local distribution of atoms and the chemical composition.

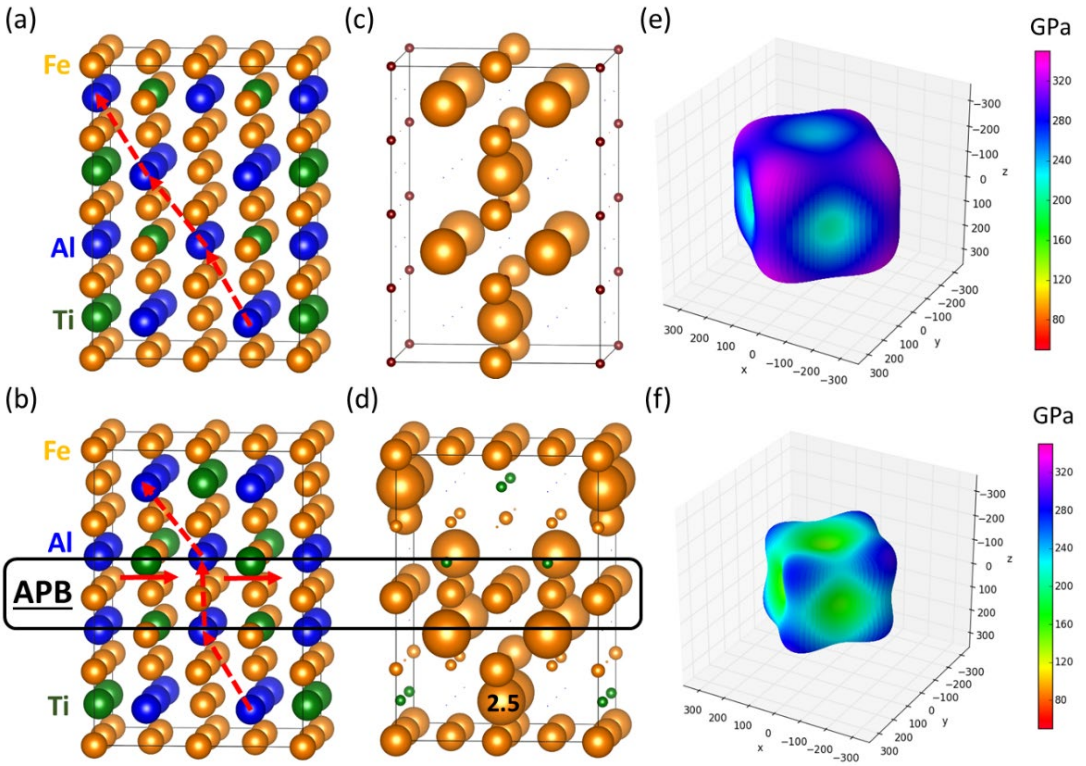

Figure 2 *Schematic visualizations of a Fe-Al-Ti intermetallic phase modeled by a 64-atom supercell without any antiphase boundaries (a) and with an antiphase boundary (APB) (b) together with the corresponding ab initio calculated local magnetic moments (c,d) and elastic properties (e,f) visualized in the form of a directional dependence of the Young's modulus. The magnitude of the local magnetic moments is visualized by the diameter of spheres representing the atoms (for the scaling see local atomic magnetic moment of 2.5 μ_B). The change of the overall magnetic*

*moment is very significant: the **APB-containing state (see part (b)) has the total magnetic moment higher by 140 % than the APB-free one**. Red dashed arrows in (a,b) indicate layering of Al atoms, which is altered due to the APB shift (red arrow), for details see [EMP_23].*

