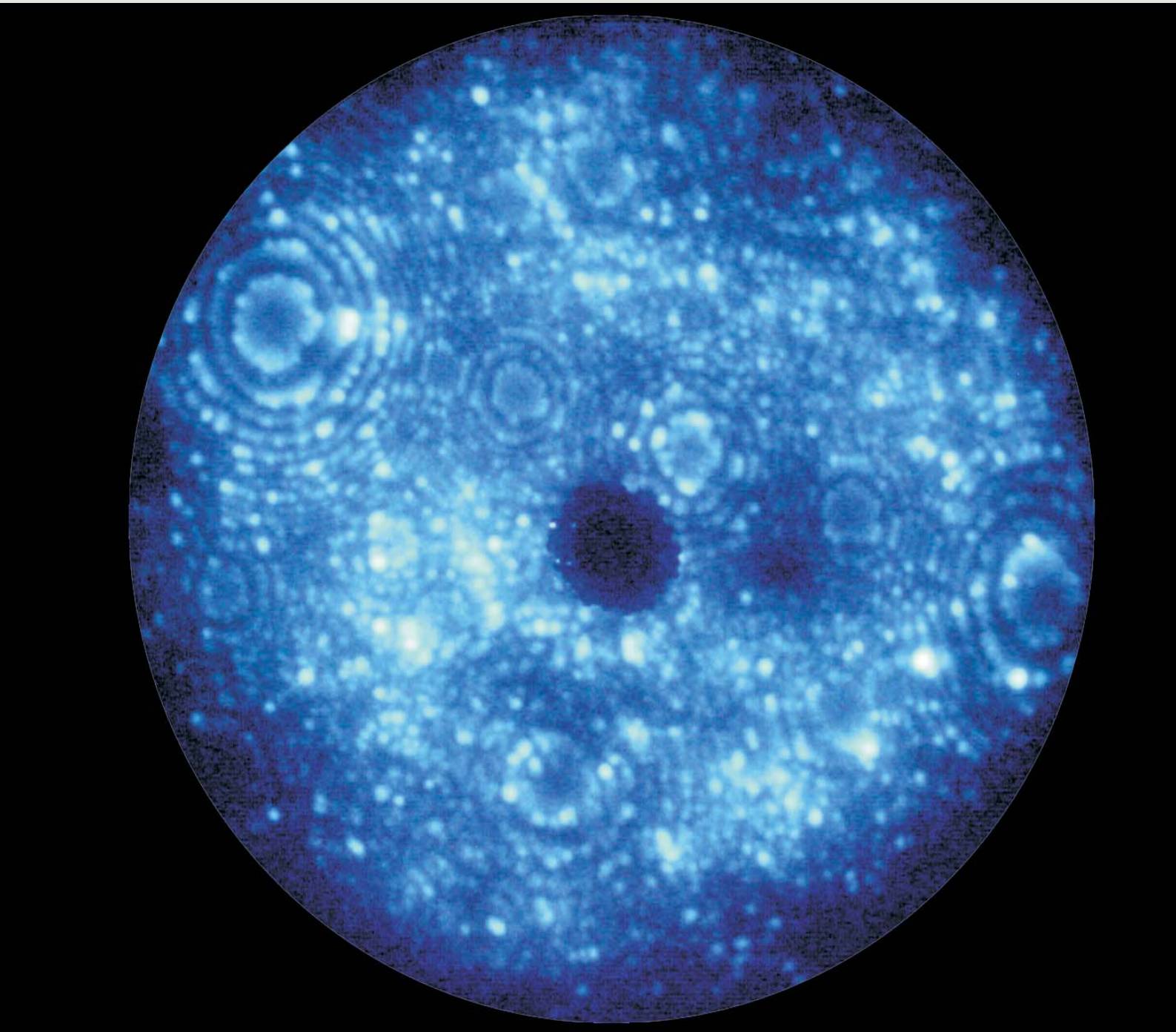
MAX-PLANCK-GESELLSCHAFT

Scientific Report 2001 / 2002

Max-Planck-Institut
für Eisenforschung GmbH



Max-Planck-Institut für Eisenforschung GmbH

Scientific Report 2001/2002

December 2002
Max-Planck-Institut für Eisenforschung GmbH
Max-Planck-Str. 1 • 40237 Düsseldorf
Germany

Front cover
Field ion image of a $D0_3$-ordered Fe_3Al alloy revealing atomic resolution. The concentric rings represent edges of atomic planes. The dark area in the centre of the image is the aperture for the time of flight analysis of field-desorbed atoms (atom probe field ion microscopy - APFIM).

Imprint

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PREFACE

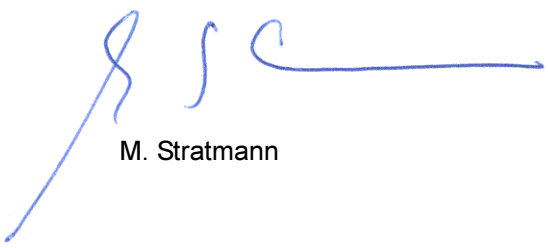
The institute has undertaken a number of changes over the last two years. Firstly, instead of a chief executive director as head, the institute established a structure similar to most other Max Planck Institutes. Namely, a board of executive directors was formed from the scientific members of the Max Planck Society, which is responsible for all institute affairs.

The institute has five departments. Two of the department head positions (Microstructure Physics and Metal Forming; Interface Chemistry and Surface Engineering) have recently been filled (D. Raabe, M. Stratmann). The third department (Physical Metallurgy) is presently headed by P. Neumann and upon his retirement will be devoted to the study of materials development. The fourth department (Materials Technology) is headed by G. Frommeyer and will merge with the new Department of Materials Development following his retirement. For the remaining department (Metallurgy and Process Technology) together with the planned Department of Materials Development, the institute is presently trying to identify new department heads.

Secondly, the institute is undergoing major renovation work. To a large extent, all institute buildings will be renovated and reconstructed, forcing all departments to either close part of their laboratories or move them during the renovation period. The first renovation stage was finished at the beginning of 2002; the second stage is due to be completed by the end of 2003 and the final stage will be carried out in 2004. The accumulated expenses will reach 20 million Euro and will be equally shared by the Max Planck Society and the Steel Institute (VDEh). The Max Planck Institute gratefully acknowledges this financial support. As a result of the renovations, cutting edge laboratories have been established with state-of-the-art instrumentation, some of which is described in Part I of this report.

Finally, the internal structure of the institute is due to ongoing changes and management reorganizations. The administrative services have been considerably reorganized, including the formation a central purchasing department. Furthermore, besides a departmental structure scientific areas of central importance have been established as a link between the departments which will serve as “focal points” in the institute. They will allow young scientists to strengthen their international reputation and scientific profile and will form a basis for future cooperative and interdisciplinary scientific work in areas of high relevance.

This report consists of three parts: Part I deals with the organization of the institute, its new laboratories in the renovated main building and its main scientific focus. Part II covers the departmental research activities and Part III summarizes the statistically relevant information of the institute.



M. Stratmann

CONTENT

PART I.

THE INSTITUTE

Structure and Organization

The Max Planck Institute for Iron Research is a joint venture between the Max Planck Society and the Steel Institute (VDEh). Since half of the institute's budget is supplied indirectly through industry, this institute is unique within the Max Planck Society.

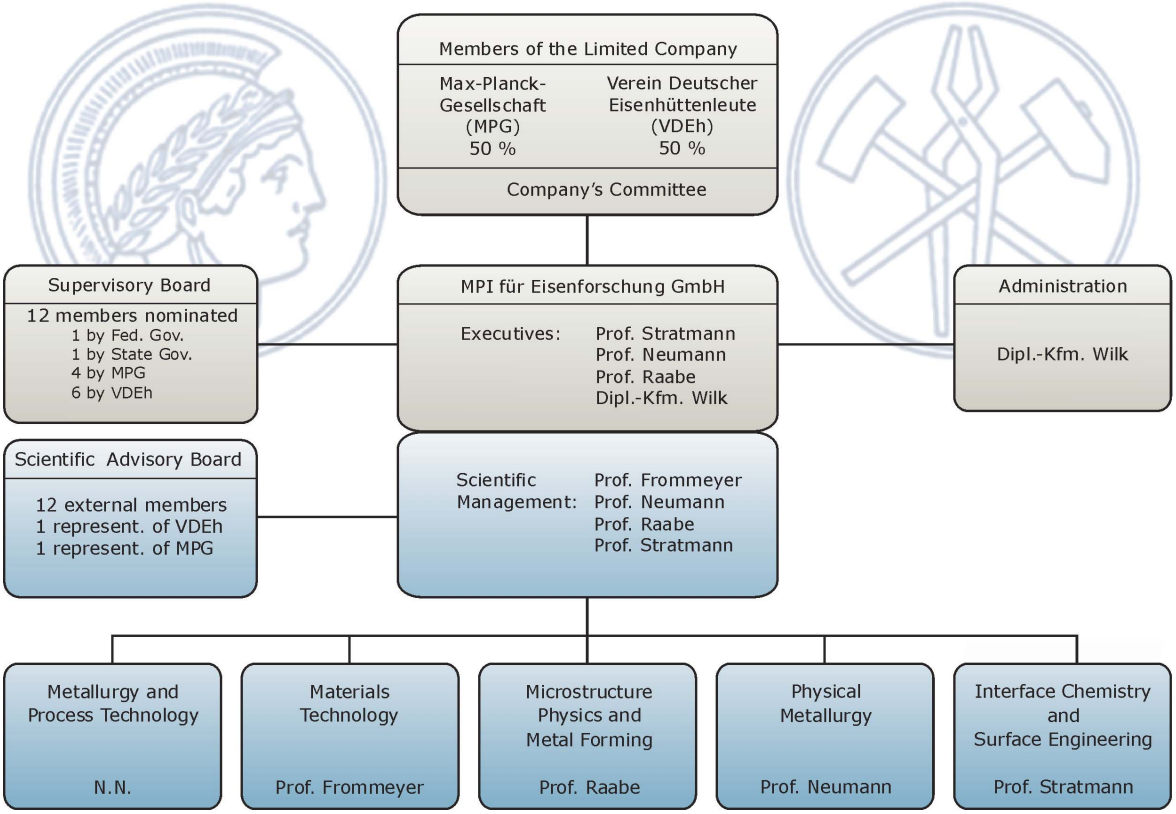
The institute was founded in 1917 by the Verein Deutscher Eisenhüttenleute (VDEh) and incorporated into the Kaiser Wilhelm Gesellschaft, the predecessor of the Max Planck Society. The institute was first located in Aachen and was associated with the Technical University of Aachen. The institute later moved in 1934/35 to its present location on a site donated by the city of Düsseldorf.

In 1946, the institute's heavily damaged buildings were reconstructed, work resumed and the institute was incorporated into the Max Planck Society. The institute rapidly expanded and new laboratory buildings were erected in the early 1960s. Following the appointment of H.J. Engell as director in 1971, a complete reorganization of the institute was carried out. Up until recently, the institute has been headed by a chief executive director (1971-1990: Prof. Engell, 1990-2002: Prof. Neumann) and an associated administrative director.

The internal structure of the institute changed again with the appointment of professors Raabe and Stratmann in 1999. Since then, all scientific members of the institute form an executive board of directors. The position of managing director will be filled, in rotation, by one of the board members. A board, which supervises the institution's activities, consists of representatives from the federal government, the state of Nordrhein-Westfalen, the Max Planck Society and the Steel Institute (VDEh). A Scientific Advisory Board comprised of prominent scientists assists the institute in finding the right balance between fundamental research and technological relevance.

From 1971 until the present, the institute has operated on the legal basis of a limited liability company (GmbH) and its budget is equally covered by the Steel Institute (VDEh) and the Max Planck Society. Nearly 100 scientists, technicians and administrative staff are employed at the institute, and an additional 90 scientists are financially supported through third party funds and stipends.

Since 2000, the institute is again under reconstruction which will result in nearly 7500 m^2 of new laboratories and offices. The first new laboratories were opened in 2002 and the renovations are expected to be completed in the beginning of 2005.

