Characteristics of main research directions investigated at the institute and the achievements 2010−2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Response to the last evaluation

Main findings of the last evaluation: The Institute as a whole was classified with the highest ranking among evaluated institutes of the Czech Academy of Sciences. It was found that the Institute produces very good scientific outputs in the field of material science. The researchers contribute to the extension of the general knowledge on the relations of microstructure of advanced engineering materials and mechanical, electrical and magnetic properties. The international research collaboration was evaluated as high-powered and effective. Further, the strong co-operation with industry, both domestic and international was highly appreciated.
The review team, however, draw attention to the age structure of the researcher staff. The structure was found to be not optimal because the age of the key researchers is high and the number of young co-workers was not increasing sufficiently in the evaluated period. The second serious recommendation for the Institute management, which resulted from the visit of the team of evaluators in the laboratories of the Institute, was to make a strong effort to stop the aging of the experimental facilities, which are crucial for the continuation of the research work on the world-calss level.
Response to the evaluation: The main orientation of the research was not changed since the last evaluation and also the ratio of the "basic" (i.e. research resulting in new knowledge suitable for publication in scientific periodicals) and "applied" (i.e. contractual and collaborative research for industry) research was held nearly constant throughout the whole evaluated period.
The management of the Institute made strong effort to attract young employees. This endeavour was successful, as can be seen from the diagram of the age structure of the whole Institute related to December 31st 2014 and more clearly from the age structure diagrams of the particular research groups. The unfavourable wages conditions for young researchers were overcome, mainly due to the benefits of the Operational Programme Research and Development for Innovations and the Programme for Educational Co-operations. Based on this, practically all research groups were strengthened by young perspective researchers. It has to be stressed that the pedagogical activity of members of the research groups of the Institute at the Brno Technical University and Masaryk University enabled to engage the first-rate young scientists. All the measures done in this direction resulted in a decrease of the average age of researchers from 47.7 at last evaluation to 43.0 at present.
The participation of the Institute in CEITEC - Central European Institute of Technology which is a large project realized under the support of the Operational Programme Research and Development for Innovations of Ministry of Education, Youth and Sport of the Czech Republic (MEYS), stopped the aging of the experimental facilities. More than EUR 3.6 million was the investment costs within this project. This subsidy led to the substantial improvement of research infrastructure of the Institute, which could not otherwise be done from the institutional support obtained from the Czech Academy of Sciences. The new CEITEC research equipment is allocated in all research groups of the Institute. More detailed information can be found in the Research reports of the particular research teams.

Moreover, the start-up grant of CIEITEC IPM improved the allowance of participating researchers.

Characteristics of main research directions

The Institute is traditionally focused on elucidation of relations between the behaviour and properties of materials and their structural and microstructural characteristics. Particular emphasis is placed upon the research into advanced metallic materials and composites with a metal base and ceramics in relation to their microstructure and production. This main research trend was kept during the last five years and thus the main achievements consist in (i) new piece of knowledge on the physical nature of processes which occur in advanced metallic materials during creep, fatigue, creep/fatigue interaction, brittle fracture and their combinations in the relation to the evolution of microstructure and in getting, (ii) new findings concerning the relations of material microstructure and thermodynamic, diffusion and magnetic properties of prospective materials and in theory-guided materials design and ab initio calculations. Structure of materials is understood in a very broad sense, ranging from atomic bonds, through crystal lattice and its imperfection, the size of crystallites in materials, to the macroscopic dimensions of loaded bodies.

Main achievements obtained in the evaluated period

The main scientific achievements are described in detail in the Research reports of the teams of the Institute. From the point of view of the whole Institute and its research potential, it is very substantial that the activity of the particular research groups is highly interlinked. This is very advantageous, because the present-day material research is very broad field requiring utilization both of demanding and very expensive experimental equipment in close combination with high-level theoretical, computational and modelling knowledge. From this point of view, the Institute manifests itself as a powerful research unit. Very important is that the young scientists, who were employed recently, strongly emphasize in their thinking just the unique combination of theoretical and experimental knowledge that can be synergically combined in materials design. During the last five years, a lot of good investigations were performed. In the following paragraphs is presented a selection of results which characterize the most important research directions which were followed during the evaluated period.

Creep processes and fatigue behavior of ultrafine-grained metallic materials

Despite its promise, the use of ultrafine-grained (UFG) metallic materials as a new generation of structural and functional materials has only recently been realized. Only in very recent years a breakthrough has been made in this area, associated both with developing new methods for fabricating bulk UFG materials and with investigating the fundamental mechanisms that lead to novel properties in these materials. Accordingly, an extensive research has been conducted in the period 2010 – 2014 to reveal the effect of UFG microstructure on the fundamental creep behaviour and properties of various UFG metallic materials. For producing bulk UFG materials under the investigations, an equal-channel angular pressing (ECAP) was used. It was found that the creep resistance of UFG materials was markedly improved with respect to their unpressed coarse-grained counterparts. In addition, the creep behaviour of UFG materials depends on the number of ECAP passes (Fig.1a) because the microstructure and microtexture are homogenized with increasing number of ECAP passes (Fig. 1b). The analysis of creep deformation mechanisms has indicated that creep in UFG materials occurs by the same mechanism(s) as in conventional coarse-grained materials with intragranular glide and climb as the dominant rate-controlling flow process. This topic has been investigated in a close co-operation with foreign colleagues from USA, UK, South Korea, Russia and Slovakia. Some of interesting results were summarized by V. Sklenička, J. Dvořák, M. Svoboda, P. Král and M. Kvapilová: in Z. Ahmad (Ed.) Aluminium Alloys – New Trends in Fabrication and Applications, InTech, Croatia, 2012, Chapter 1, pp. 3 – 45.

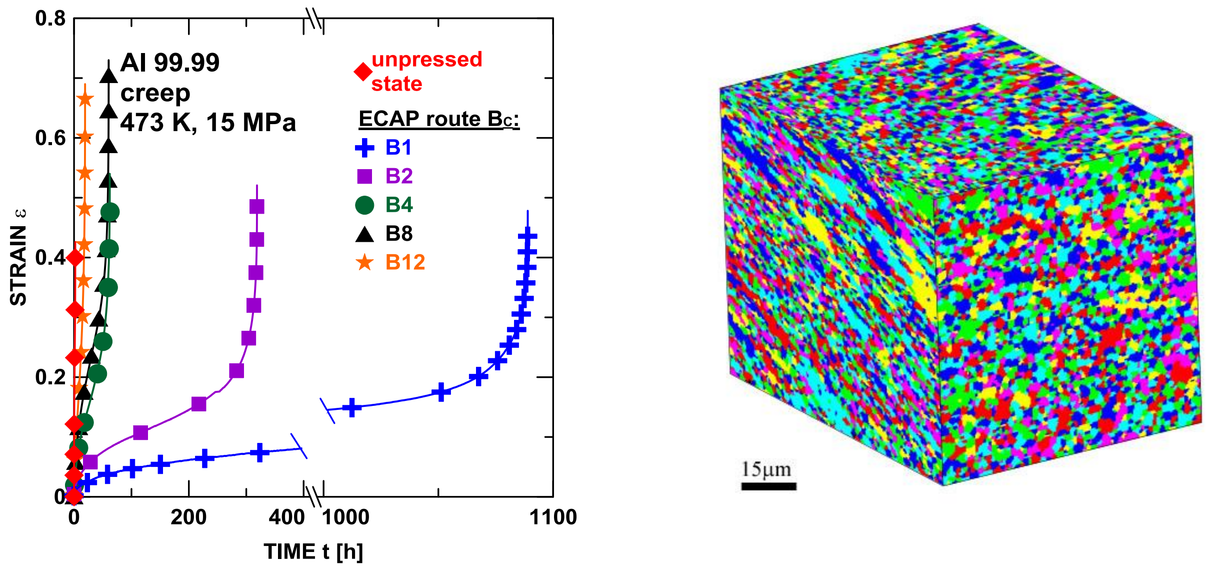


Fig. 1. (a) standard creep curves of Al 99.99 for unpressed (coarse-grained) state and different number of ECAP passes, (b) EBSD image of Al sample processed by 8 ECAP passes, (Δ ≥15°).

The investigation of fatigue behaviour of UFG materials brought answers to some of recently discussed questions in the research society, namely what are the mechanisms of the cyclic plastic strain localization in this type of materials, which microstructural, material and severe plastic deformation process parameters influence the strain localization and hence the fatigue lifetime, which factors influence the stability of UFG structure under cyclic loading and what is the difference in the mechanisms of cyclic slip localization in ultrafine-grained structures and conventionally grained ones. The results were published e.g. by L. Kunz in a book Copper Alloys - Early Applications and Current Performance - Enhancing Processes edited by L. Collini, InTech. Publisher 2012.

The effect of Zr addition on creep of Fe-30 at.% Al alloys

A wide investigation aimed on the improvement of creep behaviour of advanced metallic materials brought a lot of valuable results. As one nice example, the results of the study of the effect of Zr addition on creep properties of Fe-30 at. % Al alloys can be presented. Compressive creep of alloys based on Fe-30 at. % Al with zirconium additions in the range of 0.4-5.2 at. % was studied at temperatures from 650 to 900 °C. The alloys were tested in two different states: (i) cast and (ii) annealed at 1000 °C for 50 h. Stress exponents of creep rate were estimated. The values of the stress exponent n can be explained by dislocation motion controlled by climb and by the presence of second-phase particles. Higher stress exponents were detected only at the temperature of 900 °C and with zirconium content of up to 2 %. Creep resistance at temperatures of 700 and 900 °C increases with the increasing amounts of the secondary phases λ_1 and τ_1. At the temperature of 650 °C, creep resistance is given not only by the volume fraction of particles but also by the ratio of τ_1 / λ_1. This can be attributed to a more effective strengthening by particles of τ_1 resulting either from their mechanical properties or from their finer distribution. The research was done in co-operation with Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University, Prague and published by F. Dobeš, P. Kratochvíl: Intermetallics 43 (2013) 142-146.

Fibre reinforced composite on ceramic basis

Materials suitable for use at high temperatures are generally based on ceramics. The fundamental problem of application of these materials is their brittleness. For several decades, considerable effort is devoted to their toughening. The most effective way is the use of reinforcement in the form of long ceramic fibres. This apparent paradox, i.e. the reinforcement of brittle material by another brittle material, uses synergism effect of several reinforcing mechanisms and therefore fibre composites are of the highest resistance to the damage. Preparation of ceramic fibre reinforced composites for long-term use at temperatures higher than 1000 °C is very costly, mainly due to the high prices of inputs and expensive production. Development of the long fibre composites using economically advantageous raw materials and production processes together with maintaining sufficient mechanical properties was the main aim of the long-term co-operation between the Institute and two other institutes of the Czech Academy of Sciences, namely IRSM AS CR and IMC AS CR. On the basis of the mechanical response of the individual components of the composite and consequently the composite itself was optimized its preparation. It was possible to skip expensive processing steps like surface treatment of the fibres. The unique high temperature properties are provided by the matrix consisting of SiOC glass prepared by pyrolysis of polymeric precursors based on polysiloxane resins. The advantage is not only the high temperature stability of matrices tested at temperatures of 1550 °C, but also the possibility of adjusting their physical properties by modification of the

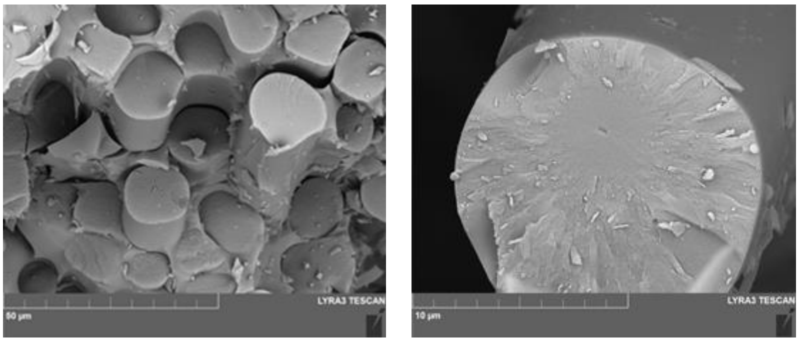
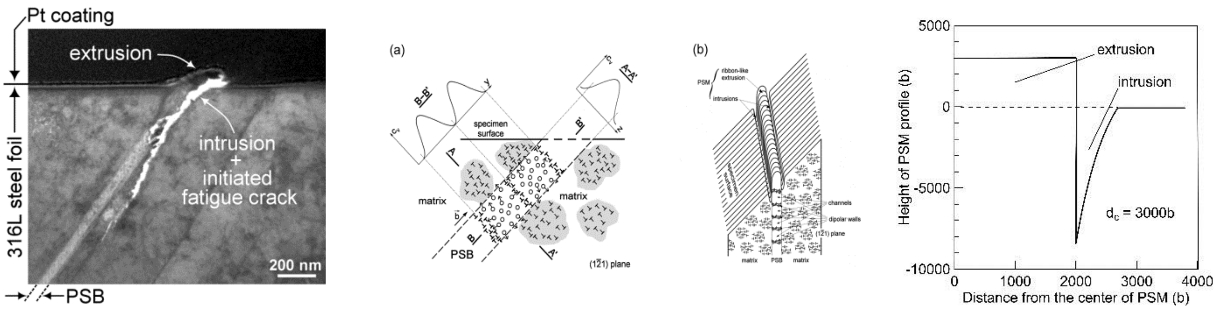


Fig. 2. Composite/fibre fracture surface showing the fibre pull-out.

resin composition. Targeted tailoring of matrix properties obtained from the instrumented indentation tests, and other methods allow optimizing of the composite function, where the basic toughening mechanism is the pull-out, which is clearly visible in Fig. 2. Thanks to the undertaken properties optimisation of the fibre-matrix interface, high values of fracture toughness exceeding 20 $MPa.m^{1/2}$ were achieved. These values of fracture toughness are comparable with top materials prepared by more expensive approaches. The research was supported by two joint projects GAP106/09/1101 and GAP107/12/2445 of the Czech Science Foundation. The results of the investigation are well summarised in number of impacted journal papers (more than 10 over the last 5 years, e.g. Halasova et al., Journal of European Ceramic Society, 2012; Chlup et al., Journal of the European Ceramic Society, 2014) and presented on numerous conferences.

Initiation of fatigue cracks

The initiation of fatigue cracks represents an important stage of the fatigue process which results in sudden fracture of structural components. The mechanisms of fatigue crack initiation have been studied since the beginning of the 19th century but important advancement has been reached only recently by applying modern experimental high resolution techniques like TEM, AFM, FEG-SEM, FIB, EBSD etc. This basic problem has been studied both experimentally and theoretically. Experimental studies of the surface relief evolution, Fig. 3a, internal structure changes and its temperature dependence in several model and structural materials lead to the deeper insight in the processes of the early fatigue damage which result in the initiation of fatigue cracks. The knowledge of the internal structure of persistent slip bands (PSBs) and kinetics of evolution of persistent slip markings (PSMs - extrusions and intrusions) with the knowledge and analysis of the properties of crystal defects allowed to propose quantitative model describing the initiation of fatigue cracks in the bands of localized cyclic slip, Fig. 3b (Polák J., Man J. Mater. Sci. Eng. A 596, 2014, 15). This model is based on the production, annihilation and migration of point defects in the bands of localized slip, the formation of inverse internal stresses in PSB and the matrix leading to PSMs. Their predictions, Fig. 3c are in good agreement with numerous experimental observations. It represents significant advancement in the field of the basic fatigue mechanisms and it helps us to analyse fatigue phenomena in structural and advanced materials.



(a) (b) (c)

Fig. 3. (a) FIB produced TEM lamella, (b) model of PSM formation, (c) prediction of the shape of PSM.

Lifetime prediction of polymer structures

The portfolio of investigated materials was widened during the last years. As an example the investigation of the fatigue behaviour of polymer structures is here described. New applications of the polymers in many cases replace metal components, and in some special loading situations modelling of crack propagation in polymer materials has similar principles as modelling of fatigue crack propagation in metals. Complex methodology for lifetime prediction of the polyolefin pipes was developed in co-operation with University of Leoben, see e.g. P. Hutař et al., Engineering Fracture Mechanics 108 (2013) 98 – 108. The developed methodology combines numerical estimation of the stress intensity factor with fatigue testing of the polymer materials, in order to be able to estimate the lifetime of a pressured component without time-consuming hydrostatic pressure tests (lifetime of the modern pipe materials is predicted from 50 to 100 years). Slow crack growth (SCG) in pressurized PE pipes usually starts at stress concentrations caused by material imperfections like impurities, cavities or flaws at or near the inside pipe surface. Originating from such initial defects, the quasi-brittle failure mechanism of SCG is characterized by a continuous formation and breakdown of crazes inside localized process zone. To describe slow crack propagation, the accelerated data from creep tests at elevated temperature (80 °C) are usually used. The novel concept of the extrapolation procedure in which the kinetics of fatigue crack growth is measured using crack round bar CRB specimens at different loading ratios R (ratio of minimum load to maximum load at fatigue cycle) was originally developed at University of Leoben and now implemented by researchers of the Institute. Crack propagation rate for different R-ratios was experimentally evaluated and then extrapolated to R = 1, corresponding to static loading. This procedure is relatively simple, time-efficient and takes into account the combination of creep and fatigue loading. Using these experimental data, numerical simulations of 3D crack propagation in polymer material were defined and lifetime of the polymeric structure was evaluated. Special algorithm for numerical estimation of the crack front was developed to be able to simulate crack propagation accurately. The methodology shows the increasing strength of the Institute based on the combination of extensive experimental fatigue testing with complex 3D numerical simulations of crack type damage.

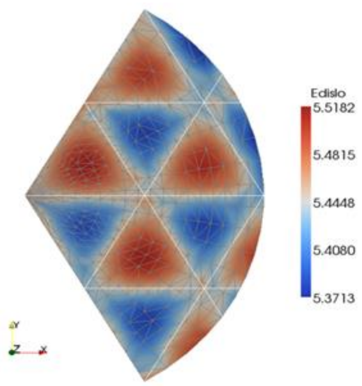
Lead free soldering

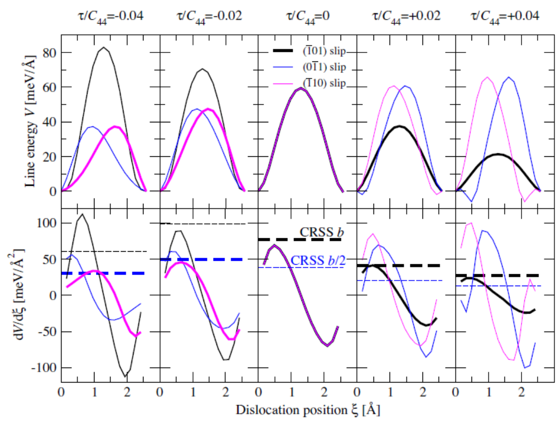
One of the very fruitful directions of the research in the Institute is modelling of phase diagrams of complex metallic materials. This investigation has a long tradition and is internationally well recognized. During the last years, the new knowledge about materials suitable for lead free soldering was acquired. Ales Kroupa lead as the Chair of Management Committee the international COST MP0602 project “Advanced Solder Materials for High Temperature Application (HISOLD)” in the years 2007-2011 oriented to the study of the lead-free materials suitable for high-temperature soldering. The lead-containing materials are still used for HT soldering (in the temperature region between 250 and 350 °C) because of the lack of non-hazardous substitution and as such they are currently exempt from the EU legislation about restriction of hazardous materials. More than 60 research teams from 20 EU countries and from Russia, Ukraine and Argentina took part in this project and presented important information about possible lead-free materials and/or new lead-free technologies usable in this field. The results of the project were published in the form of 3-volume publication “Handbook of High-Temperature Lead-Free Solders” by the COST office.

A. Kroupa and his research team at the Institute were co-authors of the volume 1 – “Dinsdale A., Kroupa A., Watson A., Vrestal J., Zemanova A., Broz P.: Atlas of Phase Diagrams”, together with colleagues from National Physical Laboratory in Teddington, University of Leeds and Masaryk University, where the phase diagrams of binary and ternary systems important for possible lead-free solders were published. These diagrams were calculated by the CALPHAD method using the thermodynamic database developed by the co-authors in the scope of the project using the data from the literature and from their own scientific research. This database is now used by many research teams for their own research projects. A. Kroupa was also the editor of the volume 3 – “Group Project Reports”, where the overview of scientific result obtained in the scope of broad international co-operation was presented. The Volume 2 of this publication "Materials Properties" lists the values of thermophysical properties and some mechanical properties obtained in the scope of the project by various research teams.

Computational studies of atomic configurations of defects

Computational studies of atomic configurations of defects in materials represent an important part of the current materials research. In this field the Institute has traditionally good international position. One of the research teams of the Group of Electrical and Magnetic Properties together with international partners have recently developed a novel theoretical model that allows for rapid calculation of the Peierls barriers of dislocations and their dependencies on the applied stress (Gröger R, Vitek V. Model Simul. Mater. Sci. Eng. 2012, 20, 035019). This is one of two crucial ingredients that were missing to link the atomistic studies of isolated screw dislocations in body-centered cubic (bcc) metals with the macroscopic response of the material, as studied in crystal plasticity finite element models (CPFEM). The second missing input is the knowledge of the curved dislocation pathway that would go beyond the traditional simplification of a straight dislocation path (also adopted above). We have shown recently that this curved path can be calculated from relaxed





(a) (b)

Fig. 4. Map of the Peierls potential of 1/2<111> screw dislocation in bcc W obtained by direct molecular statics simulations (a). The Peierls barriers for glide of the dislocation on the three {110} planes of the [111] zone calculated for different applied shear stresses perpendicular to the slip direction are shown in (b).

atomic coordinates by generalizing the Peierls-Nabarro model (Gröger R, Vitek V. Mat. Sci. Eng. A (2015); paper under review). The Peierls stresses and related choices of the glide planes determined from the Peierls barriers agree with the results of molecular statics calculations (Gröger R, Bailey AG, Vitek V. Acta Mater. 2008, 56, 5401). Knowledge of the Peierls barrier and its dependence on the applied stress completes the development of a thermodynamic description of dislocation glide by the formation of pairs of interacting kinks. This has been used for the first time to develop a Discrete Dislocation Dynamics (DDD) model of plasticity in bcc metals that allow for modelling of interacting dislocation networks (Srivastava K, Gröger R, Weygand D, Gumbsch P. Int. J. Plast. 2013, 47, 126-142). It is shown that such an atomistic based description of the dislocation mobility provides a physical basis to naturally explain many experimentally observed phenomena in bcc metals like the tension-compression asymmetry, the orientation dependence of loading, temperature dependence of the yield stress and the crystallography of slip.

Grant and programme projects

The Institute as a whole was successful in acquiring a grant support from various grant agencies. The researchers of the Institute solved more than 100 grants (see details in the Appendix 3.1). The overall subsidy for the evaluated period of 5 years was of about EUR 14 million. The management of the Institute together with the Board of the Institute made an effort to submit only research projects which fit well into the main research direction of the Institute and which can utilize the existing experimental facilities.

From the point of view of the research stimulation, the most important are the projects of the Scientific Foundation of the Czech Republic. The vast majority of publications in scientific journals with impact factor were results of the solution of projects supported by just this Agency. On the other hand, the projects with subsidy of Operational Programme Research and Development for Innovations and Programme for Educational Co-operations contributed substantially to the engagement of young post-doctoral researchers including some form the foreign countries. In this respect also EU projects GlaCERCo and RoLicer were highly beneficial.