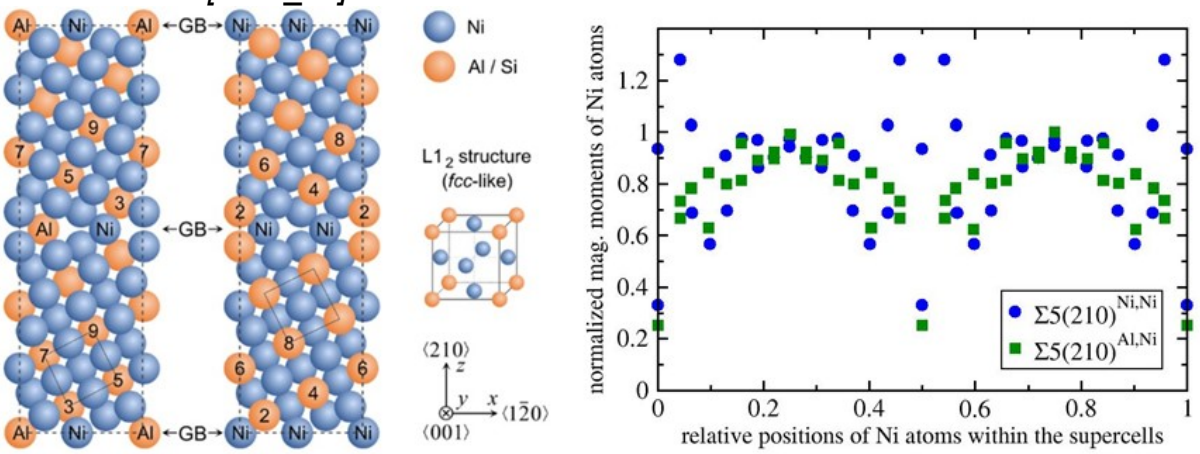

Figure 3 *Supercells used in electronic structure calculations of Σ5(210) grain boundaries (GBs) in Ni_3Al with different interface stoichiometries, $\Sigma 5(210)^{Al,Ni}$ with both Al and Ni atoms at the GB plane and $\Sigma 5(210)^{Ni,Ni}$ with only Ni atoms at the GB plane. Also shown are computed local magnetic moments of Ni atoms normalized w.r.t. their bulk value as functions of their relative positions within the computational supercells along the direction perpendicular to the simulated grain boundaries (GBs are located within the supercells at relative positions 0, 0.5 and 1). For details see [EMP_09].*

As another type of defects studied in magnetic materials, Prof. Mojmír Šob and his co-workers, such as **Dr. Monika Všianská**, performed systematic quantum-mechanical studies of grain boundaries (GBs) with and without segregated impurities. The focus was on the impact of grain boundaries on magnetism (see Fig. 3), structure, elasticity and strength of studied materials. The research covered elemental transition metals Ni and Co with sp-electron impurities [EMP_07] and magnetic intermetallics Ni_3Al with segregating Si [EMP_09]. Importantly, a very significant impact of grain boundaries as well as individual segregating atoms on the local magnetic moments was found (see also tensile loading in Fe-Al nanocomposites in [EMP_21]). The results on theoretical strength and on GB segregation were also summarized in a review article published in a prestigious international journal Progress in Materials Science (Prog. Mat. Sci. 73 (2015) 127-158, IF = 23.725) into which Prof. Šob contributed.

An example of a synergic combination of experimental research and quantum-mechanical calculations is represented by the study of **magnetic properties of equiatomic CoCrFeMnNi alloy by Dr. Oldřich Schneeweiss** and other members of the EMP groups (working also with collaborators from outside of IPM). This five-component magnetic high-entropy (or better multi-principal element) alloy was found to have an unexpected magnetic behaviour at low temperatures [EMP_10]. For the first time in this material it was shown that field assisted cooling below 40 K results in a systematic magnetization bias of the hysteresis curves which reflected the sign of the cooling field (see Fig. 4). Macro-/micro-scale experimental data were complemented by quantum-mechanical calculations of local magnetic moments of

individual atoms in this highly disordered material. The magnetic state is a complicated ferromagnetic one with local magnetic moments sensitively depending on the surrounding atoms. The main part of the experimental measurements, theoretical calculations, and interpreta-tion of the result was done at IPM in Brno. But as the results were based on applica-tions of indeed quite many experimental methods, and not all of them were available at IPM, a smaller part of data was obtained via collaboration with other laboratories.