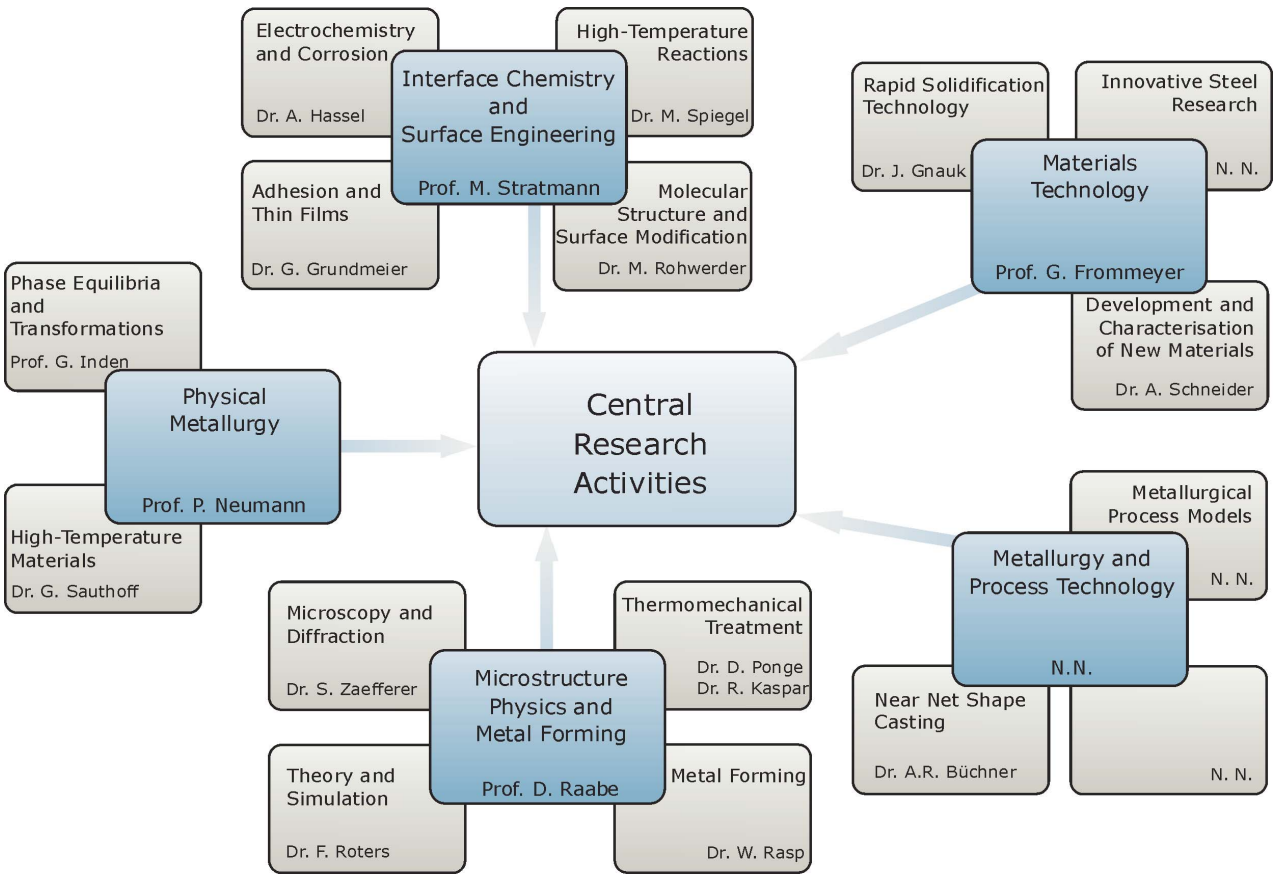


Scientific Programme

Scientific Organization

The institute devotes its research to iron, steel and related materials. In addition to the development of new materials, the institute focuses on the physical and chemical processes and reactions which are of importance for materials production, processing, materials characterisation and properties. The institute is organized into five departments (alphabetical order):

- *Interface Chemistry and Surface Engineering* (Stratmann): aspects of environmentally accelerated degradation of surfaces and interfaces like corrosion and deadhesion and the engineering of new and stable surfaces and interfaces
- *Materials Technology* (Frommeyer): novel lightweight steels and rapid solidification processes
- *Metallurgy and Process Technology* (N.N.): modeling of metallurgical processes and near net shape casting
- *Microstructure Physics and Metal Forming* (Raabe): mathematical modeling of microstructures and properties during processing and their experimental investigation using microscopy and diffraction methods
- *Physical Metallurgy* (Neumann): phase equilibria, phase transformations in multicomponent systems and high-temperature materials



Each department is broken down into research groups which are typically managed by group leaders. Each research group has its own specific focus and research activities. However, the groups are complementary within the departments, allowing for a homogeneous departmental profile. Part II of this status report summarizes the activities within the departments and provides an overview of the results obtained during the last two years.

In addition to departmental research, certain research activities are of common interest within the institute. These research areas are highly interdisciplinary and combine the experimental and theoretical expertise available from within the different departments in order to achieve a scientific and technological breakthrough in highly competitive areas of research. Such areas include:

- New steels
- Iron aluminium based materials
- Functional layers
- Surface mechanics and adhesion
- Microstructure and reactivity
- Numerical modelling

The research within the institute is therefore organized vertically in highly specialized departments and research groups and horizontally in central research activities. We believe that this form of organization encourages a high level of individual scientific work within the departmental framework of research groups as well as the development of new materials with complex properties combining e.g. high mechanical strength with high surface functionality. In a typical university setting, research activities such as metallurgy or surface science are carried out in different university departments. In contrast, these research activities are linked through the institute's research structure leading to a more efficient use of the scientific equipment and a homogeneous research profile.

Service groups provide the scientific departments with valuable experimental expertise. These services include the production of materials, chemical analysis of metallic substrates, electron microscopy, metallography, a workshop equipped for the handling of unusually hard and brittle materials, facilities to build scientific equipment and a computer department.

Central Research Activities

New Steels

The development and characterisation of new steels is one of the major fields of activity of the institute. Four departments are involved in this field. The topics are: new steels with improved mechanical properties (strength, ductility, fracture toughness, creep resistance), impact resistance, and leaner chemical compositions with better recyclability. In addition, steels with very fine-grained and stable microstructures, which can be formed superplastically at high strain rates, exhibit a high potential for applications. Besides the bulk properties the surface properties of new steels are also of high importance in particular with respect to improved corrosion resistance and other functional characteristics.

The improvement of the creep resistance of new martensitic/ferritic 12% Cr steels in power plants through the precipitation of the WFe_2 Laves phase as well as carbonitrides is a major task, which is under investigation in the Physical Metallurgy Department by the High Temperature Materials Group (*Sauthoff, Knežević*). The respective alloy design is strongly supported by theoretical simulations of the Phase Equilibria and Transformation Group (*Inden*).

The Thermomechanical Processing Group (*Ponge*) in the Microstructure Physics and Metal Forming Department is designing innovative concepts of thermomechanical processing and employing these to achieve ultra fine-grained (UFG) microstructures in C-Mn steels with 0.15 to 0.3 %C. Another innovative steel project aims at fine-grained materials produced by recrystallisation (*Storojeva, Song, Kaspar, Ponge*). A third project is focused on high-strength long products with improved toughness and fatigue properties (*Detroy, Kaspar, Ponge*).

The Innovative Steel Research Group of the Materials Technology Department is developing new classes of innovative high manganese or cobalt content TRIP and TWIP steels with superior mechanical properties with special regard to deep drawing and high impact resistance (*Frommeyer, Grässel*). Newly developed high-strength aluminium steels with about 6.5 mass % Al exhibit a reduction

in specific weight of about 10 % and are studied with respect to applications as light-weight materials (*Brüx, Frommeyer*). In view of applications of duplex and super duplex stainless steels in the chemical and offshore industry, the high-temperature deformation behaviour of the modified micro duplex stainless steel IN 744 is under investigation. Ultra-high-strength microcrystalline dual-phase steels with high carbon and aluminum contents are studied with a focus on the superplastic behaviour at high strain rates. Newly developed quasi-eutectoid high-carbon steels with chromium and aluminium additions are subject of an investigation on the deformation and strengthening mechanisms in view of developing a quantitative model (*Frommeyer, Brokmeier*).

Beside the bulk properties of new steels, the surface properties deserve great attention. In particular, the high amount of residual inert oxides on the surface after reductive treatments is a problem of high technological importance and has been chosen as one of the focal points for research in the Interface Chemistry and Surface Engineering Department. An aim of the Molecular Structure Group is to understand the wetting behaviour of liquid zinc on partially oxidised surfaces (*Rohwerder*). Novel high-performance thermal pre-treatments are developed by the High Temperature Reaction Group (*Spiegel*). The Corrosion and Electrochemistry Group studies the corrosion properties and mechanisms of various steels (*Hassel*).

Iron Aluminium Based Materials

In 2000 a major research activity on iron aluminium alloys and iron aluminides was instigated in order to perform fundamental studies and to exploit the potential of these materials. Four departments of the institute are involved in this joint project: Materials Technology (*Frommeyer, Schneider* (coordinator)), Physical Metallurgy (*Inden, Palm, Sauthoff, Stein*), Interface Chemistry and Surface Engineering (*Hassel, Spiegel*), and Microstructure Physics and Metal Forming (*Zaefferer, Raabe*).

Iron aluminium based alloys offer a considerable potential for the development of corrosion resistant materials for applications at elevated temperatures with reduced density compared to steels. In order to select appropriate alloy systems, the phase equilibria and phase transformations (*Inden, Palm, Sauthoff, Schneider, Stein*) of various Fe-Al-X(-Y-Z) systems are under investigation. Based on these studies, materials with specific combinations of phases and microstructures are produced. Microstructural characterisation of the iron aluminides Fe_3Al and FeAl using high resolution microscopy such as TEM and atom probe field-ion microscopy (APFIM) is one task in this research (*Frommeyer, Schneider*). Investigations on the temperature dependent mechanical properties, such as yield strength, creep resistance and superplasticity were performed and from these data the underlying fundamental mechanisms, i.e. creep laws, have been elucidated (*Frommeyer, Palm, Sauthoff*). The understanding of the mechanical behaviour serves as basic information for establishing modified production routes for iron aluminides. The basic principles of texture evolution during deformation processing and recrystallisation will be investigated in the department of Microstructure Physics and Metal Forming (*Zaefferer, Raabe*). As iron aluminium based materials show great potential for service in corrosive environments, the fundamental mechanisms of oxidation (*Spiegel, Stein*), salt melt induced corrosion (*Spiegel*) and carburisation (*Schneider, Spiegel*) have to be understood. For an increased corrosion resistance, a pre-treatment by electrochemical methods may offer considerable advantages (*Hassel, Spiegel*).

Objectives and first results of this joint project have been presented and discussed with scientists from research institutes and industry at the 11th annual meeting 2002 of the Intermetallics Committee of the DGM.

Functional Layers

Another thematic priority of the institute are functional layers on steels and related substrates. For the 6th framework programme of the EU, an integrated project “Knowledge-Based Multifunctional Surface Layers for Mass Produced Materials“ (Coordinators: *Spiegel, Stratmann*) is planned. Surface engineering is one of the key future technologies in Europe and applications cover fields such as medical implants, biosensors, microelectronics, fuel cells and surface tailoring of mass-produced structural materials. While surface engineering of mass-produced sheet metals is extremely important for the development of a multitude of products, until now most advanced surface technology worldwide has barely been implemented into production lines. It is thus the aim of this integrated project to strengthen the leading role of the European industry in the fabrication and the use of mass-produced structural materials and to provide the scientific basis for new surface layer developments. The departments involved in this project are Interface Chemistry and Surface Engineering (*Stratmann*), Microstructure Physics and Metal Forming (*Raabe*) and Materials Technology (*Frommeyer*).

Surface Mechanics and Adhesion

Surface mechanics and adhesion is a central research activity that considers the mechanical properties and forming behaviour of coatings on metals. The expertise of the Department of Microstructure Physics and Metal Forming (*Raabe*) in the understanding of forming operations, the mechanical properties of metal surfaces and the simulation of metal surfaces during forming is combined with the expertise in surface tailoring of metals of the department of Interface Chemistry and Surface Engineering (*Stratmann*). Within the Adhesion and Thin Films research group (*Grundmeier*), thin functional plasma polymers and adhesion-promoting model films with a thickness below 100 nanometers are synthesised and characterised with regard to their morphology and chemical composition. Moreover, wear resistant films with a thickness of up to one micrometer are developed. The mechanical properties and adhesion of these films are then studied by means of the nanoindentation technique. Simulation of the experimental data is done in close collaboration with Prof. Raabe's department. The nanoindentation of metal surfaces and metal coatings is simulated by use of crystal plasticity FEM (Finite Element Method), which is implemented into the commercial FEM software (MSC.Marc) by means of user subroutines. In this way, the full crystalline anisotropy (elastic and plastic) of the material and its development (e.g. orientation change of the crystal) during the test are taken into account. For polymer coatings and their adhesion behaviour, suitable material laws have to be evaluated.

Moreover, the formability of surface layers is of common interest. Within the department of Prof. Stratmann, the focus is on the formation and reactivity of defects in thin interfacial films (*Rohwerder*) and on interfacial crack formations and disbanding during forming (*Grundmeier*)). The complimentary activities in the department of Microstructure Physics and Metal Forming will be on the simulation of the topologic development of the metallic surface (change of roughness etc.) during the forming process. Crystal plasticity FEM will be used to first simulate the uncoated material to be followed by a study on the influence of the coating on the surface development.

Microstructure and Reactivity

In 2002 a new joint research activity on the relationship between microstructure and reactivity was established. Three groups from two departments of the institute are presently involved in this activity: Interface Chemistry and Surface Engineering (*Hassel* (coordinator), *Stratmann*), Microstructure Physics and Metal Forming (*Ponge, Zaefferer, Raabe*).