The overall research output of the Institute was strongly supported by the investments into the large experimental facilities (JEOL TEM, Field Emission Electron Microscope JEM-2100F, equipped by Oxford Aztec X-Max 80 Premium EDS, electrohydraulic computer controlled axial/torsional test system MTS 809, two creep-electromechanical machines Messphysik KAPPA LA and Maytec furnaces, system for measurement of electrical, magnetic and transport properties of materials in temperature range 2-300 K) procured with support of CEITEC IPM.

The general scientific output or the Institute, measured by the number of publications in scientific journals with impact factor, was 382 items and 339 publications in proceedings of conferences indexed in WOS and SCOPUS.

Research for practice and collaborative research are traditional activities of the Institute. The amount of the research for practice expressed in kEUR was 850 in the evaluated period, and in the sphere of the collaborative research kEUR 340. However, behind these amounts, there are valuable results for industry; part of them is naturally confidential and these results were not published. On the other hand, some collaborative projects with some companies enabled publication of results in open scientific literature.

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Advanced High-Temperature Materials Group

Response to the last evaluation

During the period 2010-2014 all younger scientists have been much more involved in the research of the team. They have been the investigators and/or co-investigators of 4 research projects, members of the research teams in other 6 grant projects and they were involved as authors and/or co-authors in the most publications of the group. In this year all of them applied for the new grant projects for the Czech Science Foundation. The research field of the team has been expanded into both new types of materials investigated (e.g. zirconium and titanium alloys, discontinuously reinforced metal matrix composites, ODS alloys, nickel-based superalloys, new types of intermetallics) and the creep response of those materials. A further upgrading of the data acquisition and computer processing in the creep laboratories have been done. Two new creep testing machines for temperature up to 1200 °C are shared with the Creep of Metallic Materials group within CEITEC-IPM project.

Achieved results

Factors influencing creep strength and ductility in ultrafine-grained metallic materials

Creep strength and ductility are the key creep properties of creep-resistant materials but these properties typically have opposing characteristics. Thus, materials with conventional grain sizes may be strong or ductile but there are rarely both. In this connection, recent findings of high strength and good ductility in several submicrometer-grained and/or nanostructured metals and alloys are of special interest. Processing through the application of severe plastic deformation are currently available but the most attractive technique for producing bulk ultrafine-grained (UFG) materials is equal-channel angular pressing (ECAP). Thus, there is a potential for using pressed material to obtain new flow processes in high temperature creep. At present, only very limited reports are available, describing the creep behaviour of these materials. Therefore, our study was initiated to provide systematic information on the creep behaviour and microstructural characteristics of the selected metallic materials. The creep behaviour of aluminium, copper and their precipitation strengthened alloys, iron, titanium and zirconium alloys were examined after processing by ECAP with an emphasis on creep strength and ductility and the ECAP microstructural homogeneity. It was found that the creep resistance of most investigated UFG materials is considerably increased in comparison with their coarse-grained state already after first ECAP pass (Fig. 1a). However, successive

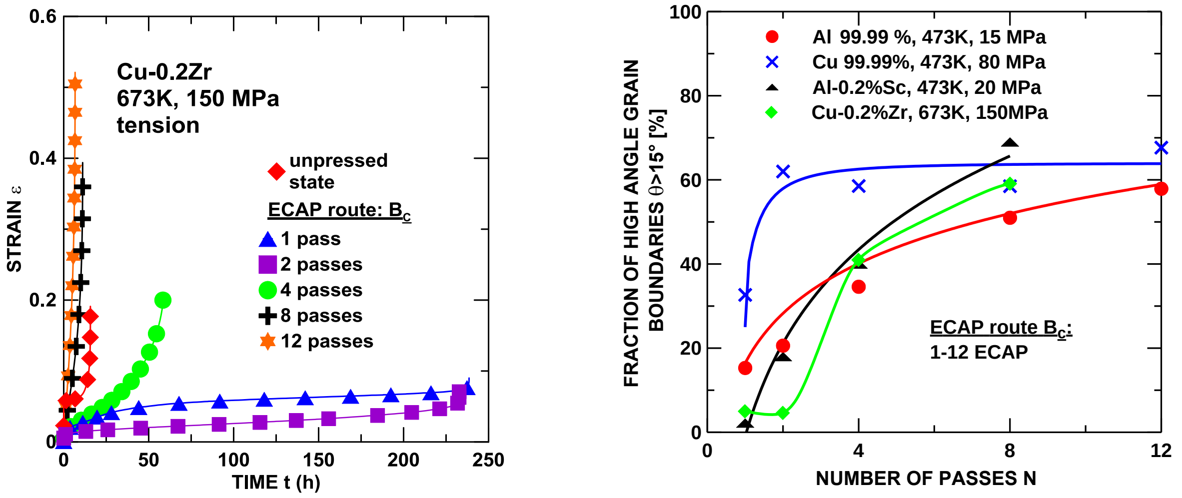


Fig.1 (a) standard creep curves of Cu-0.2wt.%Zr alloy for unpressed state and various number of ECAP passes (1,2,4,8,12), (b) the fraction of high-angle grain boundaries as a function of the number of ECAP passes.

ECAP pressing leads to a noticeable decrease of the creep properties. By contrast, it was found that, under the same loading conditions, the creep ductility of the UFG materials continually increases with increasing numbers of ECAP passes. A detailed quantitative microstructural study was conducted using the electron backscatter diffraction methods. This analysis revealed that, with increasing number of ECAP passes, the mutual misorientation of neighbouring subgrains grows and the subgrains continuously transform to grains having high-angle grain boundaries (Fig. 1b). The softening by high-angle grain boundaries may be explained in terms of indirect effect which grain boundaries exert on the creep resistance by influencing the evolution of the dislocation structure in modifying the rate of generation and annihilation of dislocations.

High-Temperature creep flow of a Zr-1wt.%Nb alloy

Zirconium-based alloys are currently used as cladding tubes in the fuel assembly of nuclear light water reactors. Although the normal operating temperature is lower than 673 K, one must ensure integrity and avoid excess ballooning of cladding tubes up to high temperatures and for high stresses under Loss-of-coolant-accident (LOCA) conditions. Such LOCA conditions have long been considered as a hypothetical accidental scenario involving high temperature cycles. Unfortunately, a possibility of real occurrence of such accident has been demonstrated by very recent disaster of the Japanese Fukushima Daichi nuclear power plant that has been blamed on a failed cooling system. The Zr-1wt.%Nb alloy has been used as one of the key structural material for fuel claddings. At present, there appears to be some insufficient quantitative creep data in the open literature describing the creep behaviour of this alloy. In our project uniaxial constant stress tensile creep tests were carried out at 823-1123 K on specimens taken from the Zr-1wt.%Nb cladding tube. For constant stress creep experiments, the minimum creep rate used to be considered as one the of main characteristics, since it represents the instant in the microstructural development when the accelerating processes, represented mainly by creep damage development, start to overweight work strengthening. Representative creep data are shown in Figs. 2a and 2b. Examination of Fig. 2a leads to following observations: First, the stress dependences of the minimum creep rate for all temperatures have the same trend. However, the slopes and therefore the

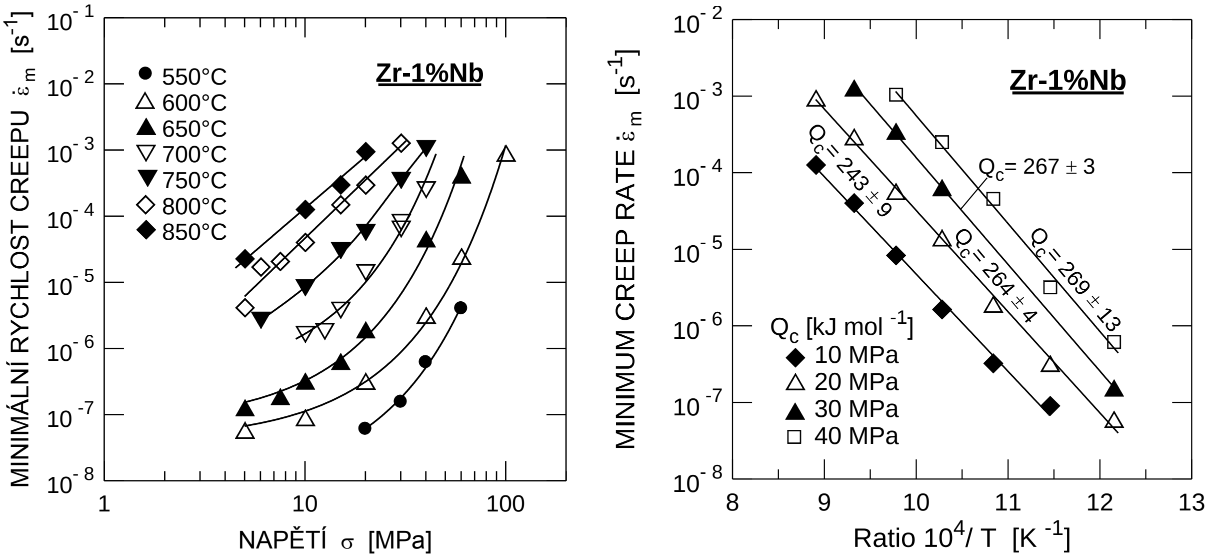


Figure 2: (a) stress dependences of the minimum creep rate and, (b) activation energy for creep Q_C .

apparent stress exponents n of creep rate for low and high stress regions are slightly different. Second, the decrease in n at the lower stresses may be indicative of some changes in the rate controlling creep deformation mechanism(s). The value of stress exponent n ~ 2.3 indicates of creep controlled by grain boundary sliding. At higher stress regime n ~ 6.3, which is within the range of so-called five-power-law creep regime, and suggesting dislocation glide and climb as a dominating creep deformation mechanism. Further, if creep of an Zr-1wt.%Nb alloy in the power-law region is controlled by dislocation climb then the activation energy for creep Q_C should be fairly consistent with that of self-diffusion. The determined activation energies of 243-269 kJ/mol (Fig. 2b) fall within the range of self-diffusion activation energies reported in the literature.

Modelling of thermodynamic processes in advanced materials

The modelling is based predominantly on the application of the Thermodynamic Extremal Principle (TEP), which represents a handy tool for the treatment of irreversible linear phenomena in solids like diffusion, grain growth or diffusional phase transformation. An overview paper about thermodynamic extermal principles has been published in Acta Materialia in 2014.

Several models concerning a number of different topics have been developed during years 2010-2014. They deal with:

- reaction of metallic nanospheres with oxygen leading to the formation of oxide hollow nanospheres,
- influence of trapping on diffusion of interstitial components significantly influencing the diffusion kinetics,
- grooving and motion of triple junctions being important phenomena in grain growth and coarsening,
- formation of denuded zones due to coarsening of precipitates at grain boundaries and significantly degrading the mechanical properties,

- solute drag in grain boundaries and interfaces being responsible for a drastic retardation of the grain growth and/or causing the abnormal grain growth,
- derivation of the phase field equations from TEP showing the link between irreversible thermodynamics and phase field method,
- application of the trapping concept to the diffusion of hydrogen in steels demonstrating that the traps represented by dislocation cores, grain boundaries, foreign atoms or incoherent interfaces of carbides can decrease the effective diffusion coefficient by several orders of magnitude,
- influence of stress state and intensity of sources and sinks for vacancies on diffusion kinetics indicating that inactive sources and sinks for vacancies can decrease the diffusion kinetics by more than one order (overview paper published in Progress in Materials Science in 2014).

Development of new advanced creep resistant materials

New ODS alloys and composites with the Fe-10wt%Al matrix have been developed. The alloys are prepared by mechanical alloying of Fe and Al powders in an oxidizing atmosphere, the composites by mechanical alloying of Fe, Al and Al_2O_3 powders in vacuum. Then the mechanically alloyed powders are deposited in a steel tube, evacuated, sealed by welding and consolidated by hot rolling. The resultant alloy exhibits an ultrafine-grained microstructure reinforced and stabilized by 6 vol.% of Al_2O_3 precipitates of the size about 50 nm, the composite is reinforced by 10 vol.% of Al_2O_3 particles of size about 300 nm. Both materials exhibit an excellent oxidation resistance up to temperatures of 1300 °C. The prize of raw materials is extremely low. A patent application was submitted.

Advanced ferritic chromium steels strengthened by carbides and vanadium nitrides are used for a construction of boilers in thermal power plants. Any increase of the operation temperature leads to the increase of efficiency of the power plant and thus to the increase of the protection of environment and climate. Our team is involved in an European project, which pursuits a new idea to replace the vanadium nitrides by finely dispersed Z-phase, which is more stable, and this enables increasing of the operation temperature of the boiler. Our team deals with the development of thermodynamic models for nucleation, growth and coarsening of precipitates, for creep, and also creep is tested experimentally in welded and non-welded specimens of the developed material. The creep behaviour of advanced chromium steels has been studied also within the European 7RP project MACPLUS.

The shape memory research

In the period 2010-2014, prof. Antonin Dlouhy and his doctoral and post-doctoral students focused on three main scientific topics: (i) high-temperature strength and plasticity of advanced intermetallics and in-situ eutectic composites, (ii) structural and functional properties of shape memory alloys based on the NiTi intermetallic, and (iii) development of a 3D discrete dislocation model that has an ability to address dislocation processes in precipitation hardened crystals subjected to loadings at elevated and high temperatures. An intensive international collaboration contributed considerably to a successful solution of these research tasks. As an example, we present a short description of results achieved in the area of the shape memory research:

1) In situ and post-mortem diffraction contrast TEM was used to study the multiplication of dislocations during a thermal martensitic forward and reverse transformation in a NiTi shape memory alloy single crystal. It is proposed that the

stress field of an approaching martensite needle activates an in-grown dislocation segment and generates characteristic narrow and elongated dislocation loops which expand on {1 1 0}B2 planes parallel to {0 0 1}B19' compound twin planes. It is suggested that the type of dislocation multiplication mechanism documented in the present study is generic and that it can account for the increase in dislocation densities during thermal and stress-induced martensitic transformations in other shape memory alloys.

2) Phase transformations in binary ultrafine-grained (UFG) pseudoelastic NiTi wires were studied in a wide temperature range using mechanical loading/unloading experiments, resistance measurements, differential scanning calorimetry (DSC), thermal infrared imaging, and transmission electron microscopy (TEM). The stress-induced B2 to R-phase transition occurs in a homogeneous manner, contrary to the localized character of the B2/R to B19' transformations. A stress-temperature map is suggested to summarize the experimentally observed sequences of trans-formation/deformation processes.

3) We have reported results of differential scanning calorimetry and TEM experiments in which martensitic transformations and microstructure of Ti–50.9 at %Ni shape memory samples were investigated after heat treatments in controlled gaseous environments. DSC charts document that the B2 → B19' or R → B19' martensitic phase transitions diminish after treatments with increasing partial pressure of hydrogen. We have suggested that the gradual suppression of the martensitic transformation is related to either chemical and/or strain modulations of the B2 lattice triggered by the presence of interstitial hydrogen atoms.

4) In a close relation to the experimental results we have employed density functional theory to show that H interstitials exhibit negative formation energies, while Ar and He interstitials yield positive values. In agreement with experimental data, we predict H atoms to have a strong impact on the martensitic phase transformation by altering the mutual thermodynamic stability of the high-temperature cubic B2 and the low-temperature monoclinic B19' phases, see Fig. 3.

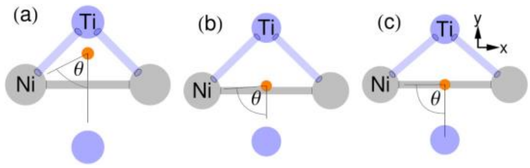


Fig. 3 Relaxation of the hydrogen atom (orange) from a tetrahedral site (a) towards an octahedral site (b,c) as predicted by the DFT calculations.

Description of the projects solved in the team, collaboration with others teams

MPO CR:FR-TI4/406 : Research of the influence of orbital head welding technology of thick-walled tubes/pipes on their long-term lifetime in condition of modern power plant service

The aim of this project is to verify the application of the progressive welding methods using orbital head in term of their influence on long-term creep behaviour of thermally loaded thick-walled pipes made of P91 and P92 chromium creep-resistant steels.

TA CR:TA02011025 : Creep and oxidation properties of E110 cladding tube under LOCA temperature transient

The main aim of the project is to implement the creep characteristics of E110 cladding tubes in LOCA temperature range into the FERMAXI software and to create the diffusion oxidation model.

TA CR:TA02010260 : Research of materials changes occurring in advanced steels used for construction and reconstruction of pipelines in advanced power and chemical plants

The main aim of the project is to determine the materials properties of steam pipes made of advanced creep-resistant steels, especially in terms of effect of materials changes on the resulting creep lifetime.

EC:FP7 Energy.2009.6.1.1.: Material component performance driven solutions for long-term efficiency increase in ultra supercritical power plants (MACPLUS)

The main aim of the project is to describe type the IV cracking mechanisms of advanced martensitic steels, austenitic steels and Ni-base alloys and the description of creep-fatigue behaviour of metallic materials.

MPO CR:2A-1TP1/057: Solutions of materials and technological innovations of new generation power and chemical plants operating at high temperatures

Creep behaviour of selected advanced power plant steels after a model long-term isothermal ageing has been studied with the aim to simulate the creep strength degradation which occurs under operating conditions.

MCL: A1.17: Inverse process chain modelling for Al-castings and induction heat treated steel rods

The main aim of the project is the inverse optimization of two process chains (i) ‘casting-solution annealing-quenching-aging’ and, (ii) ‘straightening-inductive austenitizing-quenching-inductive tempering-air cooling’.

CSF: 14-24252S: Preparation and optimization of creep resistant submicron-structured composite with Fe-Al matrix and Al_2O_3 particles

The main aim of the project is to develop a processing method leading to a uniform distribution of oxide particles of proper size and volume fraction in the matrix of the composite.

MCL: A2.26: Development of new-generation ODS alloys and ODS composites

The project deals with the development of new ODS alloys and ODS composites with the Fe-8-10wt%Al matrix. The alloys are prepared by mechanical alloying of Fe and Al powders in an oxidizing atmosphere, the composites by mechanical alloying of Fe, Al and Al_2O_3 powders in an inert atmosphere.

EC: FP7-309916-2: Z-phase strengthened steels for ultra-supercritical power plants

The project pursuits a new idea to replace the vanadium nitrides by finely dispersed Z-phase, which is more stable, and this enables increasing of the operation temperature of the boiler.

MCL: A1.9: The impact of the atomic trapping on diffusion and phase transformation kinetics

The main aim of the project is to judge the influence of the atomic traps on kinetics of diffusion of interstitial components and consequences to kinetics of diffusional phase transformations.

MCL: A6.14: Service security of welded high-strength pressurized pipelines

The main aim of the project is to describe the influence of the hydrogen on the strength of new steels for production of pipelines. Our team delt with development of models for hydrogen diffusion.

CSF: 108-10-1781: The role of stress state and vacancy supersaturation at the formation of binary hollow nanoparticles

The main aim of the project is to elucidate the mechanisms of the formation of hollow nanospheres by means of gas-solid reaction of metallic nanospheres with an external gas atmosphere.

MSMT CR: OC10029: Thermodynamic modelling of microstructure evolution in nanocomposites
The main aim of the project is to apply the thermodynamic extremal principle for description of solute segregation and solute drag by means of a small number of parameters, which opens the way for application of the models on the complex system as a polycrystal.
CSF:P204-10-1784: Modelling of diffusional phase transformations in multi-component systems with multiple stoichiometric phases
The main aim of the project is to treat effectively the interdiffusion being responsible for yielding the so-called "Kirkendall effect" and explain polyfurcation of the Kirkendall plane.
MŠMT CR:CZ.1.07/2.4.00/31.0046: NETME Working - Innovation and Technology Transfer in Mechanical Engineering
The project is focused on the strengthening of the relations among the different types of educational institutions, research institutions and the private industrial sector through cooperation between the subjects.
CSF: 106/09/1913:Martensitic transformation in NiTi alloys
The project is focused on the transformation path and the microstructural reason for pronounced changes of hysteresis loops during functional fatigue of superplastic NiTi wires.
CSF:202/09/2073: Deformation mechanisms of in-situ composite materials
The project is focused on a size effect of the submicron reinforcement, load transfer and the role of interface phenomena.
CSF: 14-22834S: Phase stability and plasticity in medium-to-high-entropy alloys
The project aims at the understanding the high-temperature stability and strength of these systems, and how the transition from quinary system to quaternary sub-system influences the mechanical properties.
AS CR-RAS: 8/12 Microstructural features and mechanical properties of nanocrystalline titanium
The aim is to investigate the creep behaviour and properties of pure nanocrystalline titanium.
AS CR-RAS: 9/12 Investigation of creep and fatigue behaviour of metallic nanomaterials
The main aim is to investigate the creep behaviour, damage and rejuvenation of selected nanomaterials.
KJB200410801 Study of nano-structure materials consolidated from power compacts
The main aim of the project is the processing of bulk materials from metallic powders by means equal-channel angular pressing.

There are very intensive informal (discussions) and formal collaboration with the teams or scientists of the other institute groups. Formal collaboration means that some projects running within this team incorporate in their research team also individual members of the other institute teams. The most intensive collaborations are carried out with following teams: Creep of Metallic Materials (creep testing at extremally low stresses), High Cycle Fatigue Group (creep-fatigue interaction in nickel-based superalloys) and Structure of Phases and Thermodynamics Group (microstructural analysis and modelling of creep exposed specimens).

It should be noted that some experimental research activities, especially analytical quantitative microstructural investigations and very high temperature creep testing, were performed using CEITEC-Central European Institute research infrastructure supported by the project CZ1.05/1.1.00/0.2.0068 financed from the European Regional Development Fund. At the same time, the salaries of mostly younger research workers of the team have been covered in part from the start-up of this project.

International co-operation

The group co-operates with many international research teams from scientific institutes and companies. In the period 2010-2014 the team has participated in 2 international EC FP7 projects and 6 bilateral international projects (4 with Materials Center Leoben, Austria, 1 with the A.F. Ioffe Physical-Technical Institute, RAS, St. Petersburg, Russia, 1 with the A.A. Baikov Institute of Metallurgy and Materials Science, Moscow, Russia). As it is obvious from the publication output of the team (more than 45 joint publications with foreign partners), the intensive international collaboration contributed to obtain, discuss and interprete the results. We have shared the effort with colleagues from University of Southern California (USA), Ohio State University (USA), Oak Ridge National Laboratory (USA), Centro Atomico Bariloche (Argentina), Hanyang University (South Korea), Max Planck Institute Duesseldorf (Germany), Ruhr University Bochum (Germany), University of Erlangen-Nuernberg (Germany), Materials Centrum Leoben (Austria), Montan Universität Leoben (Austria), Vienna University of Technology (Austria), University of Vienna (Austria), Ioffe Physical-Technical Institute RAS (Russia), Institute of Metallurgy and Materials Science RAS (Russia), Belgorod State University (Russia), Institute of Materials and Machine Mechanics SAS (Slovakia), Welding Research Institute (Slovakia), etc.

New facilities in the laboratories

The most of experimental facilities are shared with Creep of Metallic Materials Group. During the period 2010-2014 the true strain-time analog and digital continuous readings of creep elongation during creep exposures and PC-based acquisition system have been periodically upgraded. New equipment for calibration of creep furnaces to maintain long-term testing temperature within 0.5 K has been installed. The development of new ODS alloys and composites requires a powerful attrior for mechanical alloying of powders. As there are special requirements of amount, milling energy, atmosphere of vacuum and handling of powers, a new ball mill has been designed in the laboratory of our team and its fabrication in institute’s mechanical workshop is in progress. Two new creep testing machines Zwick Z250 with vacuum system enable creep exposure at temperatures up to 1200 and 1400 °C, respectively, and load up to 50 kN. These testing machines allowing experiments both under controlled stress or controlled load are shared with CEITEC-IPM and group of Creep of Metallic Materials.