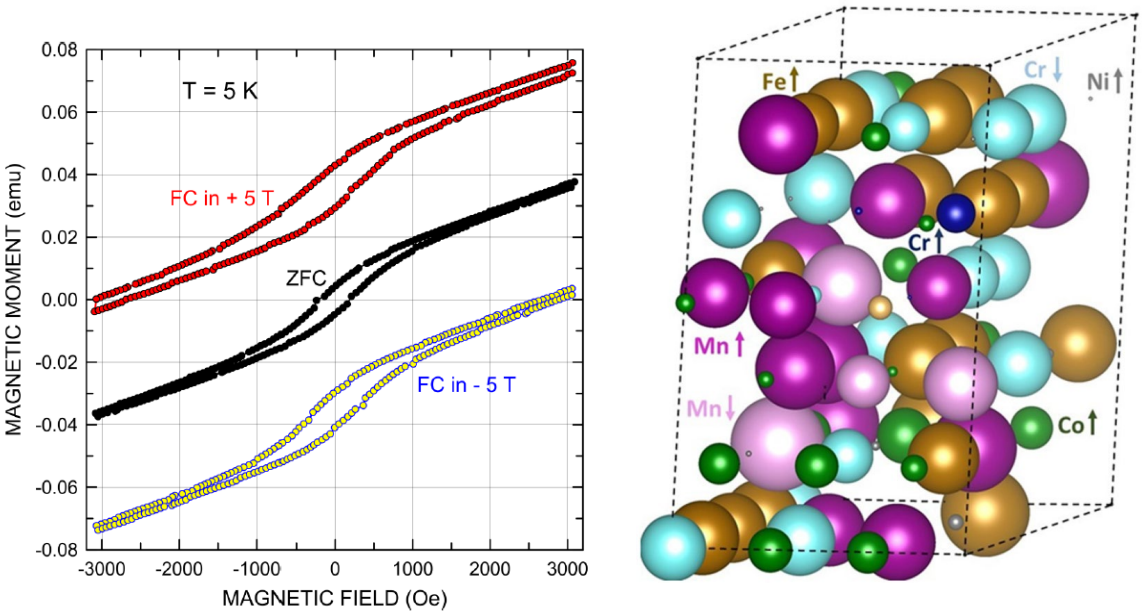

Figure 4 *Experimental (left) low-temperature magnetic properties of multi-principal element CoCrFeMnNi alloy are found tunable by external magnetic field during field cooling (FC) (in contrast to zero-field cooling - ZFC). The measurements were complemented by quantum-mechanical calculations (right) that predicted the low-temperature state of CoCrFeMnNi to be very complex [EMP_10]. The magnitude of local magnetic moments of atoms is shown by the size of spheres (maximum 2.7 μ_B).*

Dr. Yvonna Jirásková studied **relations between the structure and magnetism** in a series of studies and published 20 impacted papers between 2015 and 2019. She focused on structural and magnetic characterization of, for example, Fe and Pd alloyed with hydrogen **(interstitial point defects)** as candidate materials for hydrogen storage applications [EMP_08]. She also investigated Fe-oxide/Ce-dioxide prepared using several chemical procedures (because these are important magnetically separable sorbent for environmental applications). Dr. Jirásková also very significantly contributed into the above mentioned project related to Fe-Al superalloys containing Ti, Nb, or Co (see, e.g., Ref. [EMP_14]). Further, she studied defects created due to production technologies and the impact of composition on the structural and physical properties of magnetic Heusler materials Co_2FeAl [EMP_17] and $Fe_2Mn(Si,Al)$ alloys [EMP_28]. The results were partly obtained within the currently still ongoing research project “New Composite Materials for Environmental Applications” supported by the Czech Ministry of Education, Youth and Sports (2018/02/28 – 2021/02/27).