The correlation between microstructure and reactivity has gained more and more attention due to the progress in experimental techniques able to assign local reactivity differences to structural properties of the material. The reactivity of grains with different crystallographic orientation can vary dramatically. On titanium, for example, oxygen evolution is observed only on grains with a specific orientation while others passivate and block this reaction. Chemical compositions can also change if, e.g. alloying elements are enriched in grain boundaries.

Microelectrochemical methods such as the Scanning Droplet Cell (SDC), Scanning Kelvin Probe (SKP) and Scanning Electrochemical Microscopy (SECM) are employed to investigate the reactivity of samples in the Department of Interface Chemistry and Surface Engineering (*Hassel, Stratmann*). These results will be linked to the microstructure as determined by Electron Backscatter Diffraction (EBSD) (*Zaefferer, Raabe*) in the Microstructure Physics and Metal Forming Department. To gain an understanding of the modification of the microstructure ranging from coarse grain to ultra-fine grain material is fundamental and is also a subject investigated in the Microstructure Physics and Metal Forming department (*Ponge, Raabe*). Obviously, a mechanical modification or optimisation must take the changes in reactivity, namely the corrosion stability, into account.

Numerical Modelling

In view of a number of projects already running in the field of process modelling, the simulation of microstructure formation becomes more and more important. The microstructure, in fact, is the result of the process steps applied. The departments of the institute have already started to put considerable effort into microstructure simulation; until now with an emphasis on the individual perspectives of the respective working fields.

At present, phase equilibria in multi-component systems are calculated based on the so called CALPHAD approach (*Inden, Schneider*). The numerical treatment of diffusion controlled phase transformations in multi-component systems is performed using the DICTRA software, which was developed in cooperation between the MPIE (*Inden*) and the KTH Stockholm (*Ågren*). The treatment is based (a) on the concept of sharp interfaces, (b) on local equilibrium at moving interfaces, (c) on thermodynamic driving forces obtained from the CALPHAD approach for computational thermodynamics, (d) on mobilities rather than diffusivities. It embraces isothermal, non-isothermal conditions and various geometries. In order to treat microstructure formation and the competition between growing phases, space may be subdivided into cells which are coupled by appropriate boundary conditions.

Based on this treatment for diffusion controlled solid-solid phase transformations, a new concept is being developed for solid-gas and solid-liquid corrosion reactions (*Spiegel, Stratmann*).

The calculation of phase formation during solidification, i.e. under non-equilibrium conditions, so far uses a simplified approach in terms of single domain methods such as the generalized enthalpy method for heat and mass transport (*Gnauk, Frommeyer*). There exist more sophisticated treatments such as the phase field method which aims at simulating morphological evolutions, instabilities and complex solidification processes (multi-phase simulation) on mesoscopic and macroscopic scales.

In the department of Prof. Raabe, the group of Dr. *Roters* is developing a Texture Component Crystal Plasticity FEM to introduce texture and its evolution into simulations of the forming processes. These simulations require starting microstructures which have been taken from experiments. Heat treatments are being simulated using the cellular automaton method (recrystallisation) and the Monte Carlo method (grain growth). The starting microstructures are again determined experimentally or taken from previous simulations of crystal plasticity and metal forming. So far, phase transformations are not taken into account in these simulations.

It is planned to establish a theoretical group based on a C3 position dealing with molecular modelling of metal polymer interfaces. Modelling is envisaged which will be based on molecular dynamics and density function theories. The aim is to theoretically understand the ordering of organic molecules at the interface, the chemical interaction between functional groups of the polymer chain and the surface and the incorporation of small molecules and ions into the interfacial region. The C3 group will be associated with the Department of Interface Chemistry and Surface Engineering and will strongly interact with existing experimental groups such as the research group on Adhesion and Thin Films and the research group on Molecular Structures and Surface Modification. Furthermore, a close collaboration with the theoretical groups in the Department of Microstructure Physics and Metal Forming is anticipated. Thereby, the C3 group on „Molecular modelling of metal/polymer interfaces“ will build a bridge between the mesoscopic modelling approaches and quantum-mechanical treatments.

It is a key issue of future work in the institute to combine the efforts at the different departments in order to make one step further towards a global monitoring of all process steps in microstructural evolution, from solidification, heat treatments and forming to degradation under service conditions. For this purpose, the expertise of Prof. Inden's group in the field of phase equilibria and transformations has to be linked to the simulations of microstructure formation and evolution during casting in the department of Prof. Frommeyer. These simulated microstructures could then be introduced as starting microstructures into the simulations of forming and heat treatment in the department of Prof. Raabe. Degradation processes under service conditions shall be treated using the approaches developed by Dr. Spiegel.

Scientists at the Institute

(Departments in alphabetical order)

Interface Chemistry and Surface Engineering

Stratmann, Martin, Prof. Dr.rer.nat. (*head of department*)

Akiyama, Eiji, Dr. (Japan), guest , since 01.04.02

Barranco, Violetta, Dr.-Ing., since 01.03.2002

Baumert, Birgit, Dipl.-Phys.

Bonk, Stephan, Dipl.-Phys.

Cha, Sung-Chul, Dr.-Ing. (Korea), since 01.08.2002

Dornbusch, Michael, Dr.rer.nat., since 01.10.2001

Ehahoun, Hervé, Dr.rer.nat. (France)

Frenznick, Sascha, Dipl.-Ing., since 01.05.2001

Fushimi, Koji, Dr.(Japan), since 01.05.2002

Grundmeier, Guido, Dr.-Ing. (*group leader*), since 01.07.2001

Hassel, Achim W., Dr.rer.nat. (*group leader*)

Hausbrand, René, Dipl.-Phys., until 31.10.2002

Hornung, Elke, Dipl.-Ing., until 31.01.2002

Hüning, Boris, Dipl.-Phys.

Li, Yuanshi, Dr. (China), since 01.10.2001

Lill, Kirsten, Dipl.-Chem., since 01.12.2002

Michalik, Adam, Dipl.-Ing. (Poland)

Moszynski, Dariusz, Dr. (Poland), until 30.06.2001

Ogura, Yuzuru, Dr. (Japan), guest, until 30.09.2001

Paliwoda, Grazyna, Dipl.-Chem. (Poland), since 15.10.2001

Parezanovic, Ivana, Master of Science (Yugoslavia)

Piehl, Carmen, Dipl.-Chem., until 31.03.2001

Raacke, Jens, Dr.rer.nat., since 01.10.2002

Rohwerder, Michael, Dr.rer.nat. (*group leader*)

Roßenbeck, Britta, Dr., since 01.07.2001 (guest since 01.04.2002)

Ruh, Andreas-Christian, Dipl.-Min., since 01.02.2002

Sämann, Nicole, Dipl.-Chem., until 14.04.2002

Sanchez Pastén, Miguel, Dipl.-Chem. (Mexico)

Sauerhammer, Björn, Dr.rer.nat., since 01.11.2002

Shirtcliffe, Neil, Dr. phil. (UK), until 31.12.2001

Spiegel, Michael, Priv.-Doz. Dr.rer.nat. (*group leader*)

Tang, Shao-Lin, Dr.-Ing. (China), guest, 01.07.-14.11.2002

Tareelap, Napachat, Dipl.-Ing. (Thailand)

Tsuri, Shiro, Dipl.-Chem. (Japan), guest, until 31.01.2002

Vander Kloet, Jana, Dipl.-Phys. (Canada and The Netherlands)

Viefhaus, Helmut, Dr.rer.nat., until 31.05.2002

Wapner, Kristof, Dipl.-Chem., since 01.06.2001

Wicinski, Mariusz, Dipl.-Chem. (Poland)

Yang, Lihong, Dr. (China), since 01.20.2002

Yu, Xingwen, Dr. (China), since 01.01.02

Visiting Scientist:
Prof. Dr. Bharat Bhushan, Ohio Eminent Scholar, Department of Mechanical Engineering, Ohio State University (USA), 07.01.-13.02.2002

Materials Technology

Frommeyer, Georg, Prof. Dr.-Ing. (*head of department*)

Amichi, Rachida, Dr. (Algeria), 21.07.-20.08.2002

Brokmeier, Klaus, Dipl.-Ing., since 01.05.2001

Brüx, Udo, Dipl.-Phys.

Deges, Johannes, Dipl.-Phys., since 01.03.2002

Falat, Ladislav, Dipl.-Ing. (Slovakia), since 01.10.2001

Fischer, Rainer, Dipl.-Phys.

Gnauk, Joachim, Dr.rer.nat. (*group leader*), since 01.09.2001

Jiménez, José, Dr. (Spain), guest, 01.-31.07.2001, 01.-31.07.2002

Knippscheer, Sven, Dipl.-Ing.

Rablbauer, Ralf, Dipl.-Phys.

Sandmann, Annika, Master of Science (Sweden), since 09.09.2002

Schneider, André, Dr.rer.nat. (*group leader*)

Wenke, Rainer, Dipl.-Phys., since 01.10.2002

Visiting Scientist:
Prof. Dr. James Wittig, Department of Electrical Engineering and Computer Science, Vanderbilt University School of Engineering (USA), 01.-30.06.2001, 15.07.2002- 14.08.2002

Metallurgy and Process Technology

Büchner, Achim R., Dr.rer.nat. (*provisional head of department*)

Gnauk, Joachim, Dipl.-Phys., guest, until 31.03.2001

Heckmann, Carl Justus, Dipl.-Ing., guest, until 31.03.2001

König, Markus, Dipl.-Ing., until 30.06.2002

Pötschke, Stephan, Dipl.-Ing., since 01.09.2001

Thiemann, Michael, Dipl.-Phys.

Microstructure Physics and Metal Forming

Raabe, Dierk, Prof. Dr.-Ing. habil. (*head of department*)

Ardehali Barani, Araz, Dipl.-Ing. (Germany and Iran), since 01.09.02

Chen, Nan, Dr. (China), since 13.06.2001

Detroy, Sandra, Dipl.-Ing., since 01.09.2002

Elsner, Alexander, Dipl.-Ing. FH

Filatov, Dmitri, Dipl.-Ing. (Russia)

Jamet, Jean-Marcel, Dr. (France), until 31.05.2001

Kaspar, Radko, Dr.-Ing., until 31.10.2001

Klüber, Christian, Dipl.-Ing.

Kobayashi, Satoru, Dr. (Japan), since 11.11.2002

Kuo, Jui-Chao, Dipl.-Ing. (Taiwan)

Lücken, Hermann, Dipl.-Ing.

Ma, Anxin, Master of Science (China), since 01.06.2002

Ponge, Dirk, Dr.-Ing. (*group leader*), since 01.07.2001

Rasp, Wolfgang, Dr.-Ing. (*group leader*)

Roters, Franz, Dr.rer.nat. (*group leader*)

Sachtleber, Michael, Dipl.-Ing., since 01.02.2001

Scheele, Georg, Dipl.-Ing.

Song, Rongjie, Master if Science (China), since 01.09.2001

Storojeva, Lidia, Dr.-Ing. (Russia), since 01.07.2001

Thomas, Ingo, Dipl.-Phys., guest, since 01.08.2001

Vanik, Hans-Georg, Dipl.-Ing., until 15.02.2001

Wang, Yanwen, Dr. (China)

Wengenroth, Walter, Dipl.-Phys., until 15.01.2001

Wichern, Christan, Dr. (USA)

Yusupov, Artem, Dipl.-Ing. (Russia), since 01.06.2001

Zaefferer, Stefan, Dr.-Ing. (*group leader*)

Zhao, Zisu, Dr. (China)

Visiting Scientists:
Prof. Dr. Jingtao Han, Department of Materials Science and Engineering, University of Science and Technology Beijing (China), 08.01-31.03.2002

Prof. Dr. Weimin Mao, Department of Materials Science and Engineering, University of Science and Technology Beijing (China), 01.06.-31.07.2001, 25.06.-31.07.2002

Physical Metallurgy

Neumann, Peter, Prof. Dr.rer.nat. (*head of department*)

Balun, Jozef, Dipl.-Ing. (Slovakia)

Bernst, Reinhard, Dipl.-Phys.

Duchet, Roland, Dipl.-Ing. (France), 12.02.-15.03.2001

Inden, Gerhard, Prof. Dr.rer.nat. (*group leader*)

Jiang, Dongtao, Dr. (China), 01.03.2001-28.02.2002

Knežević, Vida, Master of Science (Yugoslavia)

Löffler, Falk, Dipl.-Ing., until 23.03.2001

Palm, Martin, Dr.rer.nat.

Risanti, Doty-Dewi, Master of Science (Indonesia), since 13.08.02

Sauthoff, Gerhard, Priv.-Doz. Dr.rer.nat. (*group leader*)

Stallybrass, Charles, Dipl.-Ing. (UK), since 01.06.2002

Stein, Frank, Dr.rer.nat.

Vilk, Julius, Dipl.-Ing. (Slovakia), until 21.06.2002

Zhang, Jianqiang, Dr. (China)

Kemnitz, Hans-Dieter, Dr.-Ing. (*head of electronics and computer services*)

Gahn, Ulrich, Dr.rer.nat.