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Brittle Fracture Group

Response to the last evaluation

The recent scientific reputation of the group is nicely reflected on its invitation to consortia of the top European Institutes and the participation of the key Group members in EU projects GlaCERCo, RoLiCer, EurAtom, and Coach. This fact stimulated additional scientific growth of the younger Group members (comparably to other Institute groups), it has also contributed to higher number of high quality publications and further increased the potential of the Group for making internationally acknowledged science in the field of micromechanics and micromechanisms of fracture.

The Group indisputable authority in the field is also shown by attraction of the long term stays of the foreing scientists: prof. M. Hasegawa from Yokohama National University – a year at IPM, prof. K. Chawla from University of Alabama at Birmingham - two months, two Ph.D. students, F. Siska from Deakin University - for permanent job.

With increasing number of high quality publications, the number of citations have also substantially increased. Head of the Group was asked to guarantee summer schools within European framework in the field of micromechanics and micromechanisms of fracture.

Achieved results

Bioglass® Scaffolds Reinforced by a Composite (PVA/MFC) Coating

For scaffolds to be used in tissue engineering and applications in human body, a mechanical response in terms of rigidity and fracture resistance is an important parameter, in addition to other key issues. Based on previous experience of the Group in this field, the effort has been focussed on how to improve the response of the foam to mechanical loading either by modification of the foam structure preparation, or by an application of the (polymer biocompatible) coating onto the foam structure.

For the foam structure preparation from Bioglass® powder, new ethanol based slurry formulation was suggested and applied. It resulted in the scaffolds formed by an open cell Bioglass® foam structure with uniform struts and high degree of open porosity and, at the same time, very good strength in tension and compression.

Damage of the structure during mechanical loading in tension has been quantified using computer tomography and consequently by finite element modelling. Struts loaded in bending were identified as a critical event. However, the surface quality of struts, as a consequence of the foam structure fabrication, is the most critical variable for the mechanical response. Coatings by polyvinyl alcohol (PVA) and composite of PVA with cellulose microfibrils (MFC) have been found as an optimum solution to improve it. Based on the rheology investigation of the polymeric solutions in order to achieve a more homogeneous distribution of the composite film on the struts surface and to enable an effective penetration and filling of the central hole in the struts, an optimum MFC/PVA ratio has been determined.

The PVA-coated samples exhibited approximately more than 5 fold increase of compressive strength compared to uncoated ones and the addition of 5 wt. % of MFC fibers led to a further increase (of the compressive strength). Also tensile strength has been found to be improved by the PVA/MFC hybrid coating; the samples with PVA/MFC coating exhibited more than 10 fold increase of tensile strength compared to the un-coated samples (Fig. B1). SEM observations showed how PVA is able to infiltrate within the struts surface defects by hindering the stress concentration and providing stress transfer from the scaffold to the MFC fibers. In addition, fracture of MFC fibers contributed to the energy dissipation process which led to the increase of the toughness of the scaffolds.

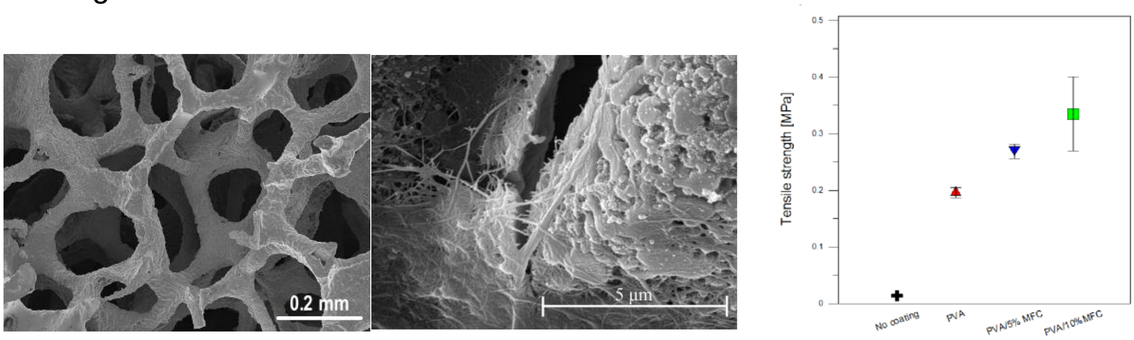


Fig. B1 (a) Structure of the bioglass foam with coating, (b) crack bridging by fibres in composite coating and (c) tensile strength properties increase for different coating including PVA/MFC composite

The findings have been generated by the Group as the one particular task within the GlaCERCo EU project (Glass and Ceramic Composites for High Technology Applications – Initial Training Network, FP7-PEOPLE-2010-ITN, 264526). The computer tomography images have been obtained in collaboration with N. Chawla (Arizona State University) and biocompatibility issues have been consulted with A. R. Boccaccini (University of Erlangen-Nuerenberg). The works in this field have been supported also by projects of Czech Science Foundation Nr. GA101/09/1821 and 13-07425S

Laminate Structures – Design, Preparation and Fracture Characterisation

The precise and comprehensive work of the team in the field of ceramic based laminates prepared by various methods was achieved during the last years. The Group covered most of the activities in the development and characterisation of enhanced ceramic based layered structures, which is documented by significant publication activity and supported by number of projects. The leadership of the Group can be seen mainly in (i) the field of understanding the processes important for design and preparation of the laminates by electrophoretic deposition and in (ii) the field of fracture behaviour understanding and crack propagation description. In the first case, the perfect knowledge of the electrophoretic deposition kinetics in the relation to quantification of the internal stresses developed during processing, this allowed to prepare theoretically based designs of individual layers with high precision and with nearly ideal interfaces as can be demonstrated in Fig. L1.

The results of work cover all aspects from the modelling and design of the laminate structures through its precise preparation using fundamental knowledge of the processing up to the description of deve- lopment of stresses in the context of resulting controlled fracture behaviour and application properties.

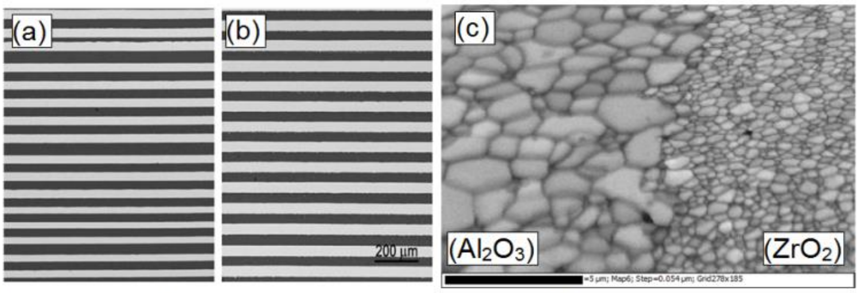


Fig. L1. 1:1 vol.% alumina/zirconia laminate prepared by electrophoretic deposition (a) before and (b) after optimisation of the deposition kinetics (SEM BSE) and (c) detailed view on the layer interface microstructure (EBSD band contrast).

The understanding of the fundamental aspects of laminated structures with strongly bonded layers incorporating internal stresses allows us to optimise the design of individual layers to achieve the enhanced properties. Basically, two approaches can be applied: i) enhancing strength and fracture resistance together or ii) predetermine threshold (minimal) strength and increase the damage tolerance. The latest effort is targeted to the second area where we have been achieving significant damage tolerance with still high strength values.

The high quality results obtained by the Group contributed to the national and international co-operation: in the field of sintering with K. Maca, Brno University of Technology and in the field of the modelling and fracture behaviour assessment with R. Bermejo from Montan University, Leoben. The development and characterisation of layered structures was carried out thanks to projects: GAP108/11/1644, GA101/09/1821, GD106/09/H035 of Czech Science Foundation and M100410902 of Academy of Sciences of the Czech Republic.

Fibre Reinforced High Temperature Composites (from design to properties characterisation)

The development and mainly the characterisation of reliable ceramic composite materials, including the fibre reinforced ones, have been investigated by the Group for decades. The research effort has been targeted to the brittle matrix composites reinforced by (nano) fibres.

As a good example of the collaboration with complementary oriented research groups, the composite materials based on polysiloxane precursors pyrolysis can be taken. The design and preparation of these materials are enabled by the beneficial cooperation with other institutes of Academy of Sciences (IRSM and IMC). They are based on our findings in the field of fracture micromechanisms quantification and investigations of modifications of the chemical composition and processing of the composite materials. Simplified aim of our activity in this field can be seen in the economically attractive preparation of high temperature resistant composite material with outstanding fracture resistance in wide range of temperatures. Some critical points in the composite optimisation have been investigated by the Group: i) the quality of matrix prepared via pyrolysis of polysiloxane resins where shrinkage and cracking due to the development of the residual stresses can be the limitation, ii) the bonding between matrix and fibre which predetermine the toughening effect, iii) the temperature stability of matrices and composites when various fibres are used and as well the loading conditions effect on the fracture behaviour. An example of the results obtained during our investigation can be seen in Fig. C1, where the crack interaction with the interface together with the temperature effect is clearly demonstrated for composite reinforced by Nextel 720 fibres.

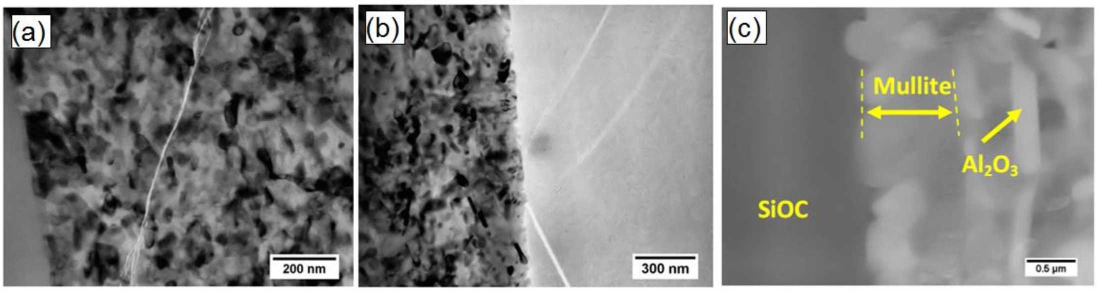


Fig. C1 Damaged area of the composite caused by exposition to the temperature of 1100 °C a) thermally induced crack, b) crack behaviour in matrix and on the fibre matrix interface and c) the microstructural changes in the vicinity of the interface after exposition at temperature of 1500°C

Findings in this field have been extensively published. The original and top results have been obtained thanks to the cooperative works with M. Černý, IRSM, in the field of composite design and production and A. Strachota, IMC, in field of siloxane resin development. The works in this field were supported financially by the common projects of Czech Science Foundation Nr. GA106/09/1101 and GAP107/12/2445.

Crack propagation modelling at microstructural level

The propagation of cracks strongly depends on the microstructure. Silicon nitride, the monolithic ceramic material, is probably the best candidate for severally loaded components. The Group team within the international network of the RoLiCer EU project investigated separate problem focused on the possibility to apply silicon nitride in the metal forming industry (rolling tools and bearings). The chal-lenges solved were connected to the crack propagation modelling on the microstructural level where nucleation and initial slow crack growth occurs. The obstacle to deal with silicon nitride is its bimodal microstructure formed from equiaxial and elongated alpha and beta grains. Their size spans from sub-micron to several microns in size. The main achievements of the Group can be seen in three levels. (i) First one is connected to the development of suitable metho-dology allowing creation of the realistic 3D model of the microstruc-ture using EBSD and FIB slicing technique (see Fig. M1), further used for calculation of elastic and thermal properties by FEM. (ii) The other one is the development of the probability damage model of the crack propagation through the microstructure, especially the influence of the grain boundaries interface in the crack development. The damage model was elaborated for four selected silicon nitride materials with various elongated grain structure (microstructure) predetermined for rolling elements and balls in the bearings. An example of the model based on the geometrical interaction between grains and the crack is displayed in Fig. M2 and an example of the typical fracture surface is added for ilustration. (iii) Finally, based on the gained knowledge about the microstructure and fracture behaviour, a prediction of the crack initiation and propagation using the developed traction-separation law for cohesive

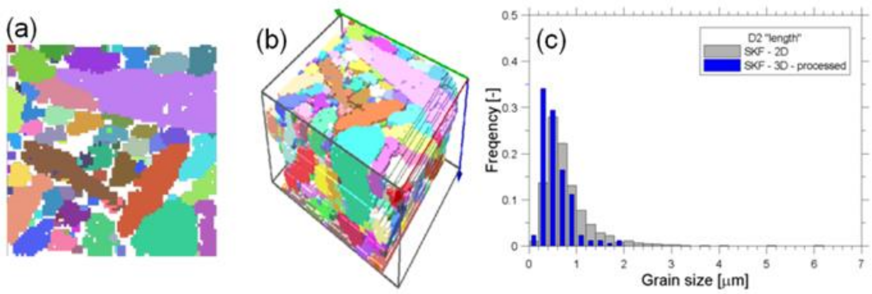


Fig. M1 Microstructure reconstruction by EBSD technique a) 2D slice, b) 3D body and c) comparison of the grain length obtained from 2D and 3D reconstruction.

zone method incorporated in to the FEM code was succefully conducted. The R-curves were obtained and compared with the experimental results.

The high quality results were achieved as a separate subtask of the RoLiCer project (“Enhanced reliability and lifetime of ceramic components through multi - scale modelling of degradation and damage“, FP7, under number 263476) and thanks to its financial support. There was a strong international co-operation with other consortium partners (Fraunhofer IWM, FCT Ingenieurkeramik GmbH, Karlsruhe Institute of Technology) in form of materials supplementation, use of the findings in other subtasks bringing the necessary feedback for the model optimisation during its development.

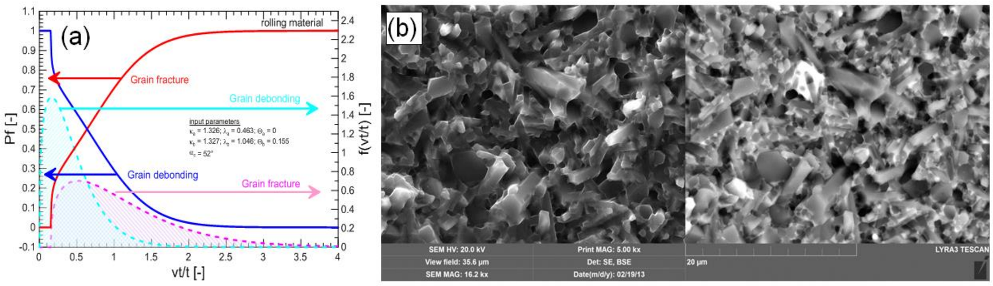


Fig. M2 a) probability model of the fracture of grain (full red line) and grain delamination (full blue line) on the geometrical grain to crack configuration represented by v_t/t ratio (left y-axis) and the probability density functions for fractured (pink dashed line) and delaminated (cyan dashed line) grains on the v_t/t ratio (right y-axis) for silicon nitride material and b) the example of the fracture surface.

Development of ODS steel for fusion applications

A big issue connected with future fusion energy power sources is the problem of structural materials compatibility. The structural components will undergo demanding service conditions as extremely high neutron doses and high temperature. The oxide dispersion strengthened (ODS) high-Cr steels alloyed by W strengthened both by solid solution (by Cr and W) and by dispersion of nanosized yttria particles are under consideration as a potential structural material for key components of fusion reactors. Due to fabrication route, the ODS steels show degradation of the low-temperature

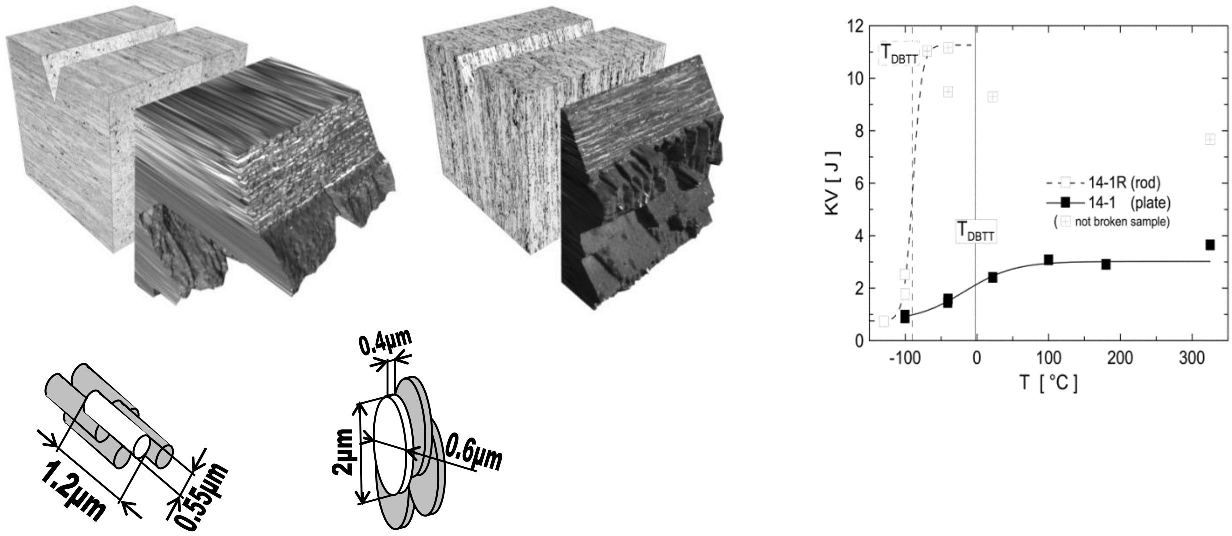


Fig. D1 Reconstruction of texture and fracture surfaces of 14%Cr-1%W ODS steel extruded as bar (having elongated grains) and as plate (having flat grains) and difference in fracture behaviour between rod-shaped and plate-shaped ODS steel.

fracture behaviour comparing to the conventional steels. The set of ODS steels having chemical composition of Fe-(9-18)Cr-(1-2)W-(0.2-0.5)Ti-(0.3-0.5)Y_2O_3 was prepared by mechanical alloying of powder from atomic and pre-alloyed powders and subsequent consolidation of the powder by extrusion at high temperature. It was found that pronounced morphologic and crystallographic texture of ODS steels leads to a strong anisotropy in fracture properties: the orientation of the fracture plane towards the extrusion direction significantly modifies the impact response of the ODS steel (see Fig. D1). It also was found that ferritic 14%Cr-1%W steel and ferritic-martensitic 9%Cr-2%W steel present much better fracture properties than ferritic 9%Cr-1%W ODS steel of Eurofer type considered as reference structural material for fusion reactors.

The results were obtained by the international co-operation with CEA Saclay, France (B. Fournier). The work was carried out thanks to the project GA106/08/1397 of the Czech Science Foundation and supported by the European Fusion Development Agency under the Contract of Association EURATOM/IPP.CR.

Description of projects solved in the team, collaboration with other teams

List of all projects solved in the evaluated period by the Group are in corresponding enclosure (3-1 List of grant and programme projects). The projects are in principle of four different types:

(i) European projects supported financially within 7^{th} framework program (GlaCerCo, RoliCer and EurAtom bottom up projects) and newly H2020 program (CoaCH).

(ii) Projects of the Czech Science Foundation, the Ministry of Education, Sport and Youth fot he Czech Reoublic and similar ones.

(iii) Projects of binational collaboration supported by the Academy of Sciences of the Czech Republic etc.

(iv) Project supporting the soft knowledge enhancement.

The projects have enabled to focus research interest on several topics:

1. Ceramic matrix composites development and damage investigation as a feedback for further development, optimisation etc.

Here the role of interefaces, nanofibers, aproachign step by step to added functions – self healing, high temperature resistence.

2. Ceramic laminates development and development of the way how to calculate the optimum laminate structure based on residual stress etc.

3. ODS steels development in terms of their toughness enhancement.

4. Transferability of fracture mechanical data from subsized specimen to larger, in terms of crack tip constraint phenomena.

5. Intermetallics – optimisation of the microstructrure by thermal and thermal mechanical treatment.

International co-operation

Long term co-operation is based on complementarity of topics and research interests of the Group and its partners. This is evident from the long term co-operation in the following cases:

(i) Yokohama National University (M. Hasegawa, H. Fukutomi, W. Nakao, A. Kotoji) - the thermal treatment of intermetallics investigated by Japanese partners is supported by the fracture micromechanisms investigation carried out by Brittle Fracture Group. Similarly self-healing material developed by A. Kotoji and W. Nakao has been investigated by the Group by using chevron notch technique and suggestion of self-healing index that enable the self-healingability to quantify.

(ii) University of Erlangen-Nuerenberg (A.R. Boccacini) - glass and ceramic matrix composites reinforced by carbon nano-tubes have been developed by Boccaccini's group whereas fracture and other properties characterisation have been explained by the Group of Brittle Fracture. Similar approach contributed to effective development of the composite coating for Bioglass foam as above mentioned.

(iii) Montan University, Leoben (R. Danzer, R. Bernejo) - long term co-operation contributed to development of an original approach to ceramic laminates modelling which has been found as a crucial step for the formulation of the rules for optimised laminate design.

The other long term international co-operations were possible thanks to and/or contributed to EU projects where the complementarity of the research is following directly from the principles of these projects (Politecnico di Torino, Nanoforce Ltd, CEA Saclay, Ciemat Madrid, Franhofer IWM, KIT Karlsruhe, IMR SAS Košice, IIC SAS Bratislava, Deakin University, Mines Paris).

There are needed from time to time special experiments conditioned by corresponding experimental facility not available at IPM or techniques and approaches asked from the Group by partners from abroad.

The Group is also developing activities within KMM vin – a virtual European Institute focused on the knowledge based on multicomponent materials. Some of the projects solved by the Group were initiated during the discusions in the consortium of KMM vin.

New facilities in the laboratories

Confocal microscope Olympus LEXT OLS3100 with AFM modul for documentation of the microstructure and fracture surfaces, 3D reconstruction of surfaces and their further geometrical analysis (covered from funds of Academy of Sciences, 173 kEUR).

Screw driven testing machine ZWICK Z250, load up to ± 250 kN, test temperatures from -196 to +1100 °C. Tensile, compressive and 3 or 4 -point bend tests. This strategic investment was covered by CEITEC-IPM, 231 kEUR.

High temperature chamber Zwick/Maytec/Messphysik for up to 1500 °C, vacuum or inert gas, for ± 50 kN, tensile, compression, three/point bend and compact tension tests. This strategic investment was covered by CEITEC-IPM, 403 kEUR.

Small pieces of equipment for ceramography: precise diamond saw Buhler ISOMET 5000, (from project FP7 – RoLiCer, 21 kEUR), two polishing machines Struers, (IPM, 19 kEUR) and for powder metallurgy laboratory ball milling device (IPM, 7 kEUR), and device for cryogenic milling (team projects, 4 kEUR).

High speed camera – Olympus i-SPEED 3 with extreme low light sensitivity and up to 150,000 fps recoding. (IPM and CSF proejct, 50 kEUR)

Work station – for large scale calculations HP ProLiant DL 360 (2x XEON E5-2650v2) server (team projects, 7 kEUR).