Dr. Jirásková and Dr. Schneeweiss together with Dr. Tomáš Žák intensively used Mössebauer spectroscopy as a technique allowing to probe essentially individual

atoms (most often Fe atoms) and their magnetic state. Figure 5 shows examples of these spectra taken in the case of Fe-Ni alloy with and without P and B atoms.

Defects induced in materials by external conditions and mechanisms were studied by **Dr. Eva Švábenská.** Her first topic was the effect of different environments and processing on material structure. She studied corrosion behaviour of silicon steel during annealing at 500° and 700°C in various atmospheres and the results were published in Ref. [EMP_22].

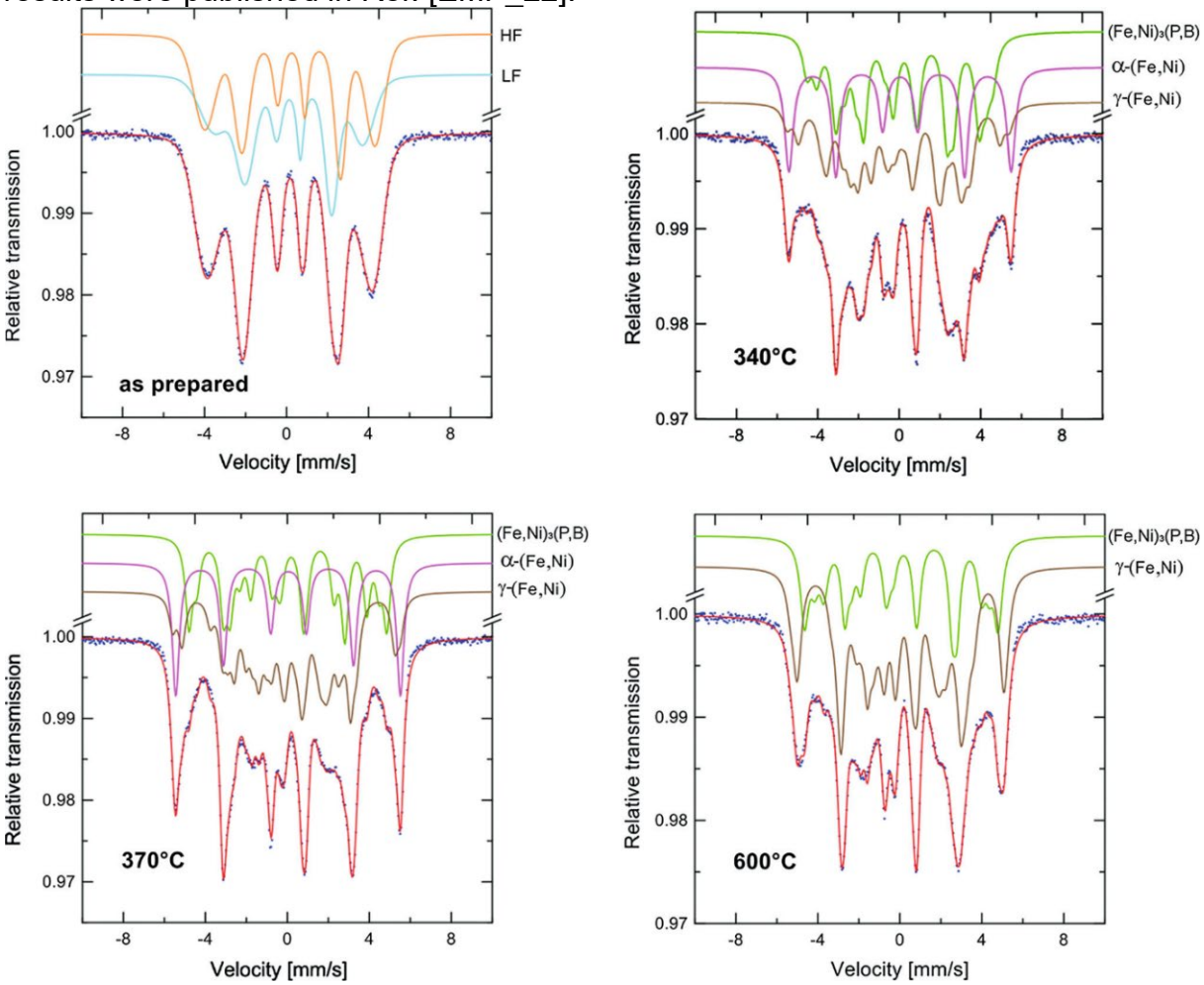

Figure 5 *Mössbauer spectra of the as-prepared Fe-Ni alloy (with and without P and B) and the alloy annealed at 613 K, 643 K, and 873 K (340 °C, 370 °C, and 600 °C).*

Further, Dr. Švábenská studied the changes in the microstructure of silicon steel after treatment by Laser Shock Peening (LSP) and Pulsating Water Jet (PWJ) (see an example of a PWJ-affected surface in Fig. 6).

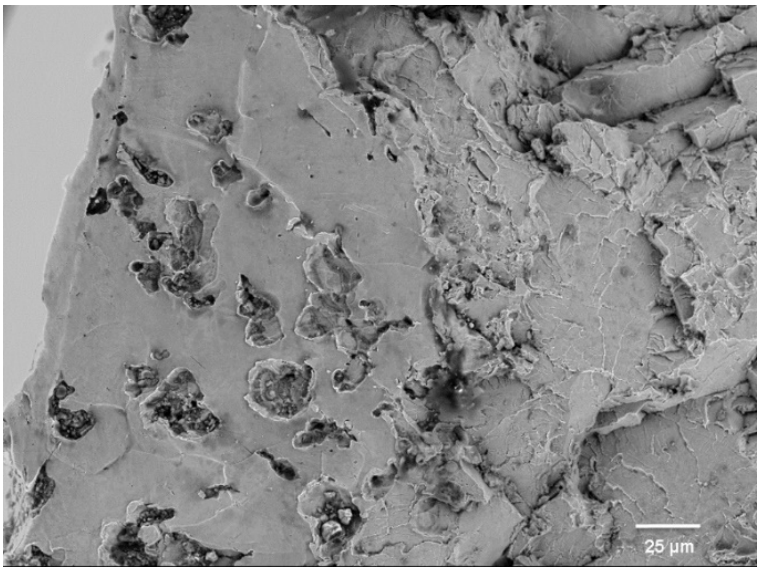

Figure 6 *An image of the surface of a silicon steel damaged by Pulsating Water Jet.*
The second area of research of Dr. Švábenská was related to brake pads. In this field, she focused on chemical and phase analysis of brake pads and particles released during their use, i.e. **a topic important for public health**. The results are being gradually processed and published.

Dr. Pavla Roupcová contributed into the above mentioned corrosion study of Fe-Si steel of Dr. Švábenská [EMP_22] and further oriented herself towards **point defects in the form of interstitials**. She focused mostly on hydrogen storage materials and theirs behaviour, typically the kinetics of (dis-)charging and the effect of materials doping (see the paper in International Journal of Hydrogen Energy 42 (2017) 6144-6151). Her studies were partly performed with co-workers of Dr. Jiří Čermák from the group Structure of phases and thermodynamics (which is led by Dr. Aleš Kroupa) and Dr. Roupcová' role was to conduct high-temperature X-ray diffraction experiments aimed at determining changes in the phase composition (Kovove Materialy - Metallic Materials 54 (2016) 389-396). Further, Dr. Roupcová also examined the structure and phase composition in magnetic spinel-structure materials at low temperature. Her X-ray measurements were compared with neutron diffraction, i.e., the crystal and magnetic structure was determined (Physica B – Cond. Matter 536 (2018) 89-920).