Research in Progress

Interface Chemistry and Surface Engineering: M. Stratmann

Adhesion and Thin Films

Wapner, Grundmeier: Long term stability of high performance structural adhesives

Dornbusch, Grundmeier: New thin conversion coatings on automotive metal sheets

Roßenbeck, Grundmeier: Corrosion mechanisms of thin latex films on steel

Thiemann, Carpentier, Barranco, Grundmeier: Tailored thin plasma polymers for surface engineering of galvanised steel

Yang, Grundmeier: Nanomechanics and adhesion of thin functional plasma polymer films

Raacke, Grundmeier: Pulsed atmospheric plasmas for surface tailoring of polymers and metals

Molecular Structure and Surface Modification

Baumert, Rohwerder: Investigation of defect formation and behaviour in plasmapolymer films under tensile loading

Michalik, Rohwerder: Potential of conducting polymers for corrosion protection

PaliwodaRohwerder: Release systems for self-healing polymer/metal interfaces

Frenznick, Rohwerder: Wetting behaviour of A/B-patterned surfaces: fundamental aspects of hot dip galvanizing of high strength steels

Hausbrand, Rohwerder: Structure of corrosion protective coatings: the Mg-effect in Mg/Zn-alloys

Ehahoun, Rohwerder: Fundamental investigations of the origin of the Volta potential measured on ordered molecular films

Mueller-Lorenz, Rohwerder: Soluble salt contamination on blast cleaned steel surfaces and the effect on the durability of subsequently applied paint systems

Yu, Rohwerder: Nanoscopic Mechanisms of cathodic delamination

Vander Kloet, Rohwerder: SKPFM investigation of filiform corrosion

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Wicinski, Bonk, Hassel: Passive/active transitions in cyclic corrosion tests

Tareelap, Hassel: In-situ microindentation of passive metals in a scanning droplet cell

Akiyama, Hassel: Detection of discrete and single impacts in particle induced flow corrosion

Fushimi, Hassel: Electrochemical micromachining of Nitinol based shape memory alloys

Mingers, Hassel: Microbiologically influenced corrosion of iron by sulfate reducing bacteria

Lill, Hassel: Corrosion and corrosion protection of TRIP/TWIP steels

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Parezanovic, Spiegel: A mechanistic study of wetting and dewetting during hot dip galvanizing of high strength steels

Sanchez, Spiegel: NaCl Thermogravimetric and electrochemical investigations on the corrosion of Fe, Ni and Cr beneath solid and molten KCl-NaCl

Li, Spiegel: Surface modification for corrosion protection on vapour deposited Zn-Mg alloy coatings of steel sheets

Li, Spiegel: High temperature corrosion of Al-containing alloys and intermetallics beneath molten KCl-$ZnCl_2$

Strauch, Parezanovic, Spiegel: Stainless steels for molten carbonate fuel cells

Vogel, Leitenberger, Spiegel: In-situ reflectometry measurements on growing oxide scales with synchrotron radiation

Ruh, Spiegel: Optimization of in-service performance of boiler steels by modelling high temperature corrosion

Cha, Spiegel: Mitigation of formation of chlorine rich deposits affecting superheater corrosion under co-combustion conditions

Sauerhammer, Spiegel: HDG behaviour of TRIP/TWIP steels

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Rapid Solidification Technology

Frommeyer, Gnauk: Continuous casting of CrNi stainless steel and heat resistant $Ni_3Al(B)$ and Ni(Fe)Cr wires. Investigations on correlations between microstructures and solidification and cooling rates

Frommeyer, Gnauk: Thin strip and foil casting of heat resistant FeCr(Al) and $Fe_3Al(Cr)$ alloys for catalytic converter systems

Frommeyer: Continuous strip casting of Fe-Al-foam-semi-finished products with optimized pore structures

Gnauk, Frommeyer: Numerical modelling of solidification processes

Gnauk, Wenke: Modelling of phase and microstructure formation under non-equilibrium conditions of aluminium titanium laser welded seams

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Frommeyer, Brüx, Brokmeier: Development of high strength austenitic and duplex lightweight steels based on Fe-Mn-Al(C) and characterization of the mechanical properties

Frommeyer: Investigations on structural superplasticity and superplastic forming at high-strain-rates of high aluminum content UHC-steels and duplex stainless steels

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Brüx, Frommeyer: Characterization of the mechanical properties of high strength and supraductile TRIP/TWIP-steels tested at very high strain rates and correlation with the deformation mechanisms

Brüx, Frommeyer: TEM investigations on deformation mechanisms of lightweight steels based on iron-aluminum

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Knippscheer, Frommeyer: Characterization of microstructures and mechanical properties of TiAl-based alloys modified with alloying elements of the transition metals

Knippscheer, Frommeyer, Wittig: Influence of alloy compositions and microstructures on processing procedures and applicability of TiAl(Cr, Mo, Cu, Si) base alloys

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Schneider, Frommeyer: TEM-investigations on antiphase boundaries and dislocation structures of ordered Fe_3Al based alloys

Fischer, Frommeyer, Deges, Schneider: Structural characterization of high temperature NiAl-(Fe, Cu, Mo) alloys by using Atom Probe Field Ion Microscopy

Deges, Schneider, Fischer, Frommeyer: APFIM investigations on Fe-Al based alloys

Schneider, Falat, Frommeyer: Thermodynamics and kinetics of phase transformations and coarsening processes in Fe-Al-M-C(M=Ti, Nb, V, Ta) alloys

Schneider, Frommeyer, Ishida: Constitution investigations on metastabile equilibria in iron rich ternary Fe-Al alloys

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Thiemann, Büchner: Heat transfer coefficient between material and rolls in twin roll strip casting

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Büchner: Thin strip casting of special steels for industrial application

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Raabe, Chen, Zaefferer, Günther: Goss grain growth in silicon steels

Raabe, Thomas, Zaefferer, Friedel: Recrystallization mechanisms in IF steels

Zaefferer, Hirano: Recrystallization mechanisms in Ni_3Al alloys

Zaefferer, Glatzel: Microstructure and texture of coatings on Ni-based superalloys

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Zaefferer: Nucleation mechanisms of recrystallization in ECAE-deformed aluminium

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Ponge, Kaspar, Rasp, Elsner: Laboratory tests on optimizing process parameter of ferritic rolling deep drawable steels

Ponge, Kaspar, Barani: Effect of tramp elements in high strength SiCr spring steels

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Raabe, Rasp, Scheele: Foil rolling

Rasp, Wichern: Tribology tests for the characterization of cold-rolling oils.

Rasp, Filatov: Influence of deformation parameters in hot rolling on scale formation and ability for pickling.

Rasp, Yusupov: Advanced modelling of lateral flow and residual stresses in flat rolling.

Rasp, NN: Improvement of formability by superposition of hydrostatic pressure.

Physical Metallurgy: P. Neumann

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Sauthoff: Creep of multiphase alloys

Sauthoff, Risanti: Deformation behaviour of Fe-Al-Ta alloys

Sauthoff, Stallybrass: Ferritic Fe-NiAl superalloys

Sauthoff, Palm: Deformation behaviour of intermetallic NiAl-Ta-Cr alloys with strengthening phases

Sauthoff, Palm, Stein: Screening of Al-rich intermetallics

Palm, Sauthoff, Stein: Phase equilibria, deformataion behaviour and oxidation resistance of Fe-rich Fe-Al-X systems (X= Nb, Mo, Zr,) alloys

Sauthoff, Knežević: Ferritic steels with maximum creep resistance: alloy development and long-term behaviour

Stein, Sauthoff: DTA studies of phase transitions in intermetallic systems

Palm, Stein, Sauthoff, Jiang: Phase equilibria in the Co-Nb and Co-Nb-Al systems

Palm, Sauthoff: Mechanical properties of single-phase and two-phase $L2_1$ Fe-Al-Ti-based alloys

Palm, Stein: Stability of the microstructure, mechanical behaviour and oxidation resistance of lamellar TiAl + Al_2Ti alloys

Stein, Palm, Viguier, Lacaze (CIRIMAT, Toulouse): Determination of the high-temperature phase equilibria and the liquidus in the Fe-Al-Ti system by DTA

Stein, Palm, Sauthoff, Kreiner (MPI CPfS Dresden), Grin (MPI CPfS Dresden): Experimental and theoretical investigations on the structure and stability of Laves phases in the binary transition metal systems Nb-X (X=Cr, Mn, Fe, Co)

Phase Equilibria and Transformations

Bréchet, Hutchinson (LTPCM-Grenoble), *Inden:* Computer supported modelling of steel transformations

Unucka, Kroupa (Univ. Brno), *Inden:* Development of a thermodynamic database for ferritic steels

Mola, Pohl (Univ. Bochum), *Inden*: Numerical modelling of duplex steels

Inden: Diffusion in multicomponent systems and complex phases

Eleno, Schön (Univ. São Paulo), *Inden:* CVM and Monte Carlo simulations of atomic/magnetic ordering and phase separation in multicomponent alloy systems

Balun, Inden: Experimental determination of phase equilibria and thermodynamic analysis of the systems Fe-Ni-Si and Fe-Rh-X (X=Co,Ti)

Bernst, Inden: Effect of alloying elements on the kinetics of carbide formation

Joint Projects

Balun, Inden, Schneider: Ferritic steels with maximum creep resistance: thermodynamics and kinetics of precipitation reactions

Falat, Schneider, Frommeyer, Sauthoff: Fe-Al-Nb/Ta/Ti-C/B- alloys for applications in chemical processing

Frommeyer, Büchner: Microstructural characterization of high silicon content transformer steels produced by strip casting

Hassel, Schneider: Nanoelectrode arrays by self organisiation during directed solidification of ternary alloys

Rasp, Filatov, Spiegel: Oxide formation during hot rolling

Rohwerder, Spiegel, Frommeyer, Frenznick, Parezanovic: A mechanistic study of wetting and dewetting during hot strip galvanizing of high strength steels

Rohwerder, Baumert, Zaefferer: Investigation of defect formation and behaviour in plasmapolymer films under tensile loading

Schneider, Frommeyer, Sauthoff: Investigation of the mechanical properties and the coarsening behaviour of Fe_3Al-C alloys with the strengthening κ carbide phase

Schneider, Spiegel: Metal dusting of Fe-Al

Schneider, Zaefferer, Frommeyer, Raabe: Optimization of texture and microstructure of Fe_3Al-based alloys

Spiegel, Sauthoff: Fe-Cr-Si alloys as coatings for superheaters

Spiegel, Stein: Initial stages and kinetics of oxidation of binary and ternary iron-aluminides

Stein, Schneider, Frommeyer: Investigation of the effect of X = Ti, V, Cr, Nb, Mo on the structure and mechanical properties of Fe_3Al-X alloys

Zhang, Bernst, Schneider, Inden: Decomposition of cementite during metal dusting

Zhang, Schneider, Inden: Analysis of carbon composition profiles in cementite and ferrite

Zhang, Schneider, Inden: Thermodynamics of diffusion controlled reactions during gas carburisation

Central Facilities

Materials Preparation

Research and development of new materials and processes necessitate the capability to produce materials according to particular specifications. The MPIE has specific facilities for the production and refinement of materials according to the scientific and technological needs of the research groups within the institute.

Alloy preparation: The three departments Metallurgy, Physical Metallurgy and Materials Technology have a variety of sophisticated technical equipment to prepare special single and polycrystalline alloy samples.

Several induction furnaces are available to produce different quantities of materials. Small amounts (about 350 - 850 g) of special alloys with melting temperatures up to about 1850 °C are routinely prepared by induction melting in ceramic crucibles. This can be carried out either in a vacuum ($2 \cdot 10^{-5}$ mbar) or in the presence of an inert gas to obtain cylindrical ingots with 20 - 40 mm in diameter and up to 200 mm in length. Two vacuum induction furnaces with a basic pressure of 10^{-7} mbar are available for producing medium amounts of pure materials and alloys (ingots of up to 7 kg). Larger amounts of metallic melts can be produced in a vacuum or a gas atmosphere in two larger furnaces (maximum capacity of 80 kg), coupled with a facility for pilot test sampling and equipped with a 100 kW medium frequency generator. A typical application is the production of iron-based alloys with melting temperatures up to 1800 °C.

Non-metallic, non-inductive coupling materials can be processed under an inert gas atmosphere in electrical resistance carbon furnaces (Tammann furnaces) with a maximum operation temperature of 3000 °C.

A high pressure induction furnace with a capacity of 10 kg is capable of a maximum pressure of 100 bar operating with a power supply of 50 kW. With this furnace, it is possible to process melts with a higher partial vapour pressure, e.g. manganese or rare-earth metals, while avoiding the formation of volatile vapours.

Two different facilities are available for melting highly reactive materials: an electric arc furnace with a 300 g capacity, equipped with a tilting mould and an induction levitation melting unit which is used for the preparation of small amounts (350 g maximum) of polycrystalline high purity alloys with high oxygen, nitrogen or hydrogen affinity. Melting temperatures up to about 1900 °C can be achieved with solidification in cold copper moulds under vacuum or inert gas as well as for the growth of long single crystals with continuous feeding stock.