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Creep of Metallic Materials

Response to the last evaluation
The following objections were raised by the respective evaluators:

Prof. Mughrabi:

- *Addition of one or two young doctoral students would be desirable*
  Response: Two young researchers were enrolled: (i) fresh post-doctoral researcher Dr. Luptaková and (ii) doctoral student Dipl. Ing. Bártková
- *As a recommendation, it is proposed to further develop the data acquisition and processing in such a way to be able to obtain directly plastic strain rates with efficient accuracy.*
  R.: Data acquisitions systems from individual machines were connected within the internal creep net. This enables an immediate on-line processing of creep data by the researchers either in their offices or even at home
- *The creep testing laboratory is very well equipped on the first sight. However, for future work on advanced high temperature materials, new equipment, permitting long-time creep tests well above 1000°°C, would be needed.*
  R.: Two new creep machines were purchased within the CEITEC-IPM project. The machines enable testing up to 1400 °C, see paragraph "New facilities in the laboratories".

Prof. Spigarelli:

- *The importance of maintaining the Institution in this top ranking position, and the continuity of long-term accumulated databases and experience, should be taken into account while considering the strengthening both personnel and equipment.*
  R.: Two new researchers were employed (cf. above). Except two above-mentioned purchased creep machines, two new standard creep machines were manufactured in the institute's workshop.

- *The position of the Section in International context is high, thanks to the quality and innovation of the scientific results, but international co-operation could be strengthened, for example by establishing agreement with foreign universities; this latter activity does not contribute to fund raising, but can significantly contribute to the "internationalization" of the IPM.*
  R.: Dr. Dymáček joined the activities of the international Working Group for preparation of the Small Punch EN Standard. At present, he is deputy of the working group that prepares Part A (SP creep tests) of the standard.
  For other co-operations see the research plan.

Results achieved within the evaluated period

The main research activity of the group was concentrated into four areas and its description is subdivided accordingly:

a) Creep of iron aluminides

Creep of Fe-Al-based alloys was studied within the joint research projects with Charles University Prague and Technical University Liberec. Both Fe_3Al and FeAl type alloys were tested.

The mechanical properties of FeAl alloys with 39-43 at.% Al, C contents up to 4.9 at.% and Si contents up to 1.2 at.% were studied using uniaxial compressive creep at temperatures from 600 to 800 °C [1]. The study was performed within a more extensive search for the explanation of improved mechanical properties of the alloy named Pyroferal, successfully applied in many industrial branches in the former Czechoslovakia.

A set of FeAl-based alloys with different amounts of carbon was prepared. The composition of the alloys was designed to be approximately within the (i) (Fe-40Al)-C and (ii) (Fe-40Al)-Al_4C_3 vertical sections of the ternary Fe-Al-C system. It should be noted that the original alloy Pyroferal also contained silicon in amounts of about 0.5 at. %. The silicon was thus another element included in the search for the origin of the properties of Pyroferal. The stress and temperature dependence of the creep rate were determined by stepwise loading and evaluated in terms of the stress exponent and the activation energy, respectively. These quantities can be interpreted by the means of dislocation motion controlled by climb and by the presence of second-phase particles. The dislocation motion is obstructed by precipitates of carbide kappa and by particles of Al_4C_3 in the alloys with either higher content of C or of C and Si. Both carbon and silicon improved the creep resistance, but the effect of silicon was more significant.

In the composition range of Fe_3Al-based alloys, the research was concentrated on the possibilities of improving creep properties by addition of elements with low solubility in Fe_3Al matrix (Zr, Nb) and their interaction with carbon to form another strengthening by the formation of carbides.

The effect of the additives Zr and C on the phase composition and high temperature mechanical properties of Fe_3Al-type alloys was investigated. The presence of phases is related to the ratio of zirconium and carbon in the alloy. The presence of tiny metastable phase particles, identified as $(Fe_{1-x}Al_x)_3Zr$ and Fe_2Zr, affects the tensile and creep properties of the alloys [2].

Compressive creep of alloys based on Fe-30 at.% Al with zirconium additions in the range of 0.4-5.2 at.% was studied at temperatures from 650 to 900 °C. The alloys were tested in two different states: (i) cast and (ii) annealed at 1000 °C for 50 h. Stress exponents and activation energies were estimated. The values of the stress exponent can be explained by the dislocation motion controlled by climb and by the presence of second-phase particles. Higher stress exponents were detected only at the temperature of 900 °C and with zirconium content of up to 2%. Creep resistance at temperatures of 700 and 900 °C increases with the increasing amounts of the secondary phases. At the temperature of 650 °C, creep resistance is given not only by the volume fraction of particles but also by the ratio of $Zr(Fe,Al)_{12}/Zr(Fe,Al)_2$. This can be attributed to a more effective strengthening by particles of $Zr(Fe,Al)_{12}$ resulting either from their mechanical properties or from their finer distribution [3].

The effect of Nb and C additions on the phase composition, microstructure and creep resistance of Fe_3Al-type alloys was investigated [4]. Two alloys, which contained (at. %) (i) 27.6 Al, 1.15 Nb and 0.19 C (Fe balance) and (ii) 27.1 Al, 1.11 Nb and 0.76 C (Fe balance), were studied in a temperature range from 600 to 800 °C. The carbide in both alloys was identified as Nb_6C_5. The creep data can be rationalized by introducing a threshold stress, below which the creep rate is negligible. The threshold stress and an effective stress exponent were found simultaneously by a numerical method. Using the obtained values of the threshold stress, the activation energy of creep was determined to be 328 kJ/mol (see Figure 1). The effective stress exponent varied from 2.0 to 3.1. A breakdown of power-law behaviour was observed at higher stresses. The transition occurred at the normalized creep rate, which agrees with the rule suggested by Sherby and Burke and the diffusion coefficient *D* corresponding to the diffusion of Al in Fe-Al.

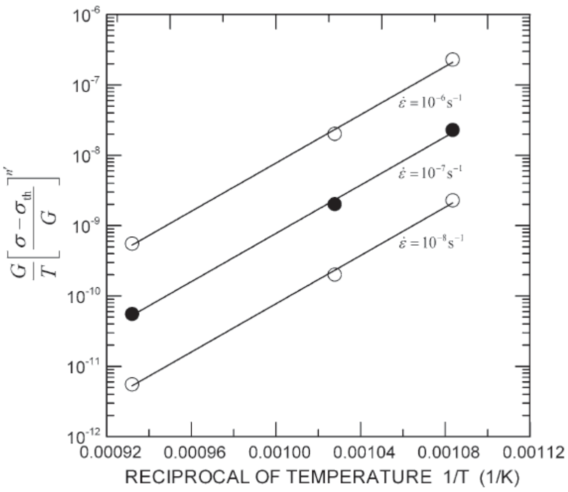


Fig. 1 Semi-logarithmic plot of the temperature-compensated effective stress vs. the reciprocal of temperature. The fitted lines are parallel and result in an activation energy that is equal to 328 kJ/mol

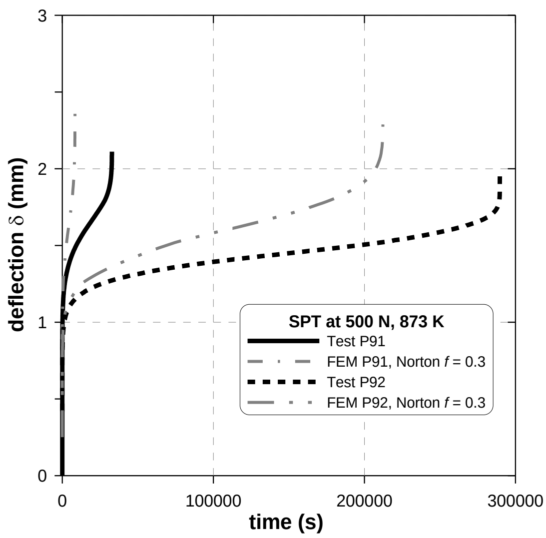


Fig. 2 Comparison of P91 and P92 test and calculated SPT-CF diagrams at 500 N

b) Small punch testing (SPT)

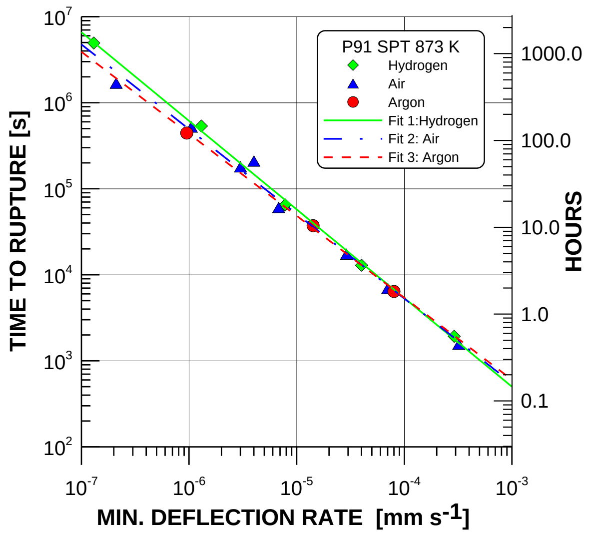
Exposed Plant piping

Non-disruptive material sample scooping is frequently applied to a range of operating components since it is supposed that a removal of small amount of material from the surface has no effect on component's integrity. As-removed material is then subjected to a mechanical testing by small punch test to estimate tensile, creep and fracture properties necessary for assessment of residual life of the component. The question arises whether the assumption that measurements on surface specimens are representative for the characterization of the whole component is justified. For the verification of this assumption, four steam pipes after either plant- or simulatedexposure up to 104 000 hours were used. Small punch specimens were prepared from different positions within pipes. The dimensions of specimens enable to estimate mechanical properties for several dozens of specimens along available power-plant pipes' walls. The work summarizes the results of small punch tests at both constant deflection rate and constant force and at room and elevated temperature, respectively. In the walls of the investigated pipes, the results obtained on surface specimens do not differ substantially from the results on specimens prepared from central parts. The uniformity of properties in the pipe walls indicated a low degree of damage in the inspected localities [5].

P91/P92 steels

Conventional creep and small punch testing performed on miniaturized discs prepared from P91 and P92 steels was studied [6]. Some of the test cases were also numerically modeled by Finite Element Method (FEM) with help of Norton's constitutive model and the results were compared with the experiment as shown in Figure 2. Presented work complements the existing empirical investigations on small punch and conventional creep tests relations. Factor Ψ was determined for P92 steel, however it has not strictly constant value in the examined range of stress and forces. There is need to perform additional tests at lower forces to confirm or disproof such trend. Obtained creep properties from conventional tests were evaluated by regression analysis and used in FEM to numerically model the SPT. The results were compared and discussed. Some discrepancies exist between the computed and measured minimum deflection rates and need to be thoroughly reexamined. It seems necessary to measure the static and dynamic friction coefficient between the puncher ball and the specimen at room and elevated temperatures.

The small punch testing under constant force condition was performed in various atmospheres: air, hydrogen and argon [7]. The material selected for the study was P91 steel and testing temperature 873 K. The effect of atmosphere on the test results such as time to rupture, minimum deflection rate and other parameters was measured and evaluated. The results show that there is not very strong but definite effect of atmosphere on SPT creep rupture time. The longest times to rupture were obtained in hydrogen atmosphere for whole range of tested forces. However the difference between tests in hydrogen and tests in air or in argon diminished with decreasing force and longer time to rupture. The validity of Monkman-Grant relationship was demonstrated in all three variable atmospheres, see Figure 3.



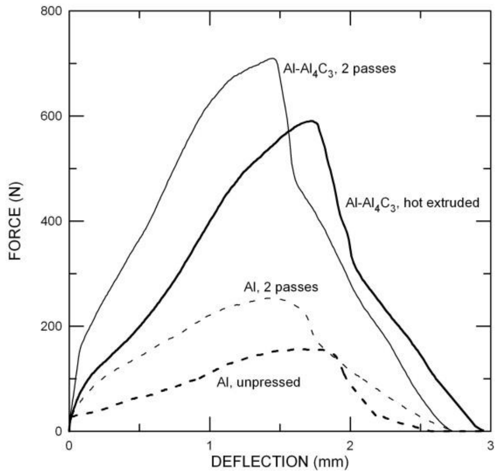


Fig. 3 Monkman-Grant relationship of P91 SPT-CF

Fig. 4 SP load-deflection curves at room temperature

$Al\text{-}Al_4C_3$ composite

Mechanical properties of pure aluminium and $Al\text{-}Al_4C_3$ composite and their changes during equal channel angular pressing up to 12 and 2 passes, respectively, were studied by the small punch tests [8]. In Figure 4 there are shown the examples of SPT curves. 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 were determined: firstly yield force corresponding to the transition from elastic-bending to plastic-bending regimes, secondly maximum load and finally the deflection corresponding to this maximum. The relations of these quantities to corresponding quantities resulting from conventional tensile tests were analyzed. The observed grain size was successfully explained with the recently published model of grain refinement during severe plastic deformation.

Creep fracture of a composite based on an aluminum matrix reinforced by 4 vol.% Al_4C_3 was studied at temperatures of 623 and 723 K by small punch testing with a constant force [9]. The composite was tested in state after equal channel angular pressing (ECAP) by two passes of route C as a final operation. It was found that the time to fracture was inversely proportional to the minimum deflection rate in a similar manner as the corresponding quantities in conventional creep tests, see Figure 5. The fractured surfaces of the studied materials had largely intercrystalline character.

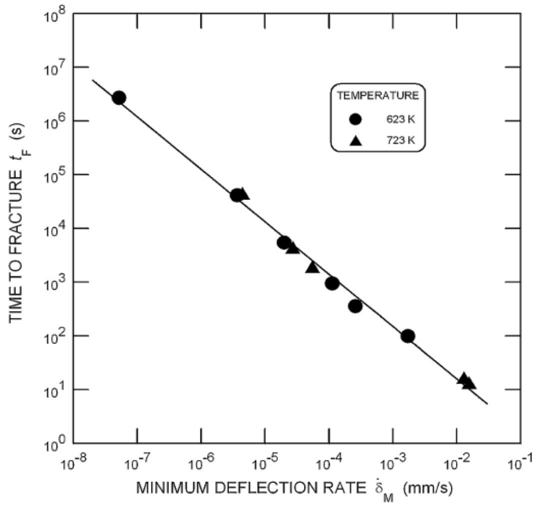


Fig. 5 Dependence of time to fracture on minimum deflection rate of $Al\text{-}Al_4C_3$ composite

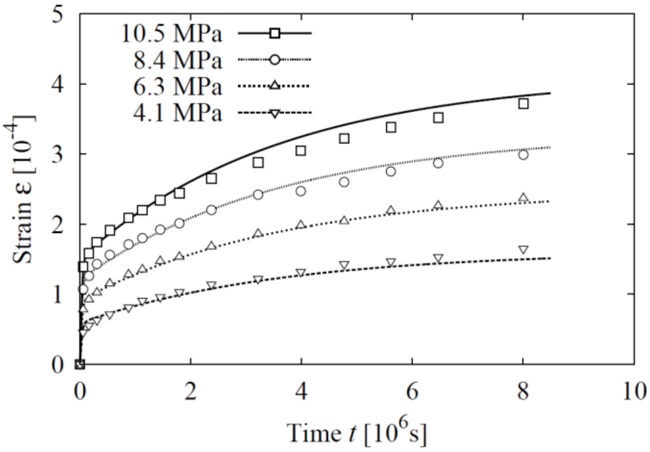


Fig. 6 Creep curves of P91 steel at 625°C and low stresses, fitted by dislocation bowing model equations.

Fe-Al intermetallics

The constant-deflection-rate mode of the small punch test can be used to estimate the ductile-to-brittle transition temperature (DBTT), the fracture toughness or other various ductility-related properties. In the creep group, the selected Fe-Al alloys with different aluminium concentration and different additional elements (Zr, Nb and V) were studied [10, 11, 12]. Three main quantities were determined: 1. maximum load, 2. deflection corresponding to this maximum and 3. fracture energy obtained by integrating the load–deflection curve. The dependences of all these quantities on temperature differ significantly for different alloys. The temperature variation of fracture energy could be interpreted in terms of changes in ductility and resistance to plastic strain. The ductile-to-brittle transition temperature estimated by this method is in reasonable agreement with the values found by conventional approach. Fractographic observations of ruptured specimens were performed with scanning electron microscope. Temperature dependence of small punch test quantities was related to the microscopic image of the fracture. Equivalent fracture strain was evaluated from both small punch test data and microscopic observation of fractured specimens. The relation between equivalent fracture strain and small punch fracture energy was examined. Fracture toughness was calculated from the equivalent fracture strain. Temperature dependence of calculated fracture toughness supports the conclusion that the ductility of investigated alloys is not deteriorated by the presence of carbides.

c) Creep of light materials

This activity tries to reassume research started by the former leader of the group, Dr. Milička. As far as Mg-based materials are considered, main results were obtained within international co-operation with CENIM Madrid. The creep resistance of three Mg-Y-Zn alloys with different microstructures was evaluated at temperatures from 200 °C to 400 °C [13, 14]. For low temperature and/or high strain rates, the creep behaviour shows a high stress exponent (n = 11) and high activation energy. The

alloy can be compared to a metal matrix composite where the magnesium matrix transfers part of its load to the Long-Period Stacking Ordered Structure phase. In this situation, the heat treated alloy shows higher creep resistance because the lamellar structure within the magnesium grains in the heat treated alloy acts as an additional barrier against creep deformation. At high temperature and/or low stresses, creep is controlled by non-basal dislocation slip. The cast alloy showed higher creep resistance with respect to the heat treated alloy. The 18R structure is slowly transformed into a 14H structure as the Zn and Y atoms diffuse through the magnesium grains. The atoms nucleate at Shockley partial dislocations and inhibit its movements. At intermediate and high strain rates at 400 °C and at intermediate strain rates between 350 °C and 400 °C, the extruded alloys show superplastic deformation with elongations to failure higher than 200 %. Cracking of coarse LPSO second-phase particles and their subsequent distribution in the magnesium matrix take place during superplastic deformation, preventing magnesium grain growth. Creep experiments were performed completely in IPM, preparation of materials, microstructure observation and constant strain-rate test in CENIM.
Research of Al-based composites is described in the preceding paragraph.

d) Low strain rate creep in metals
The project focused on transient effects in creep under low stress deformation regime was solved. Two new models were developed and published: the elastoplastic model [15] and dislocation bowing model [16]. The latter model combines climb of dislocations, controlled by self-diffusion, and viscous glide controlled by diffusion of solute atoms. The model provides very good results; the example for the P91 creep resistant steel is in Figure 6.
In collaboration with the group of Advanced High-Temperature Materials, low stress creep was investigated for Zr-1%Nb alloy, which is used for cladding tubes of nuclear fuel. This experimental work was supported by the project TA02011025 of Technology Agency of the Czech Republic and is focused on “Loss-of-Coolant Accident” conditions with high temperatures above 500 °C. Results are to be published in 2015.

Beside institutional funding from ASCR the above listed scientific results were reached with grant support of the Czech Science Foundation and the Grant Agency of the Academy of Sciences. In addition, significant part of the work has been realized in the frame of CEITEC - Central European Institute of Technology with research infrastructure supported by the project CZ.1.05/1.1.00/02.0068 financed from the European Regional Development Fund. This support was aimed mainly for the infrastructure and partly on salary of the CEITEC - IPM team members, Dr. Dymáček and Dr. Luptáková. The project no. CZ.1.07/2.3.00/20.0214 within the Operation Programs Education for Competitiveness funded from ESF through the Ministry of Education Youth and Sports of the Czech Republic, co-ordinated by Assoc. Prof. L. Náhlík from the High Cycle Fatigue Group supported several trips to international conferences and meetings and also contributed to the salaries of students and younger researchers.

International co-operation

Dr. Ferdinand Dobeš cooperates with Dr. Gerardo Garcés from Centro Nacional de Investigaciones Metalúrgicas CENIM (National Center for Metallurgical Research), Madrid, Spain. As an example of successfully solved project we can mention Creep of advanced magnesium alloys and composites prepared by powder metallurgy techniques within the Spanish-Czech bilateral cooperation.

Dr. Petr Dymáček cooperates with several teams that perform SPT, particularly with Dr. Martin Abendroth from TU-Bergakademie Freiberg, who is an expert in numerical modelling of materials and neural network approximations of SPT.

Since 2014, there is starting cooperation of IPM on the EN standardization of the SPT technique coordinated by JRC Petten (Dr. Peter Haehner) and round robin testing of steels by both types of SPT is under preparation. The cooperating institutions are: JRC Petten, MMV Ostrava, Swansea Univ., TU Freiberg, HZDR Dresden, MPA Stuttgart, Univ. of Cantabria, Univ. of Oviedo, CIEMAT, INAIL, VUJE, VTT.

An active member of this standardization cooperation is Dr. Tomas E. Garcia from University of Oviedo, who has at disposal modified small-punch testing machine for testing in corrosion environments. He absolved 1 month stay with invited lecture at IPM in February 2014.

The long term co-operation with prof. Michal Besterci from the Institute of Materials Research SAS in Košice continued on research of $Al\text{-}Al_4C_3$ composites prepared by ECAP technique. Small punch tests were performed both in constant force [9] and constant deflection rate [8].

Since 2013 the Group actively participates on the meetings and seminars of KMM-VIN institute which is a platform for research co-operation between academia and industry (see. http://www.kmm-vin.info/ where IPM has an active membership).

Short visits and lectures:
In 2013 we had short visit and lecture at IPM of prof. Roger Hurst (formerly JRC Petten, now honorary professor at Swansea University).

In 2012 we had short visit and lecture at IPM of prof. Kee Bong Yoon, professor at Chung-Ang University, Seoul Korea and president of the Korean University Council of Research & Industry cooperation.

In 2011 we had short visit and lecture at IPM of Dr. Martin Abendroth about modeling of SPT by FEM and neural networks.

In 2010 Dr. Dymáček was invited to give a lecture about SPT research at TU-Bergakademie Freiberg.

Dr. Dobeš and Dr. Dymáček had invited lectures at Chung-Ang University in Seoul and Doosan Company in Pusan in September 2012.

In 2013 Dr. Dymáček went through 2 week stay at University of Oviedo in Gijón in the team of Prof. Cristina Rodriguez and Prof. Javier Belzunce.

Ms. Elvira Oñorbe Esparraguera of the Centro Nacional de Investigaciones Metalúrgicas (CSIC) spent successfully Ph.D. internship in IPM AS CR Brno (April-June 2010) doing creep experiments for her thesis. The obtained results were published in two papers in impacted journals [13, 14].

3 month stay of Ph.D. student David Andrés from University of Cantabria who is studying creep and fracture properties of light alloys by means of SPT is planned in the last quarter of 2015.

New facilities in the laboratories

New high temperature creep laboratory was built within the CEITEC project with 2 new creep-electromechanical machines Messphysik KAPPA LA and Maytec furnaces, one up to 1200 °C, second up to 1400 °C. The cost of investment was about 500 kEUR. The machines allow tension and compression testing at constant load or stress, constant rate experiments and relaxation testing with direct measurement on the specimen or reversal grips. In addition, two new creep machines of IPM's own design for constant load experiments (up to 1000 °C) were added to the creep laboratory.

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	High Cycle Fatigue Group

Response to the last evaluation

The research activity of the group in the period 2005-2009 was highly rated by the evaluators. The results of both the basic and applied research were classified as outstanding on the international level. The international scientific reputation of the members of the Group was appreciated. However, the evaluators pointed out that there is a large age gap between key investigators (P. Lukáš, Z. Knésl and L. Kunz) and young members of the team. Extension of the Group by further young researchers and Ph.D. students was highly recommended.