Dr. Naďa Pizúrová was intensively studying **nanoparticles**, in particular those for biomedical, chemical and optoelectronic applications (see Fig. 7). She cooperated with universities in Hertfordshire (UK), Yekaterinburg (Russia), and Belgrade (Serbia). Her main contribution was the structural and chemical characterization using electron microscopy (SEM, TEM, HRTEM, and EDX). Employing these techniques, she was intensively involved in (i) the above mentioned research related to Fe-Al based superalloys (see Refs. [EMP_14] and [EMP_23]) as well as (ii) the analysis of defects induced in materials by the Pulsing Water Jet technique.

As far as the **extended defects important for plasticity of materials** are concerned, **Assoc. Prof. Roman Gröger** with his co-workers developed a physically sound mesoscopic framework that describes **non-Schmid behavior of dislocations** in body centered cubic (bcc) metals and the associated onset of crystal plasticity. This is based on atomistic studies of isolated 1/2[111] screw dislocations using Bond Order Potentials (BOP). These results serve as inputs into the mesoscopic model

based on the Master equation that provides the evolution of texture (plastic strain) in deformed bcc single crystals [EMP_04,EMP_06,EMP_31].

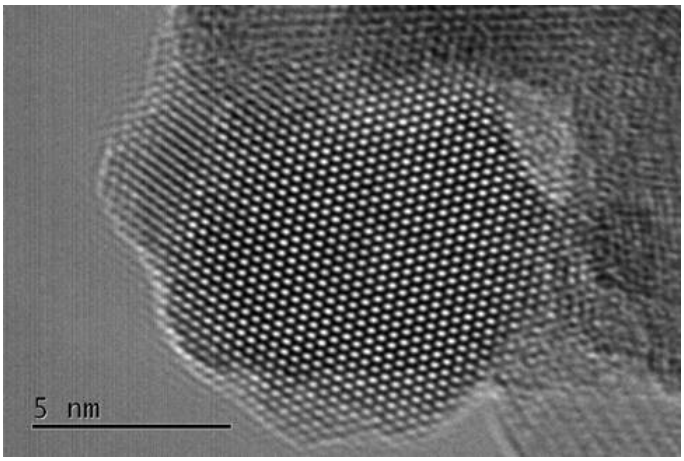

Figure 7 *Maghemite nanoparticle for hyperthermia of cancer.*

Further, **Ing. Petr Vacek, a PhD. student of Assoc. Prof. Roman Gröger**, used the technique of Electron Beam Induced Current (EBIC) in conjunction with TEM, SEM and AFM to provide a detailed characterization of **dislocation substructures** in MOCVD-grown films of wurtzite AlN/Si{111}. Systematic correlation was found between the reduced EBIC current and the size of the surface depressions caused by the threading dislocation emanating on the surface [EMP_29]. The above mentioned results of Assoc. Prof. Roman Gröger were obtained within research projects supported by the Czech Science Foundation, in particular “Origin and mechanism of anomalous slip in non-magnetic bcc metals” (2015-2018) or “Interplay of plasticity and magnetism in alpha-iron and chromium” (2019-2021), with Assoc. Prof. Gröger being the principal investigator. Further, he was part of the BACK FOR THE FUTURE (CZ) consortium (2017/08/31 – 2018/08/30) supported by the European commission.

Regarding **extended defects, such as dislocation loops, Dr. Jan Fikar** employed molecular statics simulations to investigate the interactions of circular and hexagonal 1/2<111> prismatic dislocation loops in body-centered cubic iron with two parallel {111} free surfaces of a free-standing foil. By varying the size and shape of the loop (see also Fig. 8), the critical depth at which the image stresses overcome the internal lattice friction (and thus drive the loop towards the surface) was identified. Further investigation was aimed at how this depth and the corresponding critical stress on the dislocation depend on the shape and size of the loop and outline how these results can be used to correct transmission electron microscope measurements of the densities of prismatic dislocation loops in thin foils [EMP_01]. The research was supported by the Czech Science Foundation within a project entitled “Interaction of prismatic dislocation loops in alpha iron and tungsten” (2016-2018).