Using a modified Bridgman technique, single crystals with a diameter of up to 40 mm and 130 mm in length can be prepared from various alloys with melting temperatures of up to 1700 °C in ceramic crucibles under vacuum or in an inert gas atmosphere. The crystal orientation is determined by laser light reflection from the etched crystal surface. Operating at temperatures of up to 2000 °C, another Bridgman furnace of similar geometry is used for directional solidification. Single crystal growth and high purification of metals using the method of zone refining can be performed in a 75 kW electron beam melting furnace. The molten zone is produced by a circular tungsten cathode, which is moved vertically along the sample.

Two units for rapidly solidified materials are also available in addition to the Bridgman type furnaces, which are specifically designed for controlled slow cooling processes. These are a melt drop furnace and a splat-cooling facility, operating with a modified levitation melting unit.

Near net shape casting: The two thin strip casters for direct strip casting related to Bessemer's process are unique to our institute. The as cast materials can include peritectic or other low carbon steels as well as stainless steels and non-ferrous metals. One caster is equipped with a 150 kW induction furnace with a maximum crucible capacity of 200 kg. The produced steel strips have a thickness of 1 to 4 mm and a strip width of 120 mm. The other caster is slightly smaller (30 kW furnace, maximum capacity 10 kg, strip width of 65 mm). It has the unique feature of a full housing, i.e. the strip casting process can be carried out in an inert gas atmosphere.

The institute is also equipped with a shape flow casting (SFC) and a planar flow casting (meltspin) facility for the fabrication of metallic fibres, wires and ribbons or foils. With the planar flow casting meltspinning equipment, it is possible to produce rapidly solidified ribbons with maximum cooling rates of 10^6 K/s by ejecting the melt stream onto an actively cooled rotating wheel surface. The resulting ribbons

have a variable thickness between 50 μm to 250 μm and are 30 mm in width. The SFC facility directly produces wires with diameters ranging from 1 to 3 mm by casting the melt into the guiding groove of a horizontally rotating wheel. The shape flow caster can also be used as in-rotating-liquid-spinning (INROLISP) device, where the melt is injected into a liquid cooling medium (water or other coolants), which produces fibres with a 80-400 μm diameter. All these products possess the microstructural features and properties commonly obtained by rapid solidification, e.g. fine grained microstructure and little (or no) segregation in combination with final or near net shape geometries.

Thermal treatment: In addition to the casting facilities mentioned above, several possibilities to alter the microstructure and phase distribution following the casting process are available. Several annealing furnaces with various geometries exist that are capable of temperatures up to 1700 °C, which can be used in air, a protective atmosphere or vacuum.

Mechanical alloying: Powder metallurgical processing such as mechanical alloying can be carried out in inert gas atmosphere using a planetary ball mill (500 ml capacity), filled with hardened and heat resistance steel or cemented carbide balls (5-11 mm diameter). Larger amounts of material are produced in a high energy ball mill (attritor) with a total capacity of 8 litres. Isostatic pressing with up to 3500 bar can be performed in order to produce metal powder billets. This is done in a cold isostatic press with a cylindrical chamber of 40 cm in height and 20 cm in diameter.

Electron Microscopy

S. Zaefferer, A. Schneider

Electron microscopy is one of the most important and universal techniques in the study of microstructures of crystalline materials. In particular, the combination of imaging, diffraction techniques and chemical microanalysis allows a precise and complete characterization of the material on scales ranging from several mm using scanning electron microscopy (SEM) to a few nm using transmission electron microscopy (TEM). The MPIE provides a number of instruments, most of which are particularly well equipped for diffraction techniques and chemical analysis.

For transmission electron microscopy, a Philips CM20 200 kV TEM is available. This TEM is a standard analytical instrument for diffraction, tilt experiments and energy dispersive X-ray (EDX) investigations. A unique aspect of this instrument is a software system for on-line, semiautomatic crystallographic analysis. The system is connected to the TEM via a CCD camera and makes it possible to index all kinds of diffraction patterns quickly and directly for the purpose of orientation and phase determination. Furthermore, tilt experiments can be calculated in advance on the computer and then performed with greater ease on the microscope. This allows, for example, a simple quantitative characterisation of dislocations and grain boundaries.

The flagship of the electron microscopes at the MPIE is a new JEOL JSM 6500 F high resolution SEM. With this instrument, extremely high beam currents with a high resolution can be reached due to a special construction of the field emission gun (FEG). This system is therefore dedicated to the high-speed and high-precision measurement of electron backscatter diffraction (EBSD) patterns and EDX spectra. The instrument is equipped with the latest digital CCD camera for EBSD measurements and software programes for automatic crystal orientation mapping (ACOM - OIM 3 software by TSL) and phase analysis (Delphi software by TSL). A standard of up to 30 orientation measurements per second can be obtained during ACOM analysis. Alternatively, high quality images with 1300 × 1030 pixels can be acquired for crystallographic analysis. The combination of EBSD and EDX allows the investigation of complex materials containing a variety of different phases and microstructures.

A second SEM with a standard tungsten filament gun (JEOL JSM 840A), which is also equipped with ACOM facilities (software Crystal of HKL Technology), is used particularly for large orientation mappings, in the order of several 10 mm^2, where the sample stage rather than the electron beam is scanned during the measurement. In addition, the instrument can be equipped with a microtensile testing machine for in-situ deformation experiments.

Finally, a second high resolution SEM with FEG (LEO Gemini 1540 VP) is available which permits measurements to be made under variable pressures. This instrument is currently used for ultrahigh resolution imaging, for investigations of non-conductive polymer coatings and for the in-situ observation of oxidation experiments. It is therefore equipped with a heating stage with a gas-inlet. In the future, it will be also equipped with an EBSD system.

Electron Probe Microanalysis

M. Palm

Qualitative and quantitative analysis of solid samples is carried out using a CAMECA Camebax SX50 electron microprobe equipped with four wavelength dispersive spectrometers. The instrument has additional equipment for the analysis of light elements (Z < 10), especially for the quantitative analysis of carbon. It consists out of a gas jet, a cold finger, a multilayer crystal and computer software for enhanced light element analysis. The instrument can be run in "spot mode" for the analysis of volumes down to 1 μm^3 or in "scan mode", where either the stage or the beam is moved for a line scan or a scan of predefined areas. Quantified results can be displayed in various ways or evaluated through image processing software.

The electron microprobe is currently used for three applications.

Qualitative and quantitative analysis of phases:

- Determination of the compositions of coexisting phases, e.g. to determine phase equilibria
- Identification and compositions of precipitates, e.g. to distinguish between various carbides in steels
- Characterisation of corrosion products, such as oxides and chlorides

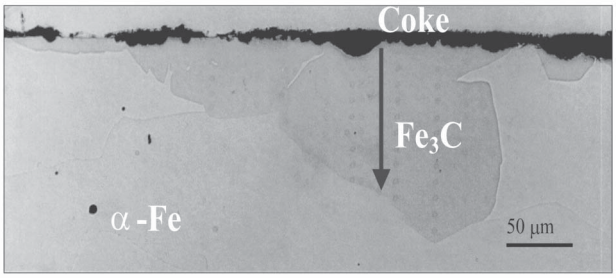
Measurement of concentration gradients:

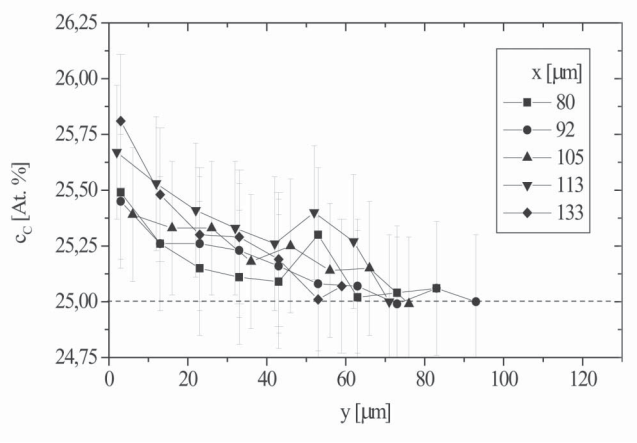
- Concentration profiles within diffusion couples, e.g. to evaluate interdiffusion coefficients
- Scales formed during corrosion, e.g. to study the underlying mechanisms by establishing sequences of different corrosion layers
- Zones formed by carburisation and nitriding, e.g. to examine the phenomenon of metal dusting

Element mapping:

- Characterisation of segregations, e.g. in sheets produced by twin-rolling

A special measuring strategy has been developed for the analysis of carbon which takes into account the effect of residual contamination, variation of the shape of the carbon Kα peak due to binding and a specific background correction for the relevant matrix. An example of a carbon measurement is given in the figure. On pure iron, large particles of





cementite, Fe_3C, have formed at the surface of the sample after carburising at 700°C. In contrast to the common assumption that cementite is a stoichiometric compound, careful examination of several line scans measured perpendicular to the growth direction of the cementite revealed a finite range of homogeneity [A. Schneider, Einfluß von H_2S auf die Bildung und den Zerfall von Eisenkarbiden beim Metal Dusting; Fortschr.-Ber. VDI Series 5, Vol. 544, VDI Verlag Düsseldorf (1999) p. 114].

In order to ensure a high quality of the analyses, participation in conferences, committees and round robin tests for electron probe microanalysis is regularly undertaken.

Metallography

S. Zaefferer

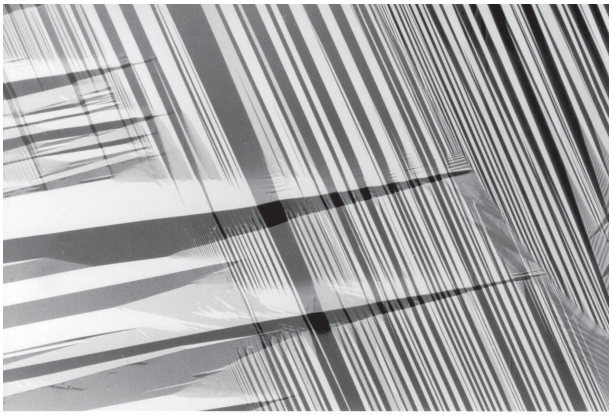
The task of the metallography service group is to perform metallographic work for the different groups of the institute or to instruct and support the members of the groups (for example Ph.D. students and post-doctoral fellows) with these tasks. Currently, the group consists of five experienced metallographic assistants and one photographer. The services offered by the group include the metallographic preparation of metallic, intermetallic and non-metallic samples for analysis or for further processing and

the observation and documentation of samples by optical and scanning electron microscopy.

A large variety of different techniques are available for sample preparation. These include cutting with various techniques (partly carried out in other departments), sample mounting (hot and cold, conductive or non-conductive resin, resin-free sample mounting), grinding and various polishing techniques. Electrolytic polishing is also available . After polishing, samples may either be chemically or electrochemically etched in order to develop the microstructure for further observation. For these tasks, a variety of different procedures, with focus on the preparation of microstructures of steels and intermetallics are available. Finally, specimens can be coated by sputtering with gold, iron, copper or carbon.

Various Leica (Aristomet), Zeiss (Axiomat) and other microscopes are available for the observation of microstructures by optical microscopy. A wide variety of observation techniques including bright field, dark field, differential interference contrast, polarised light microscopy and macrophotography with perpendicular illumination can be applied. Furthermore, a hot stage microscope for the observation at high temperatures and a micro-hardness tester are available. Photographs are either acquired conventionally on photographic film or by high resolution CCD cameras. Digital images are automatically sorted into an electronic database and can be analysed by software for digital quantitative metallography. An example of an optical photograph is given in the figure showing the microstructure of martensite developed in an Ni_3Al alloy as observed by polarized light microscopy.

For observation at higher resolutions, a scanning electron microscope Camscan CS4, equipped with an EDX system complete with a detector and ultra thin window for light element analysis is used. The instrument is operated and maintained fulltime by one of the group members.



Chemical Analysis

D. Kurz

The knowledge of the chemical composition is an important requirement for the use of materials in chemical experiments. Simultaneous atomic emission spectrometry with inductively coupled plasma and dual view optics (ICP-OES), atomic absorption spectrometry with a flame and hydride technique (FAAS, CVAAS), ion chromatography (IC), thermal conductivity and infrared absorption are the standard methods used by the group for the analysis of nearly 60 elements in different materials and matrices. The high level of accuracy and precision in analysis has made it necessary to update all the equipment in the last 4 years. The most recent investments were the purchase of a simultaneous ICP-OES and an ion chromatograph.

For more than 30 years, the group worked in co-operation with other laboratories in the German production group for European Certificated Reference Materials (ECRM). These materials are used for the calibration of the analytical equipment in the steel industry.