The recommendation to engage young scientists was taken seriously into account. During the evaluated period 2010-2014 the number of Ph.D. students strongly increased. Continuously 5-7 of them cooperate on the research projects solved by the group members. It means that every researcher is a tutor of one or two Ph.D. students. Unfortunately, during the evaluated period two of the experienced members of the Group, namely P. Lukáš and Z. Knésl, passed away unexpectedly (end of 2012 and beginning of 2013). Since that time the group consists mainly of relatively young researchers with age under 40. Thanks to the historically close co-operation of all of the Group members on the project proposals and project solution, the scientific output of the Group remained unchanged. In spite of the loss of two key researchers of the Group the employment of three postdocs (M. Ševčík, M. Šmíd, S. Fintová) and 7 Ph.D students ensured the continuity of high quality research work in High Cycle Fatigue Group.

During the reporting period the team was successful to acquire 20 research projects from various grant agencies with total foundation of about 1 500 kEUR. Additionally, since 2011 assoc. prof. L. Náhlík has coordinated the project CEITEC-IPM (Central European Institute of Technology – part of IPM). Further, he successfully applied for four projects from the Operational Programme Education for Competitiveness funded by the Ministry of Education Youth and Sports of the CR (MEYS). The total budget of these five projects in the reported period is approximately 5 000 kEUR. This coordination activity markedly exceeds the research group activity. The projects represent important financial sources for investments, personal costs and enables higher mobility especially of the young scientists across all the research groups within the whole IPM.

Main achieved results

<u>Fatigue behaviour of ultrafine-grained materials</u>

Fatigue behaviour of ultrafine-grained materials was one of the intensively studied topics. The investigation performed in the evaluated period helped to answer some recently discussed questions in the research society, namely (i) what are the mechanisms of the cyclic plastic strain localization in UFG structures, (ii) which microstructural, material and severe plastic deformation process parameters (particularly parameters of equal channel angular pressing) influence the strain localization and hence the fatigue lifetime, (iii) which factors influence the stability of UFG structure with high stored deformation energy at cyclic loading, (iv) what is the difference in the mechanisms of cyclic slip localization in ultrafine-grained structures and conventionally grained ones. Advanced experimental methods, like focussed ion beam (FIB) technique were applied for the study of the localization of cyclic plasticity and initiation of fatigue cracks. An example of the observation of fatigue damage by means of a FIB cut through cyclic slip bands developed during giga-cycle fatigue loading of UFG Cu is shown in Fig. 1. The finding on the stability of ultrafine grained cooper under fatigue loading was presented as one of the three most important achievements of the IPM in the year 2011. The research was supported mainly by the projects of the Czech Science Foundation. The most relevant publications are the following: Lukáš, P. et al.: Fatigue damage of ultrafine-grain copper in very-high cycle fatigue region, Materials Science and Engineering A 2011, 528, 7036-7040 and Kunz, L. et al.: Strain localization and fatigue crack initiation in ultrafine-grained copper in high- and giga-cycle region, International Journal of Fatigue 2014, 58, 202-208 (both outputs propound for the evaluation). Further, the overview and comparison of the behaviour of conventionally and severe plastically deformed copper was published in a chapter by L. Kunz: Mechanical properties of copper processed by severe plastic deformation in a book Copper Alloys - Early Applications and Current Performance - Enhancing Processes edited by L. Collini, InTech. Publisher 2012. The chapter was downloaded 3000 times until March 2014.

<u>High cycle fatigue of superalloys</u>

Investigation of fatigue damage mechanisms of superalloys at high temperatures is a traditional research topic of the Group. The topic is strongly relevant to the engineering practice. Whereas in the last evaluation period the research was focused on the investigation of the combined cycle loading on the high temperature behaviour of CMSX-4 single crystals and their microstructural changes (this investigation was supported by participation in the 6th FP Premeccy with overlapping to the year 2010), the majority of investigation in the evaluated period was targeted on the polycrystalline IN713LC and MAR-M247 superalloys. The research was supported by grants of the Ministry of Industry and Trade of the CR and was performed in close co-operation with industry (První brněnská strojírna, Velká Bíteš). The most important results concern the influence of defects on the high cycle fatigue life at high temperatures. New piece of knowledge on the localization of cyclic plasticity in persistent slip bands and on crack propagation at low growth rates was published e.g. by Kunz, L. et al.: Casting defects and high temperature fatigue life of IN713LC superalloy, International Journal of Fatigue 2012, 41, 47-51, (paper propound for the evaluation).

<u>Propagation of small cracks</u>

A new model for small crack propagation rate description was deduced in cooperation with Low Cycle Fatigue group. This model based on elasto-plastic fracture mechanics was applied on the low activation steels strengthened by yttrium oxide, see P. Hutař et al., Journal of Nuclear Materials 452 (2014) 370–377 (paper propound for the evaluation). It was found that simple relationship of Paris-Erdogan type can describe small crack propagation rate for high plasticity conditions da/dN=Cjp (Japl)mjp, where Japl is the amplitude of the plastic part of J-integral and Cjp and mjp are material constants. The importance of plasticity for small fatigue crack growth rate corresponds well with physical interpretation of fatigue crack propagation in the large scale yielding conditions. Example of proposed small crack propagation law applied on experimental data measured for three different steels (two of them are strengthened by yttrium oxide) is shown in Fig.2. Experimental data measured on different strain amplitudes can be described by one “material” curve, which to the contrary with other models describing small crack propagation can be used for the lifetime prediction of any structure containing small cracks loaded under large scale yielding conditions (e.g. small cracks initiated in the plastic zone of notch or at a hole). This research activity was supported mainly by the projects of Czech Science Foundation.

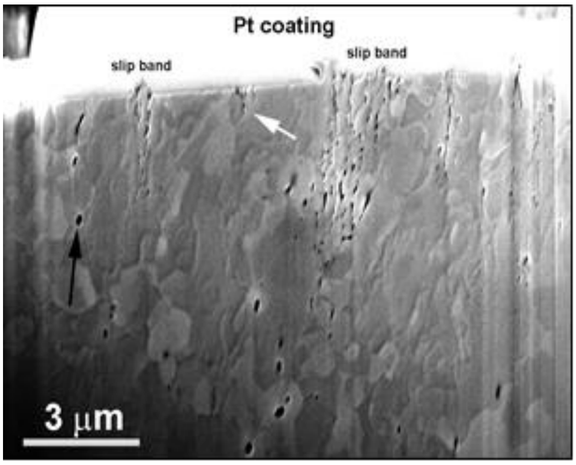


Fig. 1. FIB micrograph of a cut through cyclic slip bands and grain structure after giga-cycle fatigue of UFG Cu.

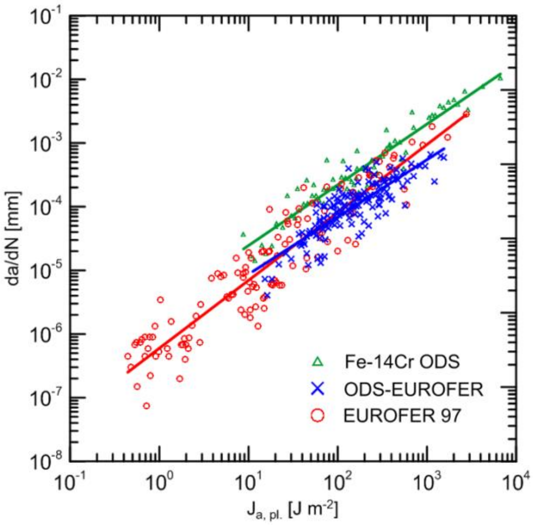


Fig. 2. Comparison of the small crack growth rate vs. plastic part of J-integral for low activation steels.

Numerical simulations of fatigue crack propagation

Simulation of fatigue crack propagation is an important long-term research topic of the group. There exists a lot of unsolved problems in this area, especially in the case of 3D crack propagation. One of these problems is the effect of the free surface on the fatigue crack propagation. This aspect was discussed in the papers P. Hutař et al., International Journal of Fatigue 32 (2010) 1265–1269 and M. Ševčík et al., International Journal of Fatigue 39 (2012) 75–80 (outputs propound for the evaluation). New methodology based on a generalised fracture mechanics concept is proposed to describe crack behaviour close to the free surface. The usual modelling of 3D cracks is related to a straight crack front intersecting perpendicularly free surface. Generally, two different singular fields exist along the crack front of this 3D crack (see Fig.3).

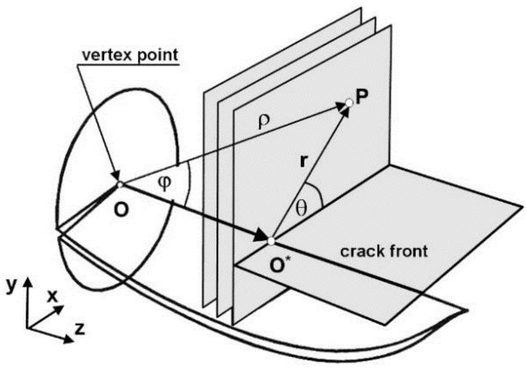


Fig. 3. Singular stress field close to the free surface

The classical square-root singular field is present in the middle of the specimen where plain strain conditions prevail. So-called vertex singularity is important in the area close to the free surface and the stress distribution can be expressed by the following relation:

$$\sigma_{ij} \approx \rho^{-p} f_{ij}\left(p,\theta,\varphi\right) \quad .$$

In this case, the stress singular field is spherical with the centre in the vertex point (O) and shape functions depend on local spherical coordinates φ,θ and on the vertex singularity exponent *p*. Following ideas are based on the assumption that the strain energy density is a parameter controlling the fatigue crack propagation. Generalised fracture mechanics description of the fatigue crack based on generalised stress intensity factor can be proposed. The formation of the fatigue crack shape due to the influence of a free surface was explained and numerically predicted. A practically similar crack front angle (the angle between crack front and free surface) for a particular Poisson's ratio was observed, which is in agreement with the available experimental studies in literature. It was found that, at least for mode I, the crack front is shaped to hold square root singularity along the whole crack front. Based on this idea, fast algorithm for prediction of the fatigue crack front shape in the 3D bodies was developed and published: M. Ševčík et al., International Journal of Fatigue 39 (2012) 75–80. The paper is conceptual in the sense of generalization of the linear elastic fracture mechanics to the 3D problems and can help to accurate numerical modelling of fatigue crack behaviour. The procedure of the reliable assessment of the prediction of the fatigue crack behaviour in layered materials, proposed by P. Hutař and co-workers was appreciated as one of the three best results achieved at the IPM in the year 2010. These research activities were supported mainly by the Czech Science Foundation.

Theoretical investigation of the generalised linear elastic fracture mechanics

Traditional topic of the Group established by Z. Knésl is generalised linear elastic fracture mechanics. In this area several important papers were published by the group members: L. Náhlík et al., Engineering Fracture Mechanics 77 (2010) 2192–2199, J. Klusák et al., Theoretical and Applied Fracture Mechanics 53 (2010) 89–93, J. Klusák et al., Engineering Fracture Mechanics 110 (2013) 438–447, M. Ševčík et al., Computational Materials Science 64 (2012) 225–228 (papers propound for the evaluation). These papers are focused on crack propagation through an interface of two-dissimilar materials or crack initiation from bi-material notch. The result on the reliability of assessment of a bi-material notch was selected as one of the five most important achievements of the IPM in the year 2013. Published papers have a valuable impact on fracture mechanics theory, and they extend its applicability to the cases with general singular fields. Complexity of this theory shows applicability of the results obtained in the wide range of materials from ceramic laminates (L. Náhlík et al., Engineering Fracture Mechanics 77 (2010) 2192–2199) to polymer layered structures (M. Ševčík et al., Computational Materials Science 64 (2012) 225–228). These research activities were supported mainly by the projects of the Czech Science Foundation and the Grant Agency of Academy of Sciences.

Lifetime prediction of polymer structures

Relatively new topic of the Group is lifetime prediction of the polymer structures. It could be argued that this area is relatively far from fatigue of metals originally studied in the Group, but new applications of the polymers in many cases replace metal components, and in some special loading situations modelling of crack propagation in polymer materials has similar principles as modelling of fatigue crack propagation in metals. Complex methodology for lifetime prediction of the polyolefin pipes was developed in co-operation with University of Leoben, see papers P. Hutař et al., Engineering Fracture Mechanics 78 (2011) 3049–3058 and P. Hutař et al., Engineering Fracture Mechanics 108 (2013) 98–108 (papers propound for the evaluation). The presented methodology combines numerical estimation of the stress intensity factor with fatigue testing of the polymer materials in order to be able to estimate the lifetime of a pressured component without time-consuming hydrostatic pressure tests (lifetime of the modern pipe materials is predicted from 50 to 100 years). Slow crack growth (SCG) in pressurized PE pipes usually starts at stress concentrations caused by material imperfections like impurities, cavities or flaws at or near the inside pipe surface. Originating from such initial defects, the quasi-brittle failure mechanism of SCG is characterized by a continuous formation and breakdown of crazes inside localized process zone. To describe slow crack propagation the accelerated data from creep tests at high temperature (80 °C) are usually used. The novel concept of the extrapolation procedure in which the kinetics of fatigue crack growth is measured using crack round bar CRB specimens at different loading ratios R (ratio of minimum load to maximum load at fatigue cycle) was originally developed at University of Leoben and now implemented in our Group. Crack propagation rate for different R-ratios is experimentally evaluated and then extrapolated to R=1, corresponding to static loading. This procedure is relatively simple, time-efficient (in the comparison with classical accelerated creep tests) and takes into account the combination of creep and fatigue loading. Using these experimental data, numerical simulations of 3D crack propagation in polymer material can be defined and lifetime of the polymeric structure can be evaluated. Special algorithm for numerical estimation of the crack shape development was developed to be able to simulate crack propagation accurately. This algorithm based on energy balance at the crack front control crack propagation utilizes the iso-lines of stress intensity factor. This algorithm allows modeling of complicated loading situations as combination of external loading, residual stresses and material non-homogenity e.g. close to the polymer weld. Whole methodology shows the strength of the Group, combination of extensive fatigue testing with complex 3D numerical simulations of crack type damage. This research activities was supported mainly by the Czech Science Foundation and by co-operation with University of Leoben.

Projects and International cooperation

Grant and project activity of the researchers of the High cycle fatigue group in the framework of the whole IPM manifests itself by the fact that the largest projects of the Institute are under leadership of members of this Group.

The largest project of the group (also the most important in the Institute) is the project CEITEC - IPM (Central European Institute of Technology) with the budget of 4 447 kEUR, co-investigator L. Náhlík. The whole project (No. CZ.1.05/1.1.00/02.0068, 2011-2015) with total budget of 223 000 kEUR is realized within the support of the Operational Programme Research and Development for Innovations (OP RDI) of the MEYS. CEITEC is a scientific center in the fields of life sciences, advanced materials and technologies whose aim is to establish itself as a recognized center for basic as well as applied research. It is a consortium of the most prominent universities and research institutes in Brno, namely Masaryk University, Brno University of Technology, Mendel University in Brno, University of Veterinary and Pharmaceutical Sciences in Brno, Veterinary Research Institute and Institute of Physics of Materials ASCR.

During years 2012-2013 all tenders of CEITEC IPM for devices listed in the Technical Annex were finalized. Based on this, the laboratories of the IPM were equipped with advanced experimental facilities, which are managed and used by other research groups. The laboratories were equipped with high resolution transmission electron microscope JEOL JEM-2100F, Creep test system Zwick/Roell - Messphysik KAPPA LA spring 20 kN and 50 kN, electrohydraulic computer controlled axial/torsional test system MTS 809, system for measurement of electrical and magnetic properties in the temperature range 2 – 300 K and new electro-spark machine for specimen preparation.

Substantial for the research output of the whole Institute are also other projects led by L. Náhlík (CZ.1.07/2.4.00/17.0006 - Building up cooperation in R&D with the research and industrial partners 2011-2014, CZ.1.07/2.3.00/30.0063 - Talented postdocs for scientific excellence in physics of materials 2012-2015, CZ.1.07/2.3.00/20.0197 - Multidisciplinary team in materials design and its involvement into international cooperation 2012-2015, CZ.1.07/2.3.00/20.0214 - Researchers4Materials 2012-2015) with total budget of about 1 500 kEUR. The aim of these projects is to strengthen research groups of the Institute by new young postdocs and Ph.D. students and help them to improve international co-operation in their research fields.

Based on these projects, the High Cycle Fatigue Group was extended by 2 post-doctoral researches Dr. Ševčík and Dr. Šmíd and several Ph.D. students. Moreover, the projects enabled the international co-operation with (i) EPFL – Lausanne (Swiss) in the field of fatigue damage of the composite structures. Dr. Ševčík spent 3 months in EPFL laboratory, Switzerland. (ii) Co-operation in the field of fatigue of metals with regard to materials used for railway industry with University of Milano (Italy). Two Ph.D. students spent together more than 5 months there. The group simultaneously performs investigation for Bonatrans group, which is a leading producer of railway wheelsets. Long term co-operation with well-known fatigue laboratory in Poitiers (France) was also financially supported from these projects. New co-operation in the field of 3D singular stress fields with University of Adelaide (Australia) was established. Members of the Group have also the possibility to attend world-class international conferences and to improve international visibility of the team.

Project focused on mechanisms of fatigue damage of ultra-fine grain materials was led by L. Kunz (P108/10/2001 - Cyclic plastic deformation and fatigue properties of ultrafine-grained materials 2010-2014 with the budget of 176 kEUR). Project aims were focused on cyclic slip localization, stability of UFG structures and mechanisms of fatigue crack initiation. Materials under investigation were ultra-fine grain copper as the model material, Mg based alloys and Ti alloys as promising materials for applications.

Research in the area of fatigue damage of superalloys was supported by three projects of the Ministry of Industry and Trade of the CR (FT-TA4/023 - Research and development of mechanical properties of the materials used for new types of turbochargers 2007-2010, FR-TI3/055 - Research and development of precise casting technology for new types of nickel-based superalloy castings of rotor parts of turbochargers and aircraft turbines 2011-2014, FR-TI4/030 - Research and development of mechanical properties for new types of nickel-based superalloy castings developed by technologies investment precision casting method 2012-2015) with the budget of 368 kEUR. Research supported by these projects was focused on fatigue and fatigue/creep damage of the nickel based superalloys (especially IN 713LC, IN 783 and MAR-M 247) with regard to our industrial partner PBS Velká Bíteš, a.s. In this field the intensive cooperation between Low Cycle Fatigue Group and Advanced High-Temperature Materials Group took place.

Damage of the polymeric material was aim of following projects of CSF (106/08/1409 - Role of Structure of Crosslinked Polymer Matrix in Particulate Composities. Multiscale Modelling and Experimental Verification 2008-2010, 101/09/J027 - Correlation between structural changes, damage evolution and crack propagation behaviour of welded thermoplastic components 2009-2011, 106/09/0279 - Fracture damage mechanism of multilayer polymer body 2009-2011, P108/12/1560 - Description of the slow crack growth in polymer materials under complex loading conditions 2012-2014) with the budget of aprox. 630 kEUR. In this area good co-operation with the University of Halle-Wittemberg (prof. Grellman), Polymer Institute Brno (Dr. Kučera, doc. Nezbedová), Institute of Macromolecular Chemistry of Academy of Sciences of the Czech Republic (prof. Dušek, Dr. Kotek) and University of Leoben (prof. Pinter, Dr. Frank) was established. Projects were focused on numerical modelling of fatigue/creep damage of the polymer materials. Unique methodology for lifetime prediction of polymer materials based on combination of experimental testing and numerical simulations are important outputs of the projects.

Theoretical investigations in the field of linear elastic fracture mechanics theory were supported by the following projects of CSF and ASCR (101/08/0994 - Determination of conditions of failure initiation in bi-material wedges composed of two orthotropic materials 2008-2010, KJB200410803 - Generalization of linear elastic fracture mechanics to crack propagation problems in non-homogenous materials 2008-2010, M100410901 - Fracture mechanics description of three dimensional structures: numerical analysis and physical consequences of constraint 2009-2012, P108/10/2049 - Crack initiation and propagation from interface-related singular stress concentrators 2010-2012) with the budget of 285 kEUR. Based on the above mentioned projects, fruitful co-operation was established with the University of Leoben (Dr. Bermejo), University of Oviedo (prof. Canteli), and Brno University of Technology (prof. Kotoul). The projects were focused on new generalized linear elastic fracture mechanics descriptions of special cases as crack initiation from bi-material notch or crack propagation through the interface of dissimilar materials. This methodology is necessary for fracture mechanics description of composites (especially laminate structures).

Completely new topic established in the Group during the reporting period is the fracture behavior and fatigue of silica-based composite. Following grant projects of CSF and GAAS were founded (103/08/0963 - Basic fatigue characteristic and fracture of advanced building materials 2008-2010, KJB200410901 - Fracture of silicate based composites studied on core drilled specimens – numerical-modelling background for advanced fracture parameters determination 2009-2011, P104/11/0833 - Response of cement based composites to fatigue loading: advanced numerical modelling and testing 2011-2013, P105/11/1551 - Energetic and stress state aspects of quasi-brittle fracture – consequences for determination of fracture-mechanical parameters of silicate composites 2011-2013) with the budget of 216 kEUR. In the framework of these projects was established co-operation with University of Oviedo (prof. Canteli), Technical University of Vienna (Dr. Merta), University of Ghent (Prof. Wouter de Corte, Prof. V Boel) and Brno University of Technology (prof. Keršner). The projects were focused on modelling of fracture behavior of silica based composites and its fatigue behaviour. Especially fatigue testing of the silica-based composites is a new scientific area driven by the need of these properties for high-strength silica based building materials for high skyscrapers buildings and bridges.

New acquisitions in laboratory

The laboratory of High Cycle Fatigue Group was equipped by a new compact servohydraulic testing machine with maximum loading force 25 kN in the year 2010 (investment approx. 130 kEUR), see Fig 4. This machine, contrary to five resonant testing machines available in the laboratory, allows testing of non-metallic materials, which became important topic of the group. For this type of test the resonant systems cannot be used, because they do not allow preset the loading frequency.

The laboratory was continuously upgraded by the workstations based on PC platform for heavy numerical simulations. At present 6 new workstations are available (investment approx. 18 kEUR).

Fig. 4. Servohydraulic testing machine acquired for high cycle fatigue laboratory.

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Low Cycle Fatigue Group

Response to the last evaluation

The diversity of the research themes in Low Cycle Fatgiue Group continues and concentrates both ton the basic studies and the industrially important subjects. The provision of new equipment, namely the thermomechanical fatigue testing system recommended during last evaluation, allows studying new fields. The provision of biaxial testing system allowing the testing also at high temperatures allows extending our interest in new fields. We did not succeed in provision of our own high quality AFM microscope but the long-term collaboration with our partner (Czech Metrology Institute, Brno, Czech Republic) allows an access to the top quality instrument and the help with the evaluation procedures.
The age distribution of the Group is still well balanced and Dr. Man has replaced Prof. Polák in the leadership of the Group.
The Group continues in international co-operation and is active in proposing grant projects (3 grants in the Czech Science Foundation (CSF) and participation in projects of the Technology Agency of the Czech Republic (TACR).

Achieved results

***Model of surface relief formation and fatigue crack initiation*.** One of the new results achieved during last 5 years is a formulation and publication of a quantitative model describing the mechanisms of surface relief formation and fatigue crack initiation in crystalline materials at ambient, depressed and slightly elevated temperatures simultaneously with the numerous experimental observations of the internal structure and surface relief evolution using high resolution techniques. The mechanism of fatigue crack initiation is a problem discussed for many years in international meetings with different views of the scientists. Our solution is based on the knowledge of the behaviour of the lattice defects in fatigue metals and experimental observation of cyclic slip localization.
Fig. 1 shows the AFM image of the plastic replica, showing the early stage of the fatigued grain of 316L austenitic steel. Both extrusions and intrusions could be seen developing along the bands of localized slip – persistent slip bands.