Another type of extended defect mediating plasticity in materials, deformation twins, was the main topic of research of **Dr. Andrej Ostapovec.** He studied the faceting of twin boundaries and facet nucleations as well as the influence of twin boundary faceting on deformation coupled with twin boundary migration [EMP_03]. He also examined dislocation reactions, which accompany twin nucleation from <a> screw dislocation, and studied them in magnesium [EMP_11]. Besides, interaction of

twin boundaries with stacking faults were studied and it was proven that such interaction can lead to non-diffusional growth of I_1 basal stacking faults [EMP_24].

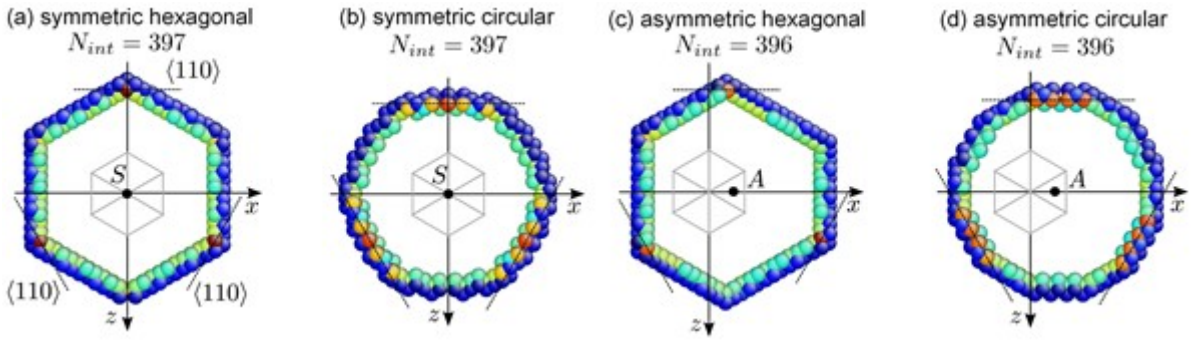

Figure 8 *Interactions of prismatic dislocation loops with free surfaces in thin foils of body-centered cubic iron. The four types of loops and the corresponding number of interstitial atoms used to create the loop. The point S is the center of the interstitial disc that generates symmetric loops, while the point A is the center of asymmetric loops. The colors of atoms represent the energy attributed to each atom, from blue (lowest energy) to red (highest energy) [EMP_01].*

The above mentioned results of Dr. Ostapovec were partly obtained within research projects supported by the Czech Science Foundation “Mechanisms of plastic deformation and twinning interfaces in hexagonal metals” (2016-2018) and “Non-Schmid behavior of dislocations in magnesium and its alloys” supported by the Czech Ministry of Education, Youth and Sports (01/2019 – 07/2020).

Next to the experimental and theoretical research described above, members of the EMP group contribute **into development of new methods**, in particular theoretical ones. **Assoc. Prof. Ilja Turek developed an ab initio theory of electronic transport** properties with focus on relativistic effects in magnetic and nonmagnetic solids. These studies included the Gilbert damping of magnetization dynamics in random ferromagnets [EMP_02] and the spin Hall effect in nonmagnetic metals and alloys [EMP_27] as well as solutions of selected theoretical problems [EMP_05]. The most important results are the very low magnetic damping predicted for a random bcc Fe-Co alloy with 25 at.% Co [EMP_02] (later confirmed experimentally), and the sizable spin Hall angles predicted for Pt-Re and Pt-Ta primary substitutional solid solutions [EMP_05,EMP_27].

The postgraduate student **Dr. David Wagenknecht** of Assoc. Prof. Ilja Turek (PhD thesis defended in June 2019) **developed a simplified and efficient theoretical approach for a treatment of effects of finite temperatures on transport properties of metallic magnetic systems.** The formalism employs an alloy analogy model which allows one to include the lattice vibrations (phonons), the spin fluctuations (magnons), and random impurities on equal footing in evaluation of the electrical conductivity tensor [EMP_12]. The developed theory has recently been used to identify the dominating structure defects in the half-metallic NiMnSb compound with potential applicability in spintronics [EMP_26].

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