Approximately 70 percent of the analyses are measured with the ICP-OES, which has high performance Echelle optics with a wavelength range from 165 nm to 1000 nm and a spectral resolution of 0,005 nm. The dual view optics and the detector, which is a charge injection device (CID), allow the simultaneous measurement of element concentrations in a dynamic range covering 4-5 orders of magnitude and make it possible to simultaneously analyse low and high contents of elements without having to prepare a new sample.

In the future, a thin electrochemical cell will be designed, which will be directly connected to the ICP-OES in order to measure the composition of the electrolyte downstream from the cell. This will permit a qualitative identification of the soluble products and a quantitative measure of the simultaneous dissolution rates of the products over a time interval of less than 0,1 seconds.

Furthermore, it is planned that the IC will be applied for the monitoring of the electrochemical behaviour of conducting polymers. Using this combination, samples are taken during the experiments via a fraction sampler in time slices. These samples will be analysed subsequently by UV and conductivity detectors.

Computer Centre

H. Kemnitz

In a high speed data network, scalable computer servers share their services with low cost personal computers. The main advantage of such a structure is that the total cost is decreased without any loss of performance. The primary task of the service group is to provide, maintain and develop all necessary network resources. National and global high speed telecommunication is of major importance for scientific success. In 1988, the service group was already able to offer email and, as of 1991, internet access. In the past, a remote access bandwidth of 25 kbps was state-of-the-art, whereas the service group is currently updating the line speed to 34 Mbps. In general, the group offers and maintains all necessary network resources and services for global access and is responsible for information technology security. Furthermore, the group supports data connections to public phone networks.

A high speed local-area-network (LAN) was recently planned and set up based on the Ethernet technique for the main building with about 400 Fast and 20 Gigabit ports. The various renovation stages of the institute´s buildings have led to a number of computer-related tasks. These tasks continue to be addressed by the personnel in the computer centre.

Electronics Workshop

H. Kemnitz

Since industrial equipment is not always suitable for experimental needs in research laboratories, the Electronics group combines different equipments to computer-based systems for data acquisition, complex data analysis, and real-time process control. In addition, signal conditioners for exceptional sensors and special interfaces for large scale systems are either developed in the institute or in cooperation with external partners.

Outstanding examples of work completed in this service group include: process instrumentation of thin strip casting equipment; hydraulic control of roll slit and thermo-mechanical heat treatment in a rolling mill; high speed data collection system for tensile test machines; position control for a scanning Kelvin probe for the characterisation of complex surfaces.

Various software applications and operating systems are maintained for a large number of personal computers. To reduce the effort of maintenance, the group has developed a concept of unified hardware and simplified software using a so-called cloning technique. Those departments without computer administrators mainly take advantage of this service.

Technical Service

M. Winkler

The technical service has several working areas, which ensure the trouble-free operation of the technical installations within the institute.

The *electricity service* is currently, and will continue to be, updated to the state-of-the-art standard in the present and upcoming stages of construction. The in-house power line is supplied by two 1000 KVA transformers. In case of a loss of power, the most relevant (e.g. security) facilities are powered by an emergency generator.

A safety lighting along evacuation routes, such as stairways and corridors is guaranteed by a separate battery facility. All emergency routes are monitored by an automatic fire alarm system. In case of an emergency, the entire staff located in the buildings is warned by an acoustic alarm. The electricity service division assists all departments of the institute with the set-up and operation of their testing facilities. In the course of the institute's renovations, a new telephone system was installed and the data network was extended.

A newly installed *central building control system* regulates all new facilities of the buildings management *centrally*. The implementation of the new facilities was indispensable in order to fulfil the operational requirements of laboratories and offices.

Air conditioning systems such as extractor hood ventilation systems, deaeration facilities and constant climatised rooms are controlled by the central building control system. The majority of laboratories are partially climatised and are equipped with a circulating air cooling unit. A partially installed heat recuperation unit isapplied for the purpose of energy saving. Two refrigerating machines with a total power of 1000 kW guarantee the supply of cooling. A low temperature vessel is used for heat supply.

The institute's dual plumbing system ensures that incoming laboratory and potable water is strictly separated. Consequently, waste water also leaves the building separated. In addition, there is a neutralisation facility for chemically polluted wastewater.

The *gas and water services* are subdivided while being controlled centrally by the building control system and by a local supply. The centrally controlled media supply facilities provide compressed air, deionised/demineralised water and special gases such as nitrogen, argon, etc. For critical gases, gas warning devices are installed.

In order to handle the requirements mentioned above, the buildings management uses the digital data control (DDC) technique, where the relevant data from all areas merge prior to recording and processing. The single stations of this service are located in technical centres, distributed throughout the building and are digitally linked to each other and controlled by the central building service.

Mechanical Workshop

R. Selbach

Within the last two years, the workshop was mainly occupied with the machining of samples and the manufacturing of scientific devices. Further tasks are locksmith works, support of maintenance and the repair of several institute facilities.

Different materials, such as rust and acid-proof stainless steels, nonferrous metals, synthetics, carbon and ceramics were machined. Samples, specimen holders and equipment were prepared to serve the needs of the different departments. The quality of scientific results is significantly affected by the accuracy maintained during sample preparation. This task of the workshop makes great demands on its staff and equipment as geometry and material vary depending on the experiment.

When alloys with unknown mechanical properties have to be machined, trial-and-error methods are applied in order to select the optimal cutting parameters and a suitable cutting material.

Highlights of the manufacturing work are a submerging basin and a vacuum chamber made of acrylic glass. Furthermore, a water model of the twin roller mechanism was developed. The construction of several Kelvin probes was one of the most sophisticated and time consuming tasks. Apertures, drill holes and inlet pipes had to be integrated into highly acid-proof, antirust stainless steel. Oscillating heads were manufactured with an extremely high accuracy. Reworking the mould wheels of the twin roll caster, a near-net-shape casting device, was another very time consuming task.

The workshop has a range of modern machinery at its disposal, e.g. CNC controlled turning lathes, milling and EDM machines.

Administration

H. Wilk

In 1998 the part of the administration department responsible for finance started to work with SAP R3. The implementation of this business-software - with the assistance of Max-Planck-Gesellschaft - was important to render possible a better and faster access to information about the financial situation of the institute. The removal into the reconstructed main-building of the institute gave further opportunities to modernize administration. The technical equipment and linkage of information at any working place make it possible to get internal and external facts faster.

The new structure of management with four executives in the institute gave the opportunity to reorganize the administration depending on the different interests. The historical assignment of Technical Service, EDP and Electronic Workshop at the administration department was changed in the beginning of 2002, the key-functions accounting, purchase of materials and staff-administration as well as cross-functions remained within the administration. The management of these functions was changed so that now “Sachgebietsleiter” are responsible for the individual tasks which are supervised by the executive of administration.

At the same time, the key function of purchasing was extended from ordering goods to real procurement and includes the responsibility for goods received and stored. This long-term plan to merge the function of procurement with receiving was made possible once the difficult search for qualified staff was successfully brought to a close. This new leader is, in the meantime, very familiar with the institute and its structure. The intention of the change of structure and organisation is to reduce the volume of stored goods in the work-shop and to close the storage of chemical and office-materials. In addition, it was necessary to reorganize the rules and responsibilities of ordering and subscribing. Above all, it is a permanent target to make procurement more efficient by using modern electronic media.

Preparations are under way to introduce the “Human Resource” module of the business-software SAP R3 in the near future by the staff-administration, again with the assistance of Max-Planck-Gesellschaft. This module will extend the integration of business-information substantially and terminate the out-sourced computation of salaries and wages.

Newly Established Scientific Laboratories in the Renovated Main Building

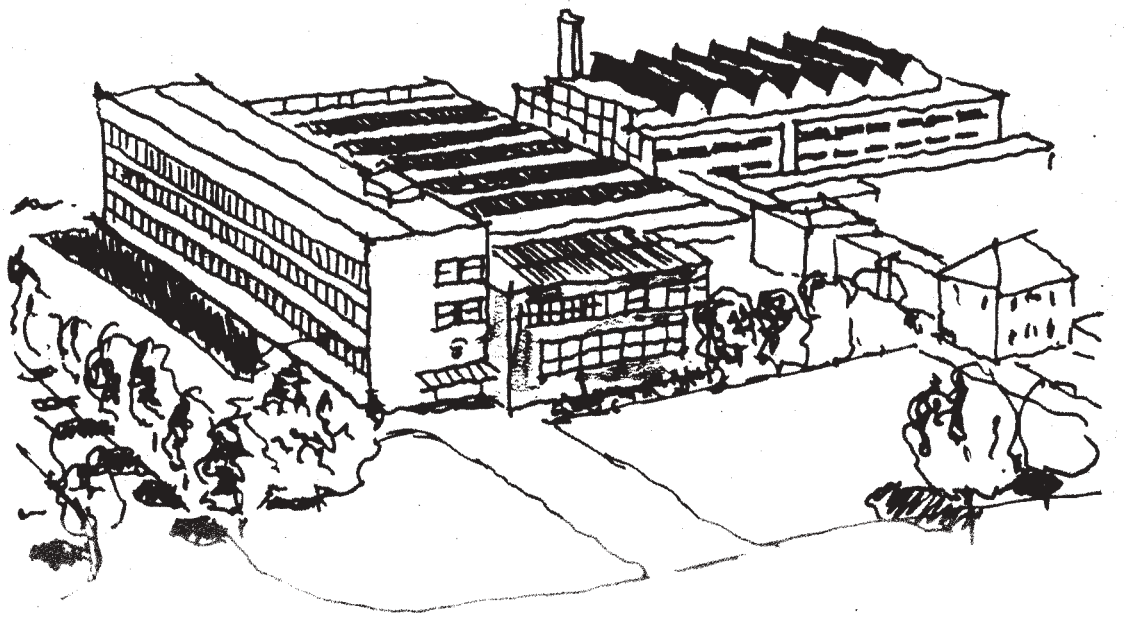
Reconstruction of the Building

The buildings of the Max-Planck-Institut für Eisenforschung were erected in two historically different periods. The construction of the main building including halls 1 to 7 was completed in 1936. In the 1960s, two further halls (hall 8 and 9), an additional laboratory- and office-block, and the isotope laboratory were built. This second construction phase was completed in 1971. The main building was designed by the renowned architect Prof. Paul Bonatz from Stuttgart. Due to its architectural importance, the main building is now under historical monument protection. Its cube-shaped form and horizontal rows of windows are very typical characteristics of Classic Modernism ushered in with Bauhaus.

Main entrance of the reconstructed building

General reconstruction work started in 1999. The initial phase was planned for the main building and hall 4; the other halls are to be restored in the second and third reconstruction phases.

Before work was started on the buildings, office and laboratory containers were erected next to the main building to allow all staff affected by the reconstruction work to continue working in temporary accommodation throughout the redevelopment. In this way, the main building could be fully cleared to enable the reconstruction work to be carried out without interruption.



Schematic drawing of the Max-Planck-Institut für Eisenforschung showing the main building on the left-hand side with halls 1 to 7 just behind it and the larger halls 8 and 9 at the back on the right side (taken from „Bauten der Max-Planck-Gesellschaft - Max-Planck-Institut für Eisenforschung, Düsseldorf" published by the MPG, Munich and VDEh, Düsseldorf)

At the time reconstruction work commenced, the buildings had preserved their original structural form, apart from the bomb damage suffered by the main building in World War II, which is still visible on the west clinker-brick façade.

The main building comprises a load-bearing steel framework encased in reinforced concrete. The floor sections are fastened between the steel frames. The interior walls are lightweight partition walls constructed of breezeblocks. The building is 75 meters in length and 15 meters wide. It has four stories with an untypical ceiling height of 4 meters.

The basic original internal organization was retained during the reconstruction. The lower basement area houses storage rooms and the building's technical systems. Bulky scientific equipment is installed in the basement, while the first floor accommodates a cafeteria, laboratories and offices. A conference hall, the institute's administration and further laboratories and offices are on the second floor. The third floor is dedicated to laboratory use. The top floor houses a number of special laboratories and the central ventilation system. The original room allocation was adopted during the redevelopment - i.e., large laboratories were constructed in the west wing, small adjoining rooms and studies in the east wing.

In addition to the spatial and functional interests of the institute, the reconstruction planning paid particular attention to architectural conservation aspects while still satisfying fire regulations and laboratory requirements.

Staircase with the typical lighting, characteristic natural stone surface, and high quality steel railings

View into the main supply shaft

Working closely with the historical monument preservation authorities, major sections of the building were restored or preserved: the main hallway with staircase and existing doorway made of stainless steel, the foyer with a ceiling-high relief in stainless steel and natural stone, the stairwells with dark shell marl on the steps and landing surfaces, along with steel banisters and stainless steel handrails. Of the interior doors, the access doors to the offices with horizontally partitioned glass panes have been preserved. The clinker-brick façades and stainless steel window frames of the south-facing gable on the entry side of the building were also left unaltered.