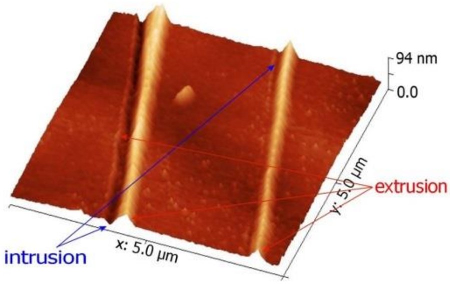


Fig. 1

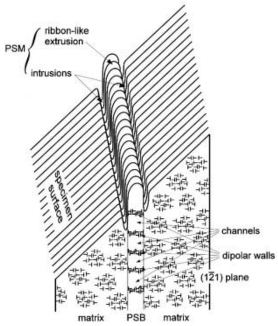


Fig. 2

Fig. 2 shows the predicted shape of the extrusion-intrusion pair (half of symmetrical profile). The quantitative model allows predicting shapes and evolution of the shape of persistent slip markings with the number of cycles. The intrusions represent crack-like defect, from which stage crack develop.

Selected publications: J. Polák and J. Man: *Int. J Fatigue* 65 (2014) 18–27; J. Polák and J. Man: *Mater. Sci. Eng. A* 596 (2014) 15–24; J. Polák, I. Kuběna and J. Man: *Mater. Sci. Eng. A* 564 (2013) 8–12; J. Man et al.: *Adv. Mater. Res.* 891-892 (2014) 524–529).

Advanced TiAl-Nb alloys – optimization of microstructure and properties. In the project, the development and structure optimization of new advanced cast multiphase gamma based TiAl-8Nb-X alloys with the graded carbon content (0.2 to 1 at. %), produced so far only by powder metallurgy was followed. The main pertinent applications of the alloys suggested are turbocharger rotor or gas turbine blade in automotive and power industry.

The thermo-mechanical stability of the alloys has been studied in virgin state, after optimized heat treatment resulting in the lamellar structure and after high temperature creep-fatigue tests. Neutron diffraction (*in-situ* and *post-mortem*), TEM, FESEM and other modern experimental techniques were used for the structure analysis and the study of damage mechanisms especially high temperature cracking.

Fig. 3 shows lamellar structure of TiAl-8Nb-X alloy with locked grain boundaries after optimized heat treatment resulting in considerable improvement of plasticity. Fig. 4 shows cracks starting from intrusions as revealed by high-resolution SEM in TiAl-7Nb cycled at temperature 750 °C with strain amplitude $\varepsilon_a = 3.9\times10^{-3}$.

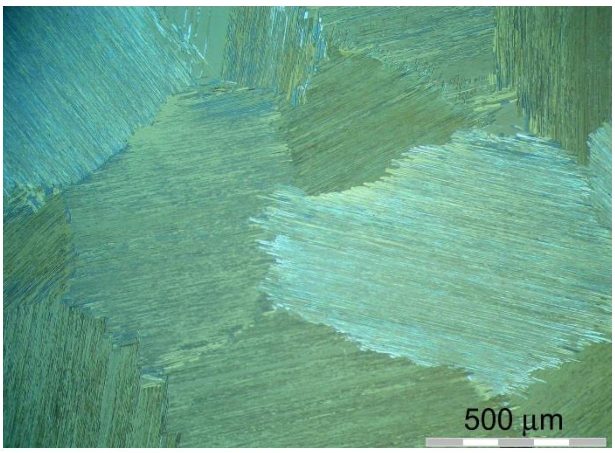


Fig. 3

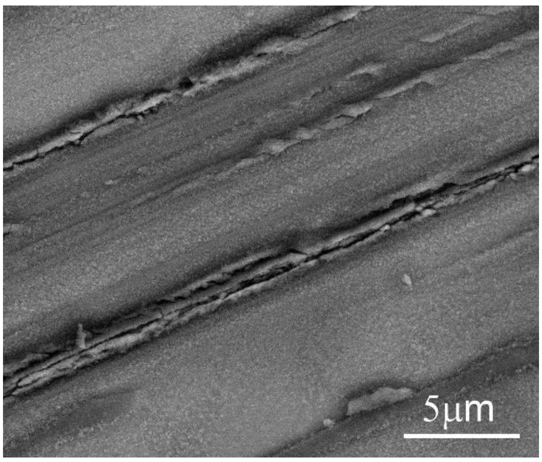


Fig. 4

Selected publications: T. Kruml et al.: *Proc. Eng.* 2 (2010) 2297–2305; M. Petrenec, J. Polák and P. Buček: *Proc. Eng.* 10 (2011) 1390–1395; P. Beran et al.: *Intermetallics* 54 (2014) 28–38; T. Kruml and K. Obrtlík: *Int. J Fatigue* 65 (2014) 28–32.

Protective coatings for high temperature applications of Ni based superalloys. The protective coatings produced by the application of the diffusion layer on the surface of the cast nickel base superalloys were tested in high temperature (800 °C) cyclic loading. The mechanical properties were accompanied by the structure of the diffusion layer, its chemical and phase composition and profile of the hardness. Microscopic observations revealed fatigue damage mechanisms in continuous cycling and using dwells with and without protective coating. The research contributed to the optimized selection of Al, Al-Si a Al-Cr coatings using CVD deposition.

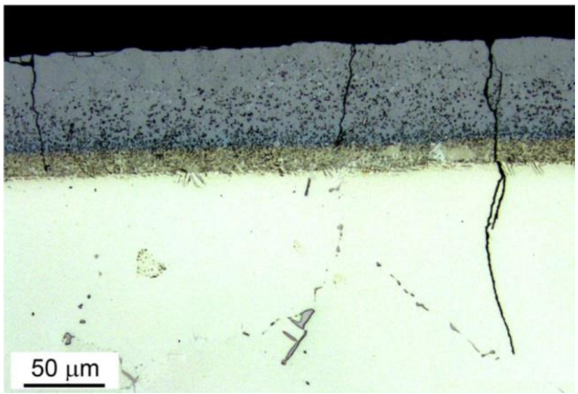


Fig. 5

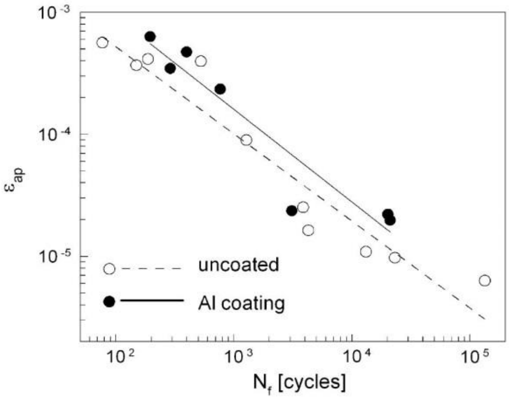


Fig. 6

Fig. 5 shows fatigue cracks growing from the surface to the substrate of an Al coated Inconel 713LC. Fig. 6 demonstrates a beneficial effect of the diffusion coating on the fatigue life of Inconel 713LC cyclically strained at 800 °C.

Selected publications: K. Obrtlík et al.: *Int. J Fatigue* 41 (2012) 101-106; M. Juliš et al.: *Key Eng. Mater.* 488–489 (2012) 307-310; K. Slámečka et al.: *Eng. Fract. Mech.* 110 (2013) 459–467; S. Hutařová et al.: *Key Eng. Mater.* 592–593 (2014) 461–464.

Description of projects solved in the team, collaborations with other teams
During the evaluated period the Low Cycle Fatigue Group solved and/or participated in the following projects:
i) 8 projects supported by the Czech Science Foundation (CSF) – see the enclosure "3-1 List of grant and programme projects" and
ii) 2 projects supported by EU and the Ministry of Education, Youth and Sports of the CR (CEITEC-IPM, Talented post-doctoral researchres for scientific excellence in physics of materials). The main contribution of the CEITEC-IPM project lies in the possibility to acquire expensive experimental facilities and also in salary support for researchers. The project Talented post-doctoral researchers funded by the MEYS supported the young researchers; in our case young post-doctoral researcher V. Škorík was thus able to participate at the research programme of the Group for three years.

The projects were dedicated to both the basic studies and industrially important subjects. Thematically the research covers the following diverse topics:

1) Advanced gamma TiAl intermetallics for high temperature applications. Development of new cast multiphase TiAl-8Nb-C type intermetallics with graded carbon content (in co-operation with A. Dlouhý, Advanced High-temperature Materials Group, IPM Brno), obtaining the lamellar structure via complex heat treatment, cyclic stress-strain response and damage mechanisms at room and high temperatures, creep-fatigue interaction at high temperatures, *in-situ* neutron diffraction study of thermal phase stability of structure (in co-operation with Nuclear Physics Institute ASCR)

2) Oxide dispersion strengthened (ODS) steels for fusion application. The role of oxide dispersion on LCF behaviour of ferritic and ferritic/martensitic ODS steels at wide temperature range 25–800 °C, detail mapping of fatigue damage mechanisms (fatigue crack initiation and short crack growth) by various microscopic techniques, FEM modelling of cyclic stress-strain response and proposition of new methodology for residual lifetime prediction (in co-operation with HCF group of IPM Brno and EFDA).

3) Advanced high-temperature resistant materials (Ni based superalloys, austenitic heat-resistant steel Sanicro25). Cyclic stress-strain response at room but especially at elevated temperatures, analysis of the cyclic stress and its effective and internal components, high temperature resistance and identification of an important role of grain boundary oxidation in crack development (in co-operation with G. Chai, Sandvik Materials Technology, Sweden).

4) Fundamental damage mechanisms in fatigued CG and UFG austenitic stainless steels. Experimental study of cyclic strain localization and surface relief evolution using high-resolution experimental techniques (AFM, FIB. TEM, EBSD, SEM–FEG), at room, elevated and depressed temperatures, cyclic slip irreversibility and slip activity (*in situ* experiments in SEM-FEG in co-operation with A. Weidner, TU Bergakademie Freiberg), stability of austenite structure and the formation of deformation induced martensite during cyclic straining at various temperatures (in co-operation with M. Smaga, TU Kaiserslautern), comparison of cyclic stress-strain response and fatigue life of coarse- and ultrafine-grained produced by special thermomechanical treatment (in co-operation with Prof. Karjalainen, Oulu University).

5) Protective coatings on cast nickel-based superalloys for high temperature applications. Development of optimum technology and characterization of protective diffusion coatings and conventional thermal barrier coatings (TBCs) based on Al, Al-Si and Al-Cr (in long-term co-operation with FME and CEITEC BUT Brno), influence of protective layers on fatigue life of nickel-based superalloy Inconel 713LC in high temperature environment with cyclic stress and identification of mechanisms of coating degradation, transfer of results of excellent research into application field.

International co-operation

The Low Cycle Fatigue Group has a long-time co-operation with several foreign partners (TU Bergakademie Freiberg, CEA Saclay, EPFL Laussane, EFDA and others), some other international co-operations have started during the period evaluated (TU Kaiserslautern, Oulu University, KIT Karlsruhe and others). Mutual creative co-operation resulted in numerous common publications (see below) which were a result of several study stays of members of Low Cycle Fatigue Group at foreign partners e.g. J. Man, V. Škorík and M. Petrenec at TU Bergakademie Freiberg. Foreign partners Dr. A. Weidner, TU Bergakademie Freiberg; Prof. M.

Sauzay and Dr. B. Fournier, CEA Saclay; Prof. G. Saada, ONERA France have stayed at IPM Brno during the period evaluated as well.
The list of foreign partners including the topic of mutual creative co-operation with the Low Cycle Fatigue Group and selected common publications is the following:

- Ecole Polytechnique Fédérale de Lausanne, Switzerland, Prof. J.L. Martin: development, application and interpretation of transient mechanical tests (stress relaxation and strain dip tests)
  T. Kruml and J.L. Martin: *Phil. Mag.* 93 (2013) 50–59; O. Couteau, T. Kruml and J.L. Martin: *Acta Mater.* 59 (2011) 4207–4215.
- ONERA, Châtillon, France, prof. G. Saada: basic mechanisms of plasticity (nanograined materials, plastic instabilities)
  G. Saada and T. Kruml: *Acta Mater.* 59 (2011) 2565–2574; G. Saada and T. Kruml: *Phil Mag*. 93 (2013) 256–271.
- European Fusion Development Agreement (EFDA) and CEA Saclay, France, Prof. M. Sauzay, Dr. B. Fournier: high temperature materials for fusion (ODS steels), modelling of cyclic plasticity and slip localization
  I. Kuběna, B. Fournier and T. Kruml: *J. Nucl. Mater.* 424 (2012) 101–108; B. Fournier et al.: *J. Nucl. Mater.* 430 (2012) 142–149; M.F. Giordana et al.: *Mater. Sci. Eng. A* 550 (2012) 103–111.
- TU Bergakademie Freiberg, Germany, Dr. A. Weidner, Prof. H. Bierman: in situ fatigue tests in the chamber of high-resolution SEM, characterization of dislocation structures using ECCI technique, cyclic slip localization
  J. Man et al.: *Proc. Eng.* **2** (2010) 1625–1633; A. Weidner et al.: *Int. J. Mater. Res.* 102 (2011) 1374–1377; J. Man, A. Weidner, P. Klapetek and J. Polák: *Key Eng. Mater.* 592–593 (2014) 785–788.
- University of Kaiserslautern, Germany, Dr. M. Smaga, Prof. D. Eifler: adoption of magnetic method for study of stability of austenitic stainless steels
  J. Man et al.: *Proc. Eng.* 10 (2011) 1279–1284.
- University of Oulu, Finland, Prof. L.P. Karjalainen: preparation of ultrafine- and coarse-grained austenitic steels in the form of sheets using special thermomechanical treatment (reversion annealing), mechanical properties of UFG materials
  A. Chlupová et al.: *Proc. Eng.* 74 (2014) 147–150.
- Sandvik Materials Technology and Linköping University Linköping, Sweden, Prof. G. Chai: high temperature resistance of advanced austenitic heat-resistant steel (Sanicro25)
  J. Polák et al.: *Mater. Sci. Eng. A* 615 (2014) 175–182; J. Polák et al.: *Int. J Fatigue* (2015) doi:10.1016/j.ijfatigue.2015.03.015.
- Karlsruhe Institute of Technology, Germany, S. Guth, Prof. K.-H. Lang: thermomechanical fatigue with and without dwells of superalloys
  S. Guth: *Int. J Fatigue* (2015) submitted for publication.

New facilities in the laboratories
The following facilities were purchased in the LCF group during the period 2010–14:
i) Electrohydraulic computer controlled axial/torsional test system MTS 809 (axial capacity: ± 100 kN, torsional capacity: ± 1100 N·m) (see Fig. 7). The system MTS 809 includes water-cooled hydraulic collet grips, room- and high-temperature axial/torsional extensometer and high temperature furnace up to 1400 °C.
ii) 10 kW induction furnace for thermomechanical fatigue (TMF) tests up to 1200 °C plus FlexTest SE digital control unit upgrade of MTS 880 testing system (see Fig. 8).

The system is used primarily for thermomechanical testing of materials but enables also performance of isothermal fatigue tests.

iii) Silent oil pumps for both axial/torsional and TMF test systems.

iv) Systems for air and water cooling (compressors and cooling unit with distilled water) for reliable performance of fatigue tests on electrohydraulic testing machines.

v) Feritscope Fischer FMP30 including accoutrements enables quantitative measurement of volume fraction of magnetic phase content (percentage of ferrite or deformation induced martensite in austenitic stainless steels).

Fig. 7

Fig. 8

The items (i-iii) were purchased in the frame of CEITEC-IPM Brno (project No. CZ.1.05/1.1.00/02.0068, the Ministry of Education, Youth and Sports of the CR), the cooling systems partially from the grant projects running in the Low Cycle Fatigue Group and partially from institutional costs and Feritscope fully from the running grant project.

The testing system enabling biaxial fatigue testing at both room and high temperatures is, at present, intensively used by T. Kruml in the frame of his running grant project of the CSF (project No. 15-08826S, Damage mechanisms in multiaxial cyclic loading, 2015–17).

The TMF testing system is presently used for systematic studies of thermo-mechanical behaviour of 316L austenitic stainless steel (Dr. V. Škorík). Within the period 09–12 2014 it was employed by Dipl.-Ing. S. Guth (KIT Karlsruhe, Germany) for TMF testing of Ni based superalloy during his three-month study stay at IPM Brno. His study resulted in the common publication (S. Guth et al.: *Int. J. Fatigue*, 2015, submitted for publication).

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Electrical and Magnetic Properties Group

Last evaluation of the Team of Electrical and Magnetic Properties for 2005 – 2009 confirmed that the activities of the Group are reasonable and that they correspond with topical research trends of the scientific community in basic research in the field of materials sciences. Therefore for the period 2010-2014 we have proposed following focusing of our effort.

- theoretical studies of electronic and magnetic properties of disordered alloys, epitaxial multilayers, surfaces and interfaces as well as quantum-mechanical studies of extended defects in metallic materials
- experimental investigations of relations among structure and magnetic, transport and mechanical properties in metallic materials

In the first topic the research encompassed several topical fields as e.g. surface magnetism, magnetic exchange coupling and spin-dependent transport in multilayered systems, magnetic properties of amorphous materials, solute segregation in bulk disordered alloys and at grain boundaries and computer simulations of atomic configurations of defects. Quantum-mechanical and quantum-statistical methods are applied to these problems, and most studies are performed from the first principles. Important part is theoretical studies of dislocations in metals.

Dr. Martin Friák focused on (i) the fundamental materials properties of prospective metallic materials and (ii) a theory-guided materials design of new alloys for industrial applications. Regarding the first topic, Ni-based ultra-hard coating materials were studied jointly with Dr. David Holec from Montanuniversität Leoben in Leoben, Austria. Another project was focused on nitrides appearing during nitridization of steels. Specifically, thermodynamic, electronic, structural and elastic properties of Ni_4N allotropes were calculated by quantum-mechanical methods. A special attention was also paid to the functional materials, such as bio-compatible shape-memory NiTi that has a number of industrial as well as medical applications. Regarding phase transitions in metallic materials, the behavior of elemental Fe, Co, and Ni was analyzed during martensitic trigonal transformations.
As far as the second topic, the theory-guided materials design, is concerned, Dr. Friák and his former colleagues from the Max Planck Institute for Iron Research in Düsseldorf, Germany, have very successfully combined the theoretical and the experimental methods in studying and designing new ductile magnesium alloys. The research was motivated by the facts that (i) Mg alloys combine low weight with relative high strength and (ii) their wider use in automotive and transport industries would significantly reduce green-house emissions. An industrial production of these alloys has been, unfortunately, so far hindered by their low room-temperature ductility. Therefore, quantum-mechanical calculations were used to explain why rare-earth additions improve the ductility of Mg alloys. Selected alloys were synthesized and their mechanical properties tested (see Figure 1).

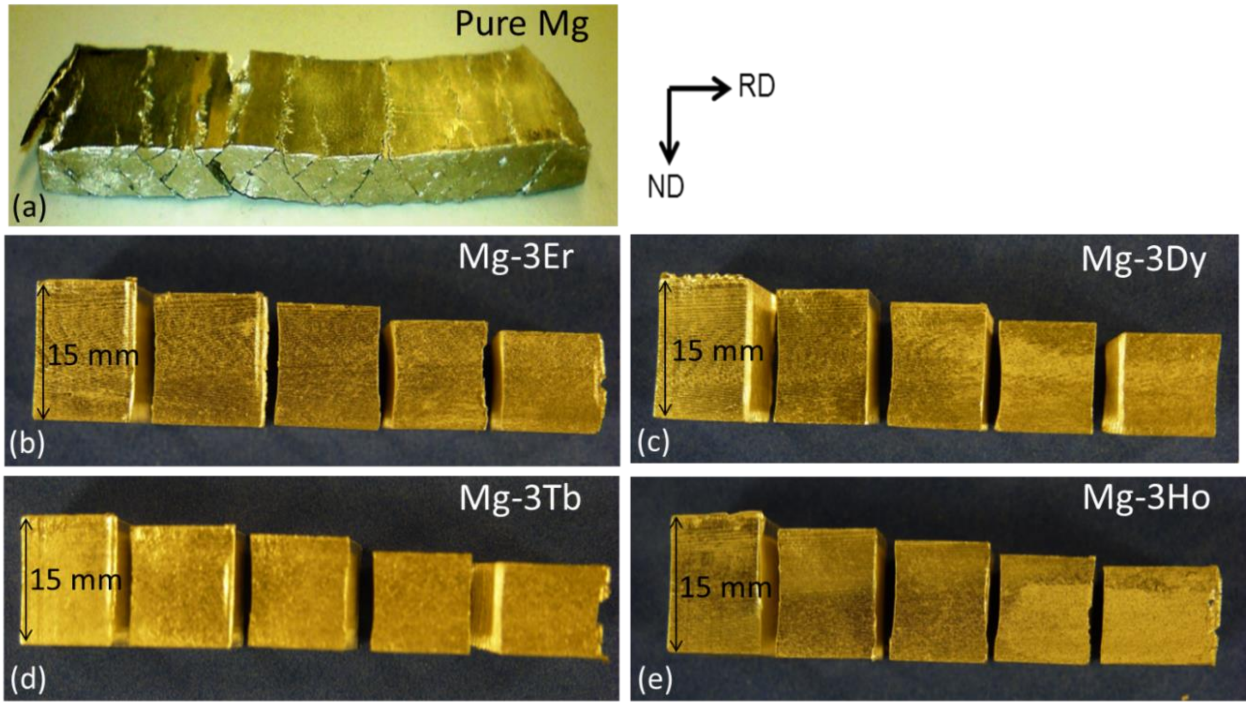


Figure 1 *Brittle Mg after mechanical loading (a) in comparison with ductile Mg alloys containing rare earth elements (b-e) exposed to the same mechanical loading conditions (for details see Acta Materialia 70 (2014) 92).*

Publications:

S. Sandlöbes, Z. Pei, M. Friák, L.-F. Zhu, F. Wang, S. Zaefferer, D. Raabe, J. Neugebauer, Acta Mater. 70 (2014) 92.

D. Holec, F. Tasnádi, P. Wagner, M. Friák, J. Neugebauer, P. H. Mayrhofer, J. Keckes, Phys. Rev. B 90 (2014) 184106.

D. Holec, M. Friák, A. Dlouhý, J. Neugebauer, Phys. Rev. B 89, 014110 (2014).

P. Hemzalová, M. Friák, M. Šob, D. Ma, A. Udyansky, D. Raabe, J. Neugebauer, Phys. Rev. B 88 (2013) 174103.

D. Holec, M. Friák, A. Dlouhý, J. Neugebauer, Phys. Rev. B 84 (2011) 224119.

M. Zelený, M. Friák, and M. Šob, Phys. Rev. B 83 (2011) 184424 (2011).

The following proposals were recently submitted:

- An ERC Consolidator Grant (call ERC-2014-CoG) grant proposal “Biomimetic design of self-similar metallic composites”, acronym "SELF-SIMILAR METALS".
- Horizon 2020 proposal (call H2020-FETOPEN-2014-2015-RIA) focused on 3D printing of metallic powders entitled “Additive Manufacturing of Particle Dispersion Strengthened High Entropy Alloys – A New Line of Technology”, acronym “HEATECH”, (coordinator: Max Planck Institute for Iron Research, Düsseldorf, Germany).
- Horizon 2020 proposal (call NMP 23 – 2015 Novel materials by design for substituting critical materials) entitled “Magnetic Materials – Search for new hard magnetic intermetallic phases without critical elements”, acronym “MagMa” (coordinator: Fraunhofer Instituite in Freiburg, Germany).
- Horizon 2020 Marie-Curie-Sklodowska Innovative training network (call H2020-MSCA-ITN-2015) proposal entitled "Corrosion science for innovative Material for

energy applications: synergy of theory and experimental studies of corrosion mechanisms on the nano-scale", acronym "Nano-CORR" (coordinator: University of Glasgow, UK).

Dr. I. Turek and his co-workers have developed an original approach to the theory of electron transport properties of magnetic metals, alloys and nanostructures. The formulation is based on a systematic neglect of intraatomic electron motion and on an explicit treatment of pure intersite electron hopping. The idea has been worked out within the first-principles relativistic tight-binding linear muffin-tin orbital (TB-LMTO) method, which provides a materials-specific theory of the conductivity tensor and of galvanomagnetic phenomena for ordered and disordered magnetic systems with potential applicability in spintronics. Concerning the theoretical aspects, we have shown that in disordered systems, the true but random velocity operators can be replaced by non-random effective velocities, which simplifies the configuration averaging in the coherent-potential approximation significantly. We have also analysed the so-called Fermi-sea contribution to the anomalous Hall conductivity and proved that this contribution in random alloys is purely coherent, with effective vertex corrections originating in the energetic dependence of the average one-particle propagator. Concerning specific applications to a wide class of bulk systems, the performed studies were focused on the residual resistivities, the anisotropic magnetoresistance (AMR) and the anomalous Hall effect and on their dependence on the atomic and magnetic structure of the alloys. The most important result has been achieved in the study of FeRh alloys with a CsCl structure, where our theoretical values of the AMR support the qualitative picture, obtained in a recent realization of the antiferromagnetic memory resistor. In particular, we have confirmed that the resistivity of this antiferromagnet for the electric current parallel to the local moments is bigger than that for the perpendicular orientation of the current and the moments, in full analogy with the sign of the AMR effect in ferromagnetic alloys known since the discovery of the AMR in 1857.

Publications:
I. Turek, J. Kudrnovský, V. Drchal, Phys. Rev. B 86 (2012) 014405.
X. Marti, I. Fina, C. Frontera, J. Liu, P. Wadley, Q. He, R.J. Paull, J.D. Clarkson, J. Kudrnovský, I. Turek, J. Kuneš, D. Yi, J.-H. Chu, C.T. Nelson, L. You, E. Arenholz, S. Salahuddin, J. Fontcuberta, T. Jungwirth, R. Ramesh, Nature Mater. 13 (2014) 367.
J. Kudrnovský, V. Drchal, I. Turek, Phys. Rev. B 88 (2013) 014422.
R. Sýkora, I. Turek: J. Phys., Condens. Matter 24 (2012) 365801.
J. Kudrnovský, V. Drchal, S. Khmelevskyi, I. Turek, Phys. Rev. B 84 (2011) 214436.
I. Turek, J. Kudrnovský, K. Carva:, Phys. Rev. B 86 (2012) 174430.
I. Turek, J. Kudrnovský, V. Drchal:, Phys. Rev. B 89 (2014) 064405.
J. Kudrnovský, V. Drchal, I. Turek, Phys. Rev. B 89 (2014) 224422.

Project: Theory of spin-dependent transport in magnetic solids and nanostructures, Investigator: doc. RNDr. Ilja Turek, DrSc.

Professor Mojmír Šob and his co-workers (Dr. Monika Všianská, Dr. Tomáš Káňa, Dr. Martin Zelený, and Dr. Martin Friák) employed ab initio electronic structure calculations to study a number of challenging scientific topics. First, they analyzed segregation of selected sp elements (Al, Si, P, S, Ga, Ge, As, Se, In, Sn, Sb, and Te) at the Σ5(210) grain boundary (GB) and (210) free surface (FS) in fcc ferromagnetic nickel. Most of these impurities nearly kill or substantially reduce the magnetic moments at the FS and, when segregating interstitially at the GB (i.e., Si, P, S, Ge, As, and Se), they produce magnetically dead layers at the boundary**.** It was demonstrated that the existence of magnetically dead layers is a common phenomenon at the sp-impurity-decorated GB and FS in nickel. This discovery may contribute to further development of materials for high-density magnetic recording.

Next, prospective materials for high-temperature applications, transition-metal disilicides, were studied. They are proposed to be suitable for devices working at high temperature, such as blades of turbines in aircraft engines or devices in furnaces. The ab initio study of Professor Šob and his colleagues connects the transition paths between the most common structures $C11_b$, C40 and C54 of $MoSi_2$, VSi_2 and $TiSi_2$ with the mutual shift of the (110), (0001) or (100) atomic planes in an attempt to shed more light on the deformation mechanisms and slips of those planes in the disilicides.

As yet another example, mechanical and magnetic properties of the Pt-rich Mn–Pt compounds $MnPt_3$, $MnPt_7$ and the $MnPt_{15}$ nanocomposite were investigated. Simulations of tensile and compressive tests have been done and the increase of compressive strength (and thus hardness) has been proven in contrast to elemental Pt. Both ferromagnetic and antiferromagnetic orderings of the studied materials have been theoretically investigated and it was shown that the $MnPt_7$ structure favors antiferromagnetism. However, when additional Pt sites in the Pt matrix are replaced by Mn atoms, the ferromagnetic ordering starts to apply locally in such sites. As a result, this theoretical research explains the so far unclear change of magnetic transition from antiferromagnetic to ferromagnetic in Pt-rich Mn–Pt structures with ~12.5 at. % Mn.

Publications:

M. Všianská, M. Šob, Progr. Mat. Sci. 56 (2011) 817.
M. Všianská, M. Šob, Phys. Rev. B 84 (2011) 014418.
T. Káňa, M. Šob, V. Vitek:, Intermetallics 19 (2011) 919.
T. Káňa and M. Šob, Phys. Rev. B 85 (2012) 214438.
T. Káňa, M. Šob, V. Vitek, Intermetallics 19 (2011) 919.
M. Zelený, M. Friák, M. Šob, Phys. Rev. B 83 (2011) 184424.

Projects:

- Strength and magnetism of composites, Investigator: prof. RNDr. Mojmír Šob, DrSc.
- Theoretical and experimental study of interfaces and martensitic phase transitions Investigator: prof. RNDr. Mojmír Šob, DrSc.

Dr. Roman Gröger dealt with stress dependence of the Peierls barrier of 1/2<111> screw dislocations in BCC metals. The recently formulated constrained Nudged Elastic Band method with atomic relaxations (NEB+r) is used to investigate the dependence of the Peierls barrier of 1/2<111> screw dislocations in body-centered cubic metals on non-glide stresses. These are the shear stresses parallel to the slip direction acting in the planes of the <111> zone different from the slip plane, and the shear stresses perpendicular to the slip direction. Both these shear stresses modify the structure of the dislocation core and thus alter both the Peierls barrier and the related Peierls stress. Understanding of this effect of loading is crucial for the development of mesoscopic models of thermally activated dislocation motion via formation and propagation of pairs of kinks. The Peierls stresses and related choices of the glide planes determined from the Peierls barriers agree with the results of molecular statics calculations, which demonstrates that the NEB+r method is a reliable tool for determining the variation in the Peierls barrier with the applied stress. However, such calculations are very time consuming, and it is shown here that an approximate approach of determining the stress dependence of the Peierls barrier can be used, combined with test calculations employing the NEB+r method.

Publications:

R. Gröger, T. Lookman, A. Saxena, Phys. Rev. B 82 (2010) 144104

R. Gröger, K. J. Dudeck, P. D. Nellist, V. Vitek, P. B. Hirsch, D. J. H. Cockayne, *Philos. Mag. 91 (2011)* 2364-2381

R. Gröger, V. Vitek, Model. Simul. Mater. Sci. Eng. 20 (2012) 035019

K. Srivastava, R. Gröger, D. Weygand, P. Gumbsch, Int. J. Plast. 47 (2013) 126-142

R. Gröger, V. Vitek, Acta Mater. 61 (2013) 6362-6371

A. Ostapovets, R. Gröger, Model. Simul. Mater. Sci. Eng. 22 (2014) 025015

R. Gröger, *Philos. Mag. 94 (*2014) 2021-2030

The following proposals were submitted

- an ERC Consolidator grant (call ERC-2014-CoG) "Dislocations in nitride-based epitaxial heterostructures", acronym "DANTE",
- H2020-TWINN-2015 "Computer aided sub-Angström electron microscopy of crystal defects", participants Dr. Roman Gröger from IPM in Brno, University of Oxford (UK), and École Polytechnique Fédérale de Lausanne (Switzerland)

Projects:

- Calculation of the Peierls barrier in bcc metals and its dependence on stress, Investigator: Ing. Roman Gröger, Ph.D.
- Mesoscopic framework for modeling physical processes in multiphase materials with defects, Investigator: Ing. Roman Gröger, Ph.D.

The second of the mentioned topics is based on broad experimental macroscopic and microscopic investigations of crystal structure in relation to electrical and magnetic properties, both integral and microscopic. Predominate amount of results has been obtained from applications of Mossbauer spectroscopy. Crystalline, microcrystalline, nanocrystalline and amorphous materials have been investigated. The main idea is to obtain a deeper understanding of relations between the changes in crystalline structure in dependence on the heat and mechanical treatment, and the electrical and magnetic properties.

Dr. Jirásková and co-workers have investigated materials prepared mainly by planar flow casting (PFC) and mechanical alloying (MA). Both technologies allow synthesizing materials of micro-, nano-crystalline and/or amorphous structures yielding new aspects to be solved, new physical properties and new possibilities for future technical applications. The bilayered ribbons (PFC) were studied from the viewpoint of interlayer formation and its effect on the physical properties important for sensor applications. The enlargement of knowledge concerning the structure, defects, and physical properties was obtained by a production of alloys using solid state diffusion (MA). It was found that this procedure allows, e.g., to enhance a mutual solubility of elements (Fe-Mo, Fe-Al-Mo) or to influence a dynamics of alloying and phase composition by milling conditions mainly atmosphere (H_2, N_2, air) observed at Fe-Al and Fe-Pd systems.

Publications:
Y. Jirásková, J. Buršík, I. Turek: J. Supercond. Nov. Magn. 26 (2013) 1717-1721.
Y. Jirásková, J. Buršík, J. Cizek, D. Jancik: J. Alloys Comp. 568 (2013) 106-111.
Y. Jirásková, J. Buršík, I. Turek, M. Hapla, A. Titov, O. Zivotsky: J. Alloys Comp. 594 (2014) 133-140.
Y. Jirásková, J. Buršík, O. Životský, J. Čuda: Mat. Sci. Eng. B 186 (2014) 73–78.
A. Titov, O. Životský, Y. Jirásková, A. Hendrych, J. Buršík, P. Švec: IEEE Trans. Magn. 50 (2014) 6500804.
Y.Jiraskova, A.Hendrych, O. Zivotsky, J. Bursik, T. Zak, I., Prochazka, D. Janickovic, Appl. Surf. Sci. 276 (2013) 68–75.

Projects:
Effects of cores and boundaries of nanograins on the structural and physical properties of ball milled and mechanically alloyed iron-based materials, Investigator: Ing. Yvonna Jirásková, Ph.D.,
Microstructure of Fe-Al based alloys, Co-investigator: Ing. Yvonna Jirásková, Ph.D.

Dr. Schneeweiss and co-workers dealt with the preparation of nanocrystalline powders of iron based materials by solid state reactions and by the method of laser-induced homogeneous pyrolysis of gaseous precursors. They used natural ferrihydrite as the precursors for the preparation of the nanocrystalline alpha-Fe powder using the heat treatment in hydrogen atmosphere. The kinetics of the transformation was described using Avrami relation applied on the data of the isothermal time dependence of magnetic moment measurements and subsequently the activation enthalpy of transformation was estimated using Arrhenius plots of their parameters. Comparison of the changes of activation enthalpies derived from a different temperature ranges indicated effects of different processes on formation of alpha-Fe nanoparticles.
By the laser-induced homogeneous pyrolysis of gaseous precursors several types of iron oxides and carbides nanoparticles were prepared. They were reinvestigated from the view of particle size and their magnetic behavior. It was shown that the nanoparticles exhibit reduced Curie temperatures and smeared ferromagnetic–paramagnetic transition.

A series of amorphous alloys with various iron content have been studied by Dr. Žák and collaborators. As a result of the preparation process of Fe81Si4B13C2 amorphous alloy ribbon, a difference has been observed between the opposite sides of the ribbon in microstructure and surface morphology. Influence of these differences on thermally induced structural transformations was studied. The influence of thermal treatment on functional properties of Fe75Ni2Si8B13C2 amorphous alloy shown thermally induced structural transformations. Thermal history of the sample was found to have a significant effect on magnetic properties. Study of Fe73.5Cu1Nb3Si15.5B7 amorphous alloy showed that it undergoes a series of thermally induced structural transformations in temperature region between 25 and

800 °C, including structural relaxation, two Curie temperatures and crystallization. Its magnetic, electrical and mechanical properties of the alloys are heavily influenced by the observed structural changes.

Publications:
O. Schneeweiss, J. Filip, B. David, R. Zboril, M. Maslan, J. Nanopart Res 13 (2011) 5677.
O. Schneeweiss, B. David, T. Žák, J. Filip, J. Tuček, R. Zbořil, Acta Physica Polonica A 118 (2010) 749.
B. David, O. Schneeweiss, N. Pizurova, F. Dumitrache, C. Fleaca, R. Alexandrescu: Surface Interface Analysis, 42 (2010) 699
B. David, N. Pizurova, P. Synek, V. Kudrle, O. Jasek, O. Schneeweiss: Mater. Letters (2014) 370.
Dušan M. Minić, V. A. Blagojević, B. David, N. Pizúrová, T. Žák, Dragica M. Minić, Intermetallics 25 (2012) 75
Dragica M. Minić, V.A. Blagojević, Dušan M. Minić, B. David, N. Pizúrová, T. Žák, Metallurgical and Materials Transactions A 43 (2012) 3062.
B. Cekić, V. Ivanovski, T. Žák, D. Stojić, J. Belošević-Čavor, V. Koteski, A. Umicević, Romanian Journal of Physics 56 (2011) 719.
T. Žák, N. Talijan, V. Ćosović, J. Stajić-Trošić, A. Grujić, J. Serb. Chem. Soc. 75 (2010) 1271.
Dragica M. Minić, V.A. Blagojević, Dušan M. Minić, A. Gavrilović, L. Rafailović, T. Žák, Metall. and Mat. Trans. A 42A (2011) 4106
Dušan M. Minić, V.A. Blagojević, A.M. Maričić, T. Žák, Dragica M. Minić, Mat. Chem. Phys. 134 (2012) 111.
V. A. Blagojević, Dušan M. Minić, T. Žák, Dragica M. Minić, Intermetal. 19 (2011) 1780.
Dragica M. Minić, Dušan M. Minić, T. Žák, P. Roupcová, B. David, J. Magn. Magn. Mater. 323 (2011) 400.
Dragica M. Minić, V. A. Blagojević, Dušan M. Minić, T. Žák, Mat. Chem. Phys. 130 (2011) 980.
Projects:
Research center of powdered nanomaterials, Investigator: Ing. Oldřich Schneeweiss, DrSc.
Iron and iron oxide nanoparticles with applications in the magnetic separation processes, Investigator: Ing. Oldřich Schneeweiss, DrSc.

The above presented achievements document intensive collaborations with the other groups in the Institute as well as with the teams on universities and academic institutes in Czech Republic.
Following most important co-operations documented by common publications and projects may be mentioned:
1/ Charles University, Praha, Faculty of Mathematics and Physics
2/ Brno University of Technology
3/ Central European Institute of Technology - CEITEC
4/ Masaryk University, Brno, Faculty of Science
5/ Palacky University, Olomouc, Faculty of Science
6/ Institute of Physics AS CR, Prague
7/ VSB-Technical University, Ostrava

The EMP team carried out several important international co-operations. The most important partners were from following institutions:

1. Max Planck Institute for Iron Research in Düsseldorf, Germany
Topic of collaboration: Research of novel materials
2. University of Pennsylvania, USA
Topic of collaboration: Dislocations in metals
3. Forschungszentrum Jülich, Germany
Topic of collaboration: Towards Atomistic Materials Design
4. Kyoto University, Japan
Topic of collaboration: Theoretical and experimental study of mechanical properties of disilicides of transition metals
5. Institute of Chemistry, Technology and Metallurgy, Belgrade, Serbia
Topic of collaboration: Research and development of functional nanomaterials for various applications
6. Institute of Physics, Slovak Academy of Sciences, Bratislava, Slovakia
Topic of collaboration: Nanocrystalline materials, preparations and properties
7. Technische Universität Ilmenau, Ilmenau, Germany
Topic of collaboration: Storage of energy by decentralized sources – proposals of common projects (the iMUSEUM consortium)
8. University of Oxford, UK
Topic: Computer-assisted HRTEM of lattice defects in metals and semiconductors
9. Los Alamos National Laboratory, USA
Topic: Incorporation of dislocation plasticity into phase field models of multiferroics
10. École Polytechnique Fédérale de Lausanne, Switzerland
Topic: Experimental studies of anomalous slip in bcc metals
11. Ghent University, Belgium, Dept. of Physics and Astronomy
Topic: Surface microstructure studied by Mössbauer spectroscopy of conversion and low-energy electrons
12. Montanuniversität Leoben, Leoben, Austria
Topic: development of new functional materials (such as bio-compatible NiTi, or novel ultra-hard coating materials for machining industry).

During the last five years, our lab was equipped by two new equipments:

1/ Physical Property Measurement System (PPMS©) 9T EverCool II (2 - 400 K; 0 - 9 T) (http://www.qdusa.com/products/ppms.html) with options: vibrating sample magnetometer (ULF, LB), DC resistivity option, ACT electro-transport option, Hall effect, thermal transport, heat capacity

2/ Remagraph-Remacomp Combination C710 (http://www.magnet-physik.de/messtechnik.html?&L=1), with options: MCE 1200N Epstein frame, MJR 5 measuring yoke, MJC coil system.

Research Report of the team in the period 2010–2014

Institute	Institute of Physics of Materials of the CAS, v. v. i.

Scientific team	Structure of Phases and Thermodynamics Group

The Structure of Phases and Thermodynamics Group was created by merging two small groups (Structure of Phases Group and Diffusion and Thermodynamics Group) after the last evaluation in 2010 as a response to the comments of the reviewers who pointed out the small size of Groups and relatively close topics solved by both teams. The cooperation between the scientists consequently increased, and it is also shown in the joint publications. As the projects solved by researchers from both former groups were already set and running, informal mutual contacts are now established in much greater extent, the experimental base of both former groups is much intensively shared by members of current group and more joint publications can be expected. The co-operation exists also with other scientific groups at the Institute, e.g. A. Zemanová participated in and was partially financed by the Operational Program of the MEYS headed by L. Náhlík in 2013-14.

The scientists of the Group have been working in three main research fields of serious importance both for the basic and applied research – theoretical and experimental modelling of phase diagrams, including size-dependent phase diagrams; studies of material structures by means of Analytical Electron Microscopy (AEM); measurements of diffusion characteristics and study of hydrogen desorption. Their work resulted not only in the high number of publications in renowned impacted scientific journals, but also in several practical results, which can be used in the applied research, materials development and industry. Their experience was also used in number of contracts with industrial partners (Form 3-9), where they helped them in problem solutions and long-term evaluation of the product quality.

2.1. Theoretical Modelling of Phase Diagrams

2.1.1. Unification of Models in Complex Metallic Materials

One of the key themes of the research group is the modelling of phase diagrams of complex metallic systems. The study of materials suitable for high temperature lead-free soldering was important topic in the years 2010-2014. The lead-containing materials are still used for HT soldering (in the temperature region between 250 and 350 °C) because of the lack of non-hazardous substitution and as such, they are currently exempt from the EU legislation about restriction of hazardous materials. Projects were mainly oriented towards modelling of phase diagrams of relevant systems and creation of thermodynamic database for such systems. This was followed by the project devoted to the modelling of size-dependent phase diagrams (aimed towards the modelling of phase diagrams of nanomaterials), where novel and original theoretical model is under development and the theoretical results are verified by the experiments on systems related to the HT lead-free soldering, as using nanomaterials in HT soldering and joining is one of the directions studied by research teams throughout the world.

In the scope of projects belonging to this field of research, the most important one was the international COST MP0602 project “Advanced Solder Materials for High Temperature Application (HISOLD)” running in years 2007-2011, led by A. Kroupa in the position of the Chair of Management Committee. This project was oriented to the study of properties of lead-free materials suitable for high-temperature soldering. More than 60 research teams from 20 EU countries and from Russia, Ukraine and Argentina took part in this project and presented important information about possible lead-free materials and/or new lead-free technologies usable in this field. The results of the project were published in the form of 3-volume publication “Handbook of High-Temperature Lead-Free Solders” by the COST office.

A. Kroupa and his research team at the IPM AS CR were co-authors of the volume 1 – “Dinsdale A., Kroupa A., Watson A., Vřešťál J., Zemanová A**.**, Brož P.: Atlas of Phase Diagrams”, together with the colleagues from National Physical Laboratory in Teddington, University of Leeds and Masaryk University, Brno, where the phase diagrams of binary and ternary systems important for possible lead-free solders were published. A. Kroupa was also the editor of the volume 3 – “Group Project reports” - where the overview of scientific results obtained in the scope of broad international co-operation was presented. The Volume 2 of this publication - “Materials Properties” - lists the values of thermophysical properties and some mechanical properties obtained in the scope of the project by various research teams.

The phase diagrams in the volume 1 of the publication were calculated by the CALPHAD method using the thermodynamic database developed by the co-authors in the scope of the project. This database is now used by many research teams for their own research projects. Currently, the MP 0602 database contains 18 elements: Ag, Al, Au, Bi, Co, Cu, Ga, Ge, Mg, Ni, P, Pb, Pd, Sb, Si, Sn, Ti and Zn. The database is being constructed from assessments taken from the scientific literature and those generated through the research programmes of research teams participating in above mentioned MP0602 including our own research group. At each stage, the assessments have been modified where necessary, in order to fulfil the consistency criteria. This criteria and more details about the work done in the scope of the COST project are presented in the book “*Lead-free Solders: Materials Reliability for Electronics*”, edited by K.N. Subramanian and published by John Willey and Sons Ltd in 2012. A. Kroupa and A. Zemanová are co-authors of chapter 3, “*Phase diagrams and alloy development*”. A number of distinct material types have been identified by various authors as having potential for use as high temperature lead free solders, alloys based on the Al-Zn, Sb-Sn, Zn-Sn systems etc. These are included in the database but it is not necessary to have thermodynamic descriptions of all combinations of the elements in the database and so its coverage was limited to those binary and ternary systems with particular relevance to lead free soldering generally, not only for materials for high temperature region.

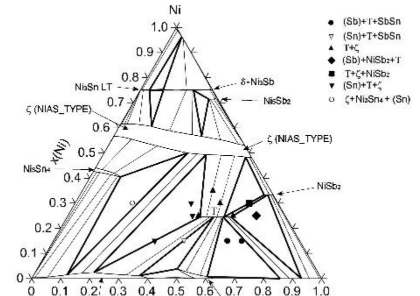
Except for the construction of the database, where they worked on evaluation of approx. 1/3 of the systems included in the database and were crucial in the technical issues connected with the preparation of the book publication, the members of this research group studied mainly the systems Ni-Sb-Sn and Sn-Zn-X. In the frame of that they had concentrated on the creation of the complete thermodynamic assessments (creation of the set of thermodynamic parameters which fully describes the thermodynamic properties of the system), which became part of the database. Important part of that was the unification of the models used for the description of the intermetallic phases existing in more binary or ternary systems. Currently, the same phases are often modelled by different models in different system depending on the analysis of authors of those papers. To allow the extension of calculations to multicomponent systems using specialized databases as this, created in the scope of COST project, all identical phases need to be modelled by the same model. This usually means that the complete reassessment of the system has to be done. With respect to the extent of the database, this work is still running and new version of the database is planned.