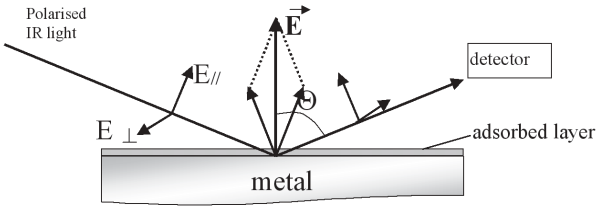
The building now must serve much more demanding uses than in its original state. The reorientation of the building's functionality resulted in an increased wiring and equipping density with new primary and secondary shafts. Thanks to the intensive co-operation between all parties involved in the planning of the reconstruction, working areas installed with modern technology have been created in the historical building. *(Photos: Lukas Roth, Köln)*

New Scientific Laboratories

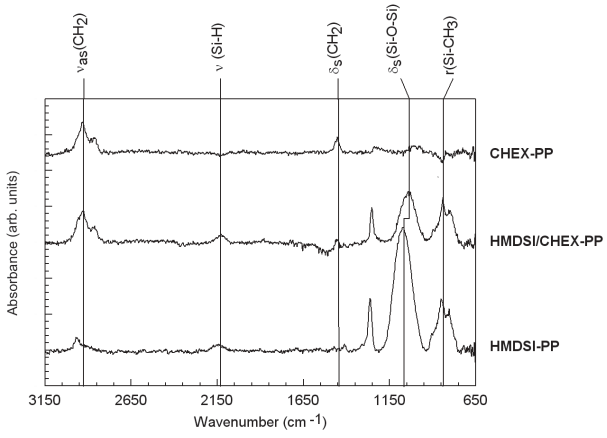
Optical spectroscopy laboratory

P. Thiemann

The laboratory for optical spectroscopy consists of various techniques to study the thickness and chemical structure of ultra-thin and thin inorganic and organic films on metals, coatings and adhesives, and colloidal dispersions. Moreover, reactions on surfaces can be studied in-situ. Internal spectroscopy provides the possibility to study the adsorption of organic molecules from solutions or the gas phase and the swelling of polymer layers. Complementary to the UHV surface analysis, optical spectroscopy permits the study of surfaces in real environments and, in most cases, without damaging of even highly sensitive surface films. Optical spectroscopy yields information on the chemical structure and orientation of molecular units, electronic and vibronic excitations and relaxations, and optical materials constants such as absorption coefficients and refractive indices. Microscopy further allows the local study of heterogeneous surfaces with micrometer resolution.



Principle of FTIR measurements under grazing incidence on highly reflecting metals (IRRAS).



In-situ IRRAS measurement of an ultra-thin gradient layer.

Spectroscopy of ultra-thin and thin inorganic and organic films on metal surfaces: Infrared Reflection Absorption Spectroscopy (IRRAS) permits the study of films at the monolayer level and of the orientation of the adsorbed molecules.

Infrared spectroscopy at buried interfaces: Attenuated total reflection spectroscopy (ATR) enables the measurement of the inner interface of coatings and adhesives. Light is totally reflected at the interface between a prism and an organic layer or electrolyte. The resulting evanescent wave propagates into the adjacent medium in the submicrometer range. Thus, spectroscopy can be used to investigate buried interfaces. This is extremely interesting for the study of the swelling process in polymer layers or the degradation of these polymer films in aggressive media.

Microscopic spectroscopy: Spatially resolved optical spectroscopy can be done by means of Raman microscopy (~1 μm) or FTIR microscopy (~10 - 100 μm). The dispersive Raman microscope allows measurement of the film composition even if material to be investigated is located underneath an electrolyte film. In principle, the sensitivity for thin films is low and can only be increased using Surface Enhanced Raman Spectroscopy (SERS) which requires a complex surface preparation technique. IR microscopy can be done in transmission, internal reflection and grazing incidence modes. With grazing incidence, films with a thickness of a several nanometers can be investigated with a spatial resolution of about 100 μm.

Film thickness measurements by means of ellipsometry: Film thickness measurements can be done by means of imaging ellipsometry (one wavelength, multiple angle) and as of January 2003 by means of spectroscopic UV-VIS ellipsometry. Thicknesses that can be measured range from subnanometer to micrometer thick films. Optical models can take nonhomogeneities such as roughness and chemical gradients into account.

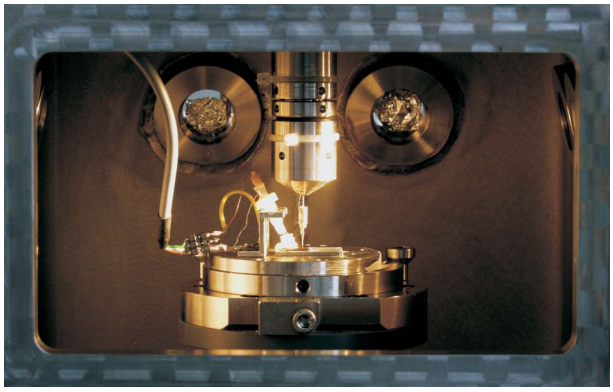
Studies of solution chemistry and film structure by means of UV-VIS spectroscopy: A state of the art UV-VIS ellipsometer is used to study the absorption of thin films on surfaces and to reveal the structure of colloidal dispersions. The measured wavelength region can be extended to 175 nm when the spectrometer is purged with nitrogen.

Application of a „Multiskop“ which combines one-wavelength, multi-angle ellipsometry, surface plasmon spectroscopy (SPS), waveguide modes, imaging ellipsometry, and SPS microscopy in a single modular set-up.

Kelvin probe laboratory

A. Mingers

The *Kelvin probe* is a non-contact, non-destructive vibrating capacitor device used to measure the work function (wf) difference or, for non-metals, the surface potential (sp) between a conducting specimen and a vibrating tip. The wf is a multiparameter variable and is an extremely sensitive indicator of the surface condition or of surface modification via adsorption, surface roughness, etc. Under certain circumstances, the measured work function difference can be calibrated in such a way that SKP measures electrochemically relevant quantities. Therefore, the Kelvin probe is able to measure local electrode or corrosion potentials. The major advantage of the Kelvin probe is the capability of measuring potentials across a dielectric medium without contacting the sample surface.



The Scanning Kelvin Probe is a non-contact method for in-situ investigations of buried interfaces and delamination kinetics.

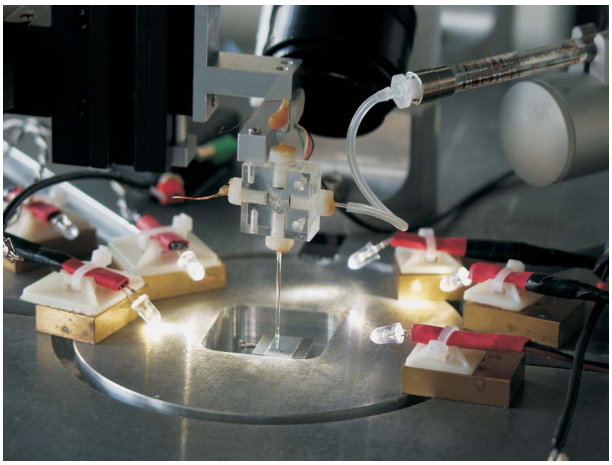
The scanning Kelvin probe has successfully been applied in the investigation of the following corrosion systems:

- delamination of polymer-coated materials in the vicinity of the defects
- the outdoor corrosion of metals
- the stability of modified metal surfaces
- the indoor corrosion of electronic materials such as aluminium and copper.

Electrochemistry / microelectrochemistry laboratory

A. Mingers

The electrochemistry laboratory consists of four work places. Each of them consists of a table with the electronic equipment and a chamber, which is under constant suction and where the experimental



Scanning Droplet Cell for microelectrochemical investigations of surface inhomogeneities or local corrosion processes.

set-up can be placed. There are three work places for standard electrochemical investigations; two of them can also be used for *impedance spectroscopy*. The fourth work place is dedicated for an “*electrochemical nanomachining apparatus*“, in which ultrashort pulse voltages can be applied between a scanning microelectrode and a specimen electrode to etch a local area of the specimen surface or deposit layers. Since 2002, this technique has been introduced for the micro-fabrication of shape memory NiTi-based alloys, which are difficult to fabricate in the micro to nanometer range by general mechanical machining techniques.

The microelectrochemical work is based on the fact that corrosion at electrode surfaces is often inhomogeneous. Well-known examples are pitting, filiform corrosion and intergranular corrosion. This local

breakdown of passivity may result from weak spots in the passive film formed by mechanical, chemical or thermal stress or may result from the structure of the base material. Since the stability of a material is usually determined by its weakest part, a microscopic investigation of the electrode surface is necessary to understand the macroscopic corrosion behaviour.

A novel method, namely the *scanning droplet cell* allows an effective, spatially resolved, in-situ investigation by all standard electrochemical techniques such as cyclic voltammetry, electrochemical impedance spectroscopy, current transients of potentiostatic pulse steps or rest potential measurements. The basic idea of this method is to position a small drop of electrolyte with a capillary on the investigated surface. The wetted area acts as a working electrode and the glass capillary contains the counter and reference electrodes, which are electrolytically connected to the surface through the drop.

Another microelectrochemical method is the Slurry jet system that is used for the investigation of electrochemical microelectrode transients generated by *slurry jet impingement*. This system consists of a slurry pump, a microelectrode, and a home-made potentiostat suitable for measurements of rapid current transient for small currents generated by slurry particle impacts, and a high-frequency data acquisition computer board with a maximum data acquisition frequency of 20 MHz.

Corrosion laboratory

A. Mingers

There are two kinds of corrosion tests that can be performed in this working group.

One of these analyses is *salt spray testing* performed in a chamber, which is available for performing accelerated corrosion testing of sample specimens. Standard tests such as salt spray (DIN 50021), condensed water (DIN 50017) or changing climatic conditions (VDA 621-415) can be performed.

In addition, programmable tests with parameters different from the standard testing are carried out. These include temperature, humidity, exposure time periods or the ratio between different test phases for the delamination and corrosion behaviour of various test specimens.

In the near future, newly developed steel-based materials and coatings with an increased resistance to environmental corrosion will be tested using the salt spray chamber.

The second kind of corrosion test is a new *dip / dry corrosion testing unit*. This unit has been developed for material screening by performing fast cyclic corrosion tests by dipping test specimens into various electrolyte solutions. The experimental set-up allows the exposure of up to eight different samples in each of the three water basins. The corrosion conditions range from ambient dry air to total immersion in electrolyte as well as a well-defined humidity gradient by adjusting the position of the samples over the electrolyte surface. The exposure times for different test phases can be selected through the control software. Potential transients and impedance spectra can be taken during the test specimens' immersion.

The experimental set-up was used for investigations of the passive layer formation on weathering steels in different types of electrolyte solutions, thereby simulating the various corrosion conditions of marine environments. Present corrosion experiments focus on the cathodic delamination activation time of polymer coated, zinc-plated steel sheets in cyclic corrosion tests.

Surface science laboratory

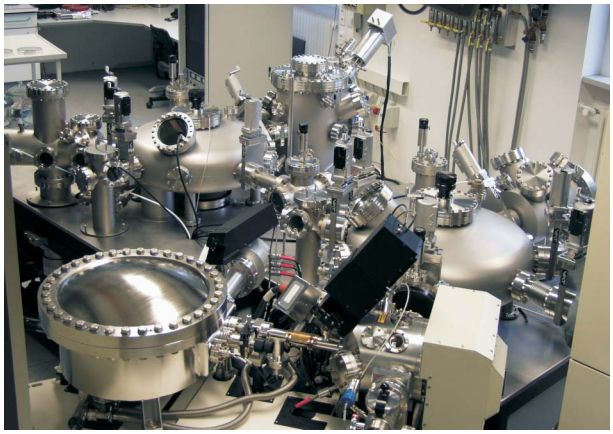
E. Heinen

At the centre of the laboratory is a multifunctional UHV system, which combines several surface treatment and preparation units with a number of sophisticated state-of-the-art surface analytical systems. The concept of this complex unit is to enable the transfer of samples between the different units at any user-defined stage of the sample treatment and permits surface analytical characterisation without exposure to air. Thus, complex processes such as plasma treatment or initial stages of oxide film growth can be studied at different stages.

The different units are grouped around two distribution chambers. Attached to the first distribution chamber are the routine surface analytical instruments, the small spot ESCA (PHI Quantum 2000) and the ToF-SIMS (PHI TRIFT II), as well as two process chambers not usually found with UHV systems, i.e., the plasma chamber and the high-temperature oxidation chamber. In the latter chamber, surfaces can be prepared by thermal oxidation or reduction processes and subsequently transferred to the analytical devices.

A high percentage of the samples investigated in these instruments is not prepared in the UHV system itself, but come from external corrosion and delamination experiments. For this reason, the first distribution chamber is referred to as the *service part*. Within this part of the system, the base pressure may not always remain within the specified pressure range for the distribution chambers (10^{-10} to 10^{-9} mbar).

The second part of the system is referred to as the *surface physics part*. Here a dedicated preparation chamber equipped with LEED/AES and an ion gun, an MBE chamber which allows the well-defined evaporation of model alloys and oxides, and a variable temperature STM (VT-STM, Omicron) are grouped around the second distribution chamber, which is in turn connected to the first one. In this part, mandatory precautions must be followed in order to maintain a base pressure within the 10^{-10} mbar range.



Partial view of the UHV system.

Sample transfer between the different units can be carried out quickly and simply. This is guaranteed by a sophisticated sample transfer and piggy-back concept between the different units. Sample transfer is also possible to a VG Microlab (ESCA and SAM), to the LEO FE-SEM and to a glove-box (allowing electrochemical experiments under controlled conditions - below 1ppm oxygen) via a UHV transfer vessel that can be attached to the system's loading locks.

Since a high number of UHV instruments are present in the laboratory, the roughing pumps (which provide the pre-vacuum) were installed in the basement below the laboratory. This ensures a quiet and vibration-free environment. The pre-vacuum tubing and other cabling is installed in the double floor of the laboratory.

Most of the specialised systems (such as the VT-STM, LEED or also the MBE) are, more or less, reserved for special projects and their use is restricted to a limited number of qualified personal. However it is an integral part of the lab´s concept to provide measurements with the standard surface analytical equipment, such as the Quantum, the TRIFT or the Microlab, on a routine basis to all interested parties.

The Quantum 2000 small spot ESCA works with monochromated $K_{\alpha\ Al}$ focused to a spot of 10 μm diameter. The characteristic information depth of ESCA lies between 5 and 30 nm. The TRIFT II ToF-SIMS is extremely surface sensitive and is an ideal tool for analysing polymeric materials. In particular, the use of isotopes provides fundamental insight into reactions can be obtained. The lateral resolution is limited by the spot size of the Ga ion beam, ca. 100 nm. The sensitivity of the instrument, while dependent on a number of different parameters, typically lies in the ppb range.

The Thermo VG Scientific Microlab 310 D combines ESCA and SAM (Scanning Auger Micro-scopy). The SAM function has been upgraded with a novel field emission electron source, which will provide mappings with a resolution in the 20 nm range.

Secondary neutral mass spectroscopy (SNMS) (E. Strauch): This analytical method gives information about the elemental distribution within a corrosion scale by mass spectrometry. Ar ions are generated in a plasma chamber and accelerated to the sample surface for sputtering. Sputtered neutrals are ionized in the plasma chamber and subsequently detected within the mass spectrometer. By continuous sputtering, depth profiles of certain elements can be recorded. Thus, SNMS is an essential method to characterise all types of layers formed by corrosion and coating experiments.

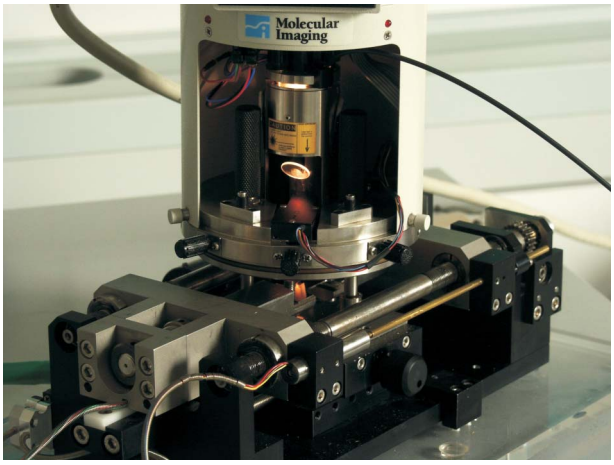
The scanning probe microscopy (SPM) laboratory

P. Thiemann

The routine measurements in the SPM lab are usually carried out with the Nanoscope Dimension 3100 SPM with Nanoscope IIIa controller. The most commonly used tapping mode AFM function is easy to use and provides high quality topographical AFM images even on problematic, technical samples. The Nanoscope system can also be used in a number of different other modes, including: lateral force microscopy, magnetic force microscopy and Kelvin mode microscopy (Scanning Kelvin Probe Force Microscopy, SKPFM). In particular, the SKPFM mode has attracted considerable attention. A special humidity cell was designed and the first experiments on microscopic aspects of filiform corrosion and cathodic delamination have been carried out.

A dedicated in-situ instrument is the Pico SPM from Molecular Imaging. Owing to its special design, the Pico SPM is an ideal tool for STM and AFM analysis in diverse chemical environments. It can also be positioned on a specially designed tensile stretching device. Using this set-up, it is possible to perform in-situ AFM investigations on the forming behaviour of the substrate. During the last two years, this set-up was used for investigating the defect formation in ultra-thin plasma polymer coatings upon tensile

stretching of the underlying steel. In the near future, it is planned to resume in-situ STM investigations on the de-alloying of binary alloys, which were stopped two years ago due to of the adverse conditions during the interim period.



Experimental set-up which combines an Atomic Force Microscope with a uniaxial stretching device. The forming behaviour of thin films on metal substrates can be studied in-situ as a function of strain.

High-temperature corrosion laboratory
D. Vogel

Exposures: The long-term performance of metals in corrosive atmospheres can be determined by exposure over longer time periods. Typical concerns are the behaviour of alloys and coatings in waste incineration or biomass combustion atmospheres in order to develop more resistant materials and to extend the service life of the plant components. These corrosion measurements are accomplished in complex and extremely aggressive atmospheres, including the use of molten salt deposits on surfaces. The gas mixtures are composed of a carrier gas and several corrosive species, i.e. SO_2 and CO_2. Water and hydrochloric acid are supplied as vapor by saturators or evaporators. Gas analytical equipment enables the changing of certain gas species during the experiment.

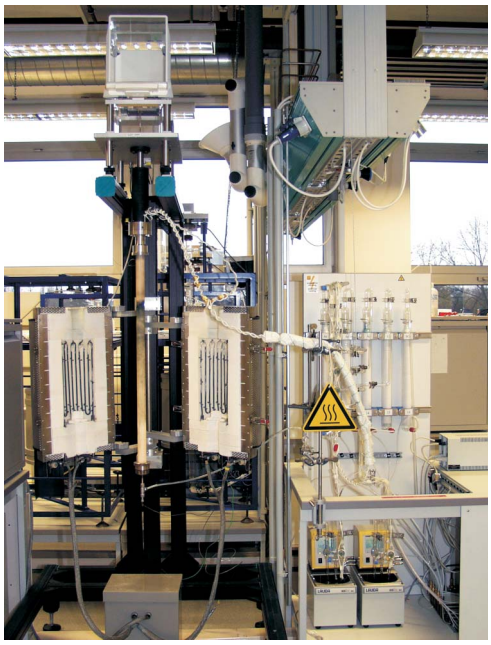
Thermogravimetry: The kinetics of solid-gas reactions can be measured by thermogravimetry. With this technique, the mass changes of metallic specimens are recorded time-resolved at a constant temperature. As in the case of exposure experiments, investigations are possible in aggressive gases as well as beneath salt deposits.

To investigate the initial state of these reactions, it is necessary to combine a rapid heating with a sensitive mass gain recording system during oxidation. This is realized by a setup, which consists of a microbalance (accurate to 1 µg and a heating chamber with four quartz lamps and quad elliptical reflectors. The reflectors are configured for an optimum focus of the infrared energy from the lamps to the specimen without influencing the passing gas.

In-situ analytical high-temperature laboratory
E. M. Müller-Lorenz, D. Vogel

In-situ high-resolution microscopy: High-resolution (1.3 nm, theoretically) FE-SEM with EDX is used successfully for the characterisation of the surfaces during and after high-temperature exposures. EDX analysis gives quantitative and qualitative information about the elements present on the surface and their distribution (by mapping). In addition a high-temperature chamber placed in the FE-SEM allows in-situ experiments (up to 1050 °C) in different gas atmospheres and beneath salts. The oxide formation and growth with time and temperature can be obtained during this investigation.

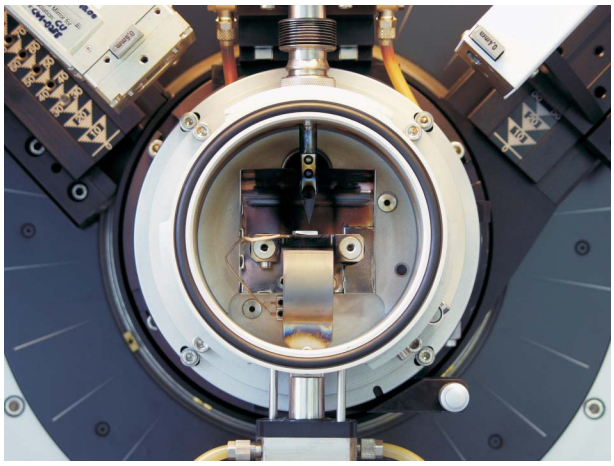
In-situ grazing incidence X-ray scattering: For in-situ studies of the morphology, structure and growth kinetics of oxide scales formed by thermal oxidation of metals, an X-ray diffractometer with a high-temperature chamber for non-ambient reflectometry (XRR) and diffraction (XRD) measurements is available. With XRD, especially grazing incidence XRD, ultrathin oxide scales can be investigated with



Experimental set-up for kinetic investigations consisting of a microbalance, a reaction furnace and gas conditioning equipment.

respect to structural changes during heating. In addition, XRR gives information about the morphological characteristics of single and multilayers regarding thickness, roughness and electron density. Through the combination of these structural and morphological aspects, characterisation of ultra thin films and oxide layers is possible.

With this high-temperature stage, sample temperatures of up to 800 °C are attainable in a high vacuum, in inert gases as well as in oxidising atmospheres. The chamber enables a wide field of application. The front cover of the chamber is easily removed and providing an easy access to of the specimen, so that more complicated sample preparations on the stage, i.e. reactions beneath molten salts, are possible. The oven-like arrangement of the heater provides reliable, homogenous sample temperatures of the sample and fast heating.



High-temperature reflectometry chamber for the Bruker D8 diffractometer. The system can be flushed with reactive gas in order to study in-situ.

Materials characterisation laboratory
W. Vogt, T. Schildheuer

X-ray diffraction: X-ray diffraction is used for the characterisation of microstructures. Diffractometers with Bragg-Brentano geometry and monochromatic radiation are used for powder specimens or metallographically prepared samples. Position sensitive detectors with 120° angular range in 2θ are used for fast registration. For high precision lattice parameter determinations a conventional detector system with high angular resolution is available.

Phase equilibria may not be quenched to room temperature without alteration. In such cases in-situ high temperature observations are necessary. For this purpose two systems are available with an operating temperature range up to 1200 °C and 1600 °C. A special specimen holder was constructed in order to achieve good homogeneity in temperature (5 °C at 1600 °C). One system works in Bragg-Brentano geometry, a second one in Seemann-Bohlin geometry with Ge-monochromator for high intensity, both with position sensitive detectors. The fast recording allows not only to study equilibria at high temperatures, but also the kinetics of phase transformations. The Seemann-Bohlin equipment is designed to operate under gas atmospheres. This opens a new field of in-situ studies where phase transformations can be studied under given activities imposed by the controlled gas atmosphere.

Thermal analysis: High temperature thermal analysis (DTA) and thermogravimetry (TGA) is available up to 1700 °C . The DTA equipment operates under vacuum or inert gases, usually with a controlled temperature-time profile. The main purpose is the determination of transition points (from liquidus-solidus to solid-solid). Appropriate calibration also permits to determine heat effects during phase transitions and to give information about the kinetics by varying the temperature-time profile.

The TGA instruments operate preferentially at a given temperature and under given gas atmospheres. Reactions taking place between sample and gas are recorded by the weight changes versus time.

PART II.

THE DEPARTMENTS

(in alphabetical order)

Department of Interface Chemistry and Surface Engineering

M. Stratmann

Scientific Concepts

The department has its main focus on chemical reactions and physical properties of surfaces and interfaces. Materials based on metals, polymers and metal/ceramics or metal/polymer composites are of particular interest. Scientific studies concentrate on degradation reactions such as aqueous corrosion, high-temperature corrosion, tribocorrosion and de-adhesion with the aim to understand their underlying physico-chemical reaction mechanisms. Based on this knowledge, new and superior surfaces and interfaces are designed, characterised by their novel chemical composition, morphology and molecular and atomistic structure. Frequently, this requires surface modification techniques. In particular, technologies necessary to change chemical and physical properties of surfaces and interfaces are among the main activities of the department.

Besides the formal structure of the department in form of scientific groups having their own experimental and scientific expertise, a strong collaboration of these groups aimed to reach major scientific achievements in clearly defined areas of interest is strongly encouraged. The following topics can be regarded as such central scientific issues of the department:

Stability of buried interfaces

The aim is to understand de-adhesion reactions on a molecular level. In order to reach this aim experimental methods have been improved significantly and simultaneously model interfaces have been developed which allow the study of fundamental physico-chemical processes leading to de-adhesion (Fig. 1).

The *experimental methodology* is unique even on an international scale. It includes further developments of the scanning Kelvin probe [1-3] including a 3-dimensional Kelvin probe able to scan the local electrode potential over non-even surfaces and a Kelvin probe, which for the first time allows the detection of the local electrode potential in the vicinity