After the end of the COST project in 2011, the work on this topic continued and was extended into two directions. One project is devoted to the modelling of intermetallic phases, unification of their models with special attention being given to the intermetallic phases which play important role in materials related to the soldering (e.g. NiAs type and γ-brass phases). As mentioned above, the second project is oriented towards the modelling of size-dependent phase diagrams, which are crucial for the study of phase diagrams of nanomaterials. Both projects used, as a methodology, the combination of semiempirical CALPHAD method with the ab initio calculations. The results of those calculations allows us to obtain physically sound values for energies or materials properties, which are not available experimentally. For the first project, assessments were published of Ni-Sn and Ni-Sb-Sn systems up to now in the journals, where the unification of the models for the NiAs-type and Ni_3Sn-type phases were done (Fig. 1) and the results of the model unification of γ-brass phase was presented at the international conference CALPHAD in 2014.

2.1.2. Nanomaterials and Modelling of Size-dependent Phase Diagrams

New and original method for the modelling of the complex size-dependent phase diagrams with intermetallic phases was developed in co-operation with the team from "Electrical and Magnetic Properties Group". Here the ab initio calculations were used to calculate the surface stress of intermetallic phases, which are not available from experiment. New model is under construction for the modelling of surface stress of nonstoichiometric intermetallic phases. This values are used in the standard CALPHAD model which allows to calculate the contribution of the surface energy to the total Gibbs energy of the system. Results of this new approach were presented at the Nanocon 2012 (A. Kroupa, T. Káňa, A. Zemanová: CALPHAD type modelling of phase diagrams of nanomaterials, in Conference proceedings NANOCON 2012, Oct. 2012, Brno, Czech Republic) and CALPHAD 2013 conferences (Fig. 2). Publication was accepted for the Physical Chemistry Chemical Physics journal in 2015.

The results of both mentioned research topics were published in impacted international journals and on international conferences. 12 papers were published in the journals and 2 in the conference proceedings. Most interesting publications are shown in the List of selected publications of the SPaT group for this Evaluation under the reference numbers [3,4].



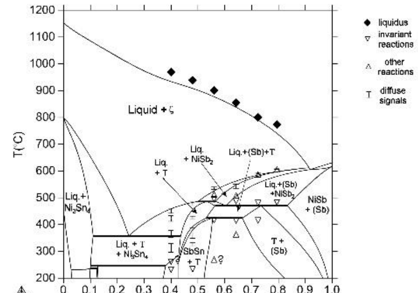


Fig. 1: Example of the assessment results for Ni-Sb-Sn system, the isothermal section for 400 °C, and the isopleth between Ni_3Sn_4 and Sb.

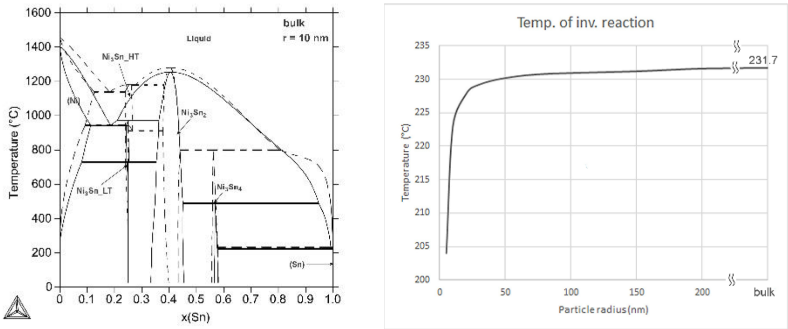


Fig. 2: The comparison of calculated phase diagrams for the Ni-Sn system for the bulk and radius of particles 10 nm, and the value of the temperature of invariant reaction (L→Sn+Ni_3Sn_4) in dependence on particle radius.

The nanomaterials for lead free soldering were studied also from the experimental point of view in the Group, exploiting also the second crucial research field of the SFaT Group - the study of structure of materials by means of AEM. This part of the work was studied in the scope of CSF project in co-operation with the Department of Chemistry, Faculty of Science, Masaryk University, Brno. The materials studied were nature-friendly and non-toxic nanopowders of pure metals and alloys (Cu, Ag, Sn, Sn-Zn, Sn-Ni). We studied nanopowders on their own as well as structures substrate/nanopowder/substrate. Interactions of nanoparticles with each other, interactions of nanoparticles with the surrounding atmosphere (gaseous oxygen) and with the substrate (Cu, Ag, Ni, Sn-Zn, Sn-Ni) were studied. An important feature of the use of metal nanoparticles was a decrease of their melting point in comparison with the corresponding bulk material. This behaviour was investigated by the thermal analysis (DTA and DSC) partially in our laboratory, and also at the Masaryk University. The oxidation of nanoparticles was investigated using the termogravimetry (DSC/TG) at MU. The interactions of nanoparticles with the substrate and the formation of intermetallic phases were analysed using the AEM exclusively in our Group. The results were published in 7 papers in impacted international journal (e.g. Zobač O., Sopoušek J., Buršík J., Zemanová A., Roupcová P.: Met. Mater. Trans A, 45A(2014), 1181-1188, DOI: 10.1007/s11661-013-2104-1).

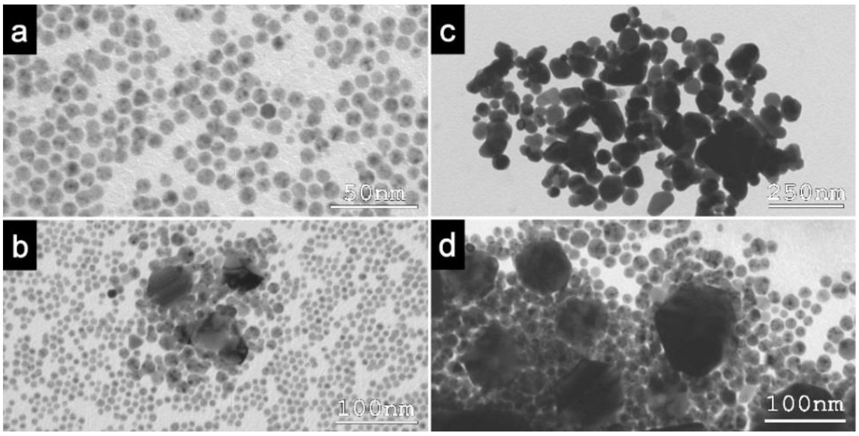


Fig. 3: TEM micrographs of in-house prepared Ag nanoparticles (a,b), a commercial product (c) and an older variant of in-house prepared Ag nanoparticles (d).

2.2. AEM Studies on Relations between the Structure and Properties of Materials

The AEM methods were applied by members of our Group for the study of the nucleation and growth of oxygen precipitates in silicon. This CSF project was solved in co-operation with the Department of Condensed Matter Physics, Faculty of Science, Masaryk University, Brno. It was devoted to a complex study of nucleation and growth of oxygen precipitates in Czochralski grown silicon crystals. A broad range of experimental methods was applied: X-ray scattering, infrared absorption spectroscopy, wet etching, optical microscopy and transmission electron microscopy; their results were compared in detail. These methods allowed us to characterize both globally and locally the distribution of precipitates (size, density of clusters) and of the associated defects (crystal lattice deformation) in samples subjected to the controlled annealing processes. Namely the TEM observations performed solely by our Group were very profitable in critical evaluation of all data (Meduna M., Růzička J., Caha O., Buršík J., Svoboda M.: PHYSICA B-COND. MATTER, 407(2012), 3002-3005, DOI: 10.1016/j.physb.2011.08.063). The experimental results were compared with theoretical modelling, based on theories of nucleation and growth, going beyond the classical theory of nucleation. The numerical analysis allowed us to determine the associated material constants needed for appropriate numerical modelling of the precipitate growth. The prediction and understanding of precipitation processes kinetics lead to development of recipes for annealing treatments resulting in controlled sample parameters. Results were published in 4 papers in international journals.

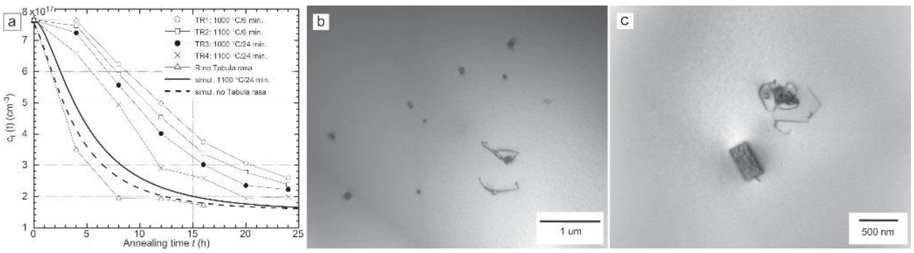


Fig. 4: Decrease of interstitial oxygen during precipitation annealing at temperature 1000 °C for samples differing in pre-annealing process (so called Tabula Rasa, TR) (a). Connected points correspond to experimental data obtained from IR spectroscopy; plain lines are the simulations. Typical TEM images showing SiO_x precipitates in the sample without (b) and with (c) TR process applied.

TEM expertise of our Group was also used for collaboration with team of Prof. Rogl at the University of Vienna. We worked on microstructure studies and crystal structure identification of phases in various ternary systems, namely Ti-Ni-Al, (Zr,Hf)-Ni-Al, Ta-V-Ge, Ta-V-Si, Ni-Zn-B and (Ta,Nb)-Co-B. TEM was employed e.g. to corroborate unit cell, symmetry and formation of superstructures in τ_2-Ta(Ta,V,Ge)$_2$ ternary Laves phase and to confirm the crystal symmetry, the dimensions of the unit cell and the absence of superstructure reflections in τ_5-TiNi$_{(2-x)}$Al$_5$, x=0.48 ternary phase. The results were published in 6 papers in international journals. Detailed results can be found e.g. in Ref. [11,12] in the List of Selected Publications for SPaT group.

Integral part of the structural research in this Group is devoted to the studies of phase structures after specific loading conditions and treatments. The link between the microstructure and the creep behaviour of precipitate strengthened alloys processed by ECAP (Equal Channel Angular Pressing) was studied in the scope of CSF project. Detailed examination of the effect of precipitates on the microstructural stability and the creep resistance of the selected precipitate strengthened aluminium and copper based alloys processed by SPD (Severe Plastic Deformation), mostly ECAP, was carried out. The results should allow to develop the optimum processing schemes for attaining the desired creep properties of SPD processed materials. Three main tasks were followed, namely:

- *Effect of the initial microstructure (coarse-grained materials) on its ECAP microstructure:* The comparison of grain size of selected UFG (Ultra Fine Grains) Cu alloys indicates that pre-ECAP solid solution plus post-ECAP low temperature precipitation treatment could be more effective for thermal stability of the microstructure than the short precipitation treatment at higher temperature before ECAP. It was observed that microstructure after creep contained significantly larger grains in comparison with the mean grain size observed in the microstructure after static annealing. It means that the microstructure after creep is significantly more influenced by the dynamic processes than the deformation-induced grain growth or dynamic recrystallization.

- *Microstructure changes and the texture developed during creep testing evaluated by analytical microscopic techniques:* The static recrystallization during heating to creep test temperature generates a microcrystalline structure; its homogeneity increases with the increasing ECAP-predeformation. The high-angle boundaries are sufficiently closely spaced to exert a significant influence on work hardening and quasi-stationary deformation where the generation and the loss of free dislocations are at approximate balance.

- *The link between the ECAP microstructure and the creep behaviour in ultrafine-grained materials:* The microstructural investigations and the creep testing of all routes examined indicated a little difference in the grain size and the creep behaviour of the pressed materials. It was generally observed that with increasing number of ECAP passes, a considerable amount of subgrain boundaries was gradually transformed to high-angle grain boundaries. At the same time, the local homogeneity of structure with the increasing number of ECAP passes was gradually improved. In such circumstances, a great variability of creep life and fracture ductility is a natural consequence of the short as well as long-range microstructure inhomogeneity. These result and the whole project is an example of close collaboration within the Institute of Physics of Materials. The results obtained together with the researcher from the Advanced High-Temperature Materials Group who carried out creep tests and creep behaviour interpretation. Details were published e.g. in V. Sklenička, J. Dvořák, P. Král, M. Svoboda, et. al.: Mater. Sci. Eng. A 558/(2012), 403-411, DOI:10.1016/j.msea.2012.08.019.

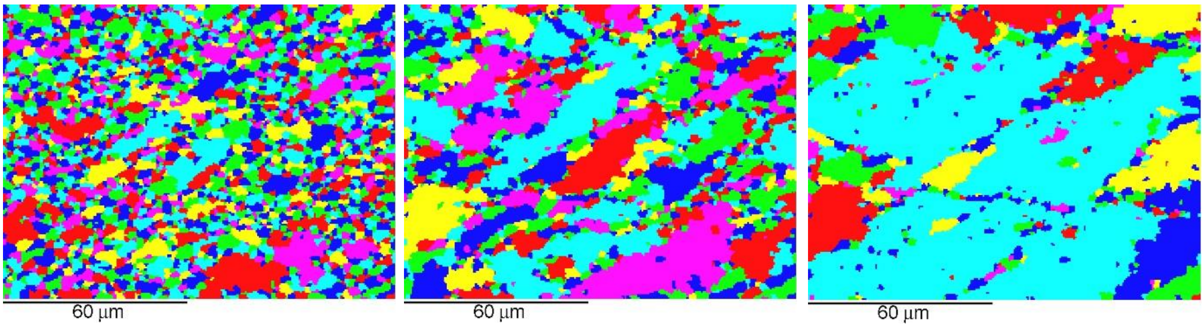


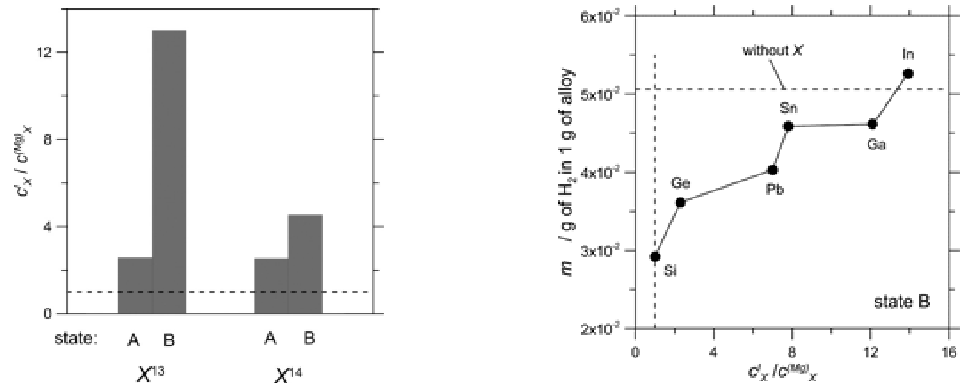
Fig. 5: Selected examples of EBSD maps of an Al-0.2wt.%Sc alloys after ECAP taken at different ranges of EBSD misorientation Δ showing: (a) subboundaries $\Delta \geq 2°$, (b) transitive subboundaries and high-angle boundaries $\Delta \geq 10°$ and, (c) mostly high-angle grain boundaries $\Delta \geq 15°$ (from left to right).

2.3. Diffusion and Transport Properties, Hydrogen Storage Studies

Important results were obtained in the field of hydrogen desorption and their results have both scientific value and importance for practical application. The project was devoted to Hydrogen storage materials in complex hydrides based on Mg. Mg is very cheap metal that is abundant in the Earth crust, it can be relatively easily recycled from the waste and it is biocompatible. Hydride of Mg, MgH_2, is known for relatively high hydrogen content (7.6 wt. % H_2) and low specific gravity. These are the main reasons for extraordinary interest of R/D sphere devoted to effective energy storage/transport.

Hydrogen storage in pure Mg, however, suffers from substantial problems, the most serious being poor hydrogen sorption kinetics. The hydride MgH_2 is, moreover, very stable and hence, the activation energy of hydrogen desorption is too high. Nevertheless, the invitingly high hydrogen storage capacity remains the main reason that attracts constant interest of both basic and applied research.

We contributed to the intensive research directed to the improvement of the hydrogen sorption kinetics: Complex materials with Ni as the main catalyst and a series of added elements from 13th and 14th group were investigated. First of all, the phase composition of perspective ternary and quaternary alloys were studied and the preference of elemental additions to existing phases was found [7, 8]. It was found that additives show affinity to Ni-containing phase, which is even much more expressive in hydrogen-charged state – see Fig. 6a. This preference was correlated with storage characteristics, rate of sorption, ionization potential of additives etc. It was interesting that the storage capacity, e.g., increased with increasing index of separation – Fig. 6b. These studies enabled to find an optimum chemical composition of the storage alloy, which was successfully patent-protected (J. Čermák, L. Král, B. David: The porous material for hydrogen storage and method of his preparation [CZ patent, No. 302464, B6]) in 2011. We paid our attention to the explanation of spontaneous phase changes appearing in the hydrogen storage material during a prolonged time of service. We contributed to a better understanding of ageing process that affects hydrogen sorption process [9]. We also estimated relative catalytic efficiency of individual phases present in the hydrogen storage alloy ([10]). The scientific results were published in 9 international journals and 8 proceedings from conferences. Detailed results can be found e.g. in the Ref. [7-10] in the List of selected publications of the SPaT group.



a) b)

Fig. 6: a) Average concentration ratio of X (X – additives) in phase Mg_2NiH (I) and (Mg), respectively, measured for added elements from group 13 and 14 in discharged and charged states - A and B, respectively. Dashed line: $(c^I_X/c^{(Mg)}_X) = 1$ – no preference in distribution of X.

b) Correlation between hydrogen storage capacity m of Mg–Ni–X alloys and preference of X to phase I scaled by the ratio $(c^I_X/c^{(Mg)}_X)$. Dashed horizontal – practically attainable hydrogen storage capacity measured in non-alloyed binary alloy Mg–Ni, dashed vertical – $(c^I_X/c^{(Mg)}_X) = 1$ (no preference in distribution of X)

In the field of diffusion and transport properties, the long-term research programme is focused on the study of the relationship between the sample fabrication, thermal treatment, microstructure and transport behaviour in nickel. The motivation for this work was a large scatter in the available experimental data on the grain boundary self-diffusion in nickel. For microstructural characterization, light and electron microscopy techniques (LM, SEM, EBSD) were used. As a most sensitive tool to study the diffusion in metals, a radiotracer measurement method was chosen to investigate the transport properties. Preliminary experimental results were published mainly in the conference proceedings, because of a lack of a satisfactory theory to interpret the data. Currently, a theoretical model is developed to predict the diffusion behaviour in pure and commercial nickel.

Within this work, a new original technique was developed in our radioisotope laboratory and published (Rothova, V: Nucl. Instrum. Meth., A 2013, 729, 702−706, https://dx.doi.org/10.1016/j.nima.2013.08.038), which provides an effective and reproducible measurement of the diffusion profiles by means of the Gruzin's residual activity method and the liquid scintillation counter. The new method is original, simple, sensitive and fundamentally reduces the disposal costs (substantial reduction in the amount of solid radioactive waste and, as a crucial advantage, no liquid radioactive waste). Moreover, the method allows a reliable determination of very low-level activities when measuring the low-energy pure beta emitters (as are, for example, ^{1}H, ^{63}Ni and ^{14}C). Thus, this is very important for the nickel grain boundary self-diffusion studies conducted in our laboratory because the proportion of the diffusion paths is very low in the case of grain boundary diffusion.

2.4. International co-operation

The Structure of Phases and Thermodynamics research Group has very broad international contacts. The researchers from the Group cooperate intensively with Prof. G. Borzone from University of Genoa, and Prof. H. Ipser, K.W. Richter, H. Flandorfer from University of Vienna, Austria (lead free soldering), and P. Rogl, Univ. of Vienna (various ternary systems, Ti-Ni-Al, (Zr,Hf)-Ni-Al, Ta-V-Ge, Ta-V-Si, Ni-Zn-

B, Ta-Co-B, Nb-Co-B). They also work on joint topics in the field of soldering with researchers from industrial laboratories, e.g. Swerea (Sweden), Ceravision (UK), ITRI, (UK). Very intensive co-operation in the field of thermodynamic database development exists between A. Kroupa, A. Zemanová, J. Vřešťál, P. Brož (both from Masaryk University) and Dr. A.T. Dinsdale (NPL Teddington, UK) and Dr. A. Watson (University Leeds, UK). One of the results of this co-operation are the books on phase diagrams of systems for lead-free soldering, published in the scope of the COST MP0602 Action and the thermodynamic databases for lead-free solders MP0602. Also a chapter in book (*Lead-free Solders: Materials Reliability for Electronics*) was published by this international research team. Contacts were established also with Dr. Tofail Syed from University of Limerick in 2014 (nanomaterials, phase diagrams modelling).

Intensive co-operation exists with the teams of Prof. J. Janovec from Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava, and Dr. A. Výrostková from Institute for Materials Research SAS, Košice, Slovakia (research on carbidic and intermetallic phases in advanced steels, properties of superalloys, and properties of joints). Dr. Viera Homolová from IMR SAS, Košice cooperates with SPaT group in the phase diagram modelling of Fe-B-X systems.

A. Kroupa is a member of the MSIT team (Materials Science International Team, http://www.msiport.com/communities/msit/) and in the scope of projects related to this team, he published two evaluation of ternary systems (Kroupa A., Watson A., “C-Mo-Ti”, http://www.springermaterials.com/docs/info/978-3-642-02700-0_31.html, and Watson A., Kroupa A., “C-Cr-W”, http://www.springermaterials.com/docs/info/978-3-642-02700-0_25.html in LANDOLT-BOERNSTEIN, NUMERICAL DATA AND FUNCTIONAL RELATIONSHIPS IN SCIENCE AND TECHNOLOGY (NEW SERIES). GROUP IV*: Physical Chemistry.* Ed. W. Martiensen, "Ternary Alloy Systems. Phase Diagrams, Crystallographic and Thermodynamic Data", Vol. 11E2, G.Effenberg, S.Ilyenko (Eds.), Springer-Verlag, Berlin, Heidelberg, 2010, published electronically only).

2.5. New facilities in the laboratory

The SPaT Group extended significantly the experimental facilities in its laboratories. It has to be stressed that all of this new devices are used by researchers from the whole Institute and were obtained in the scope of institutional investments or from the financial support connected with our participation in the CEITEC-IPM project.

Thanks to mentioned support, two new Scanning Electron Microscopes and Transmission Electron Microscope with broad analytical equipment were purchased:

- FIB-FESEM Lyra 3 XMU by Tescan, equipped by Oxford Aztec X-Max 80 Premium EDS, and HKL EBSD NordlysNano, in 2011
- FESEM Lyra 3 XMH by Tescan, equipped by Oxford Aztec X-Max 80 Premium EDS, in 2014
- JEOL TEM, Field Emission Electron Microscope JEM-2100F, equipped by Oxford Aztec X-Max 80 Premium EDS, in 2013.
- PCT-pro SETARAM, fully automated apparatus for measurement of hydrogen sorption characteristics in hydrogen storage materials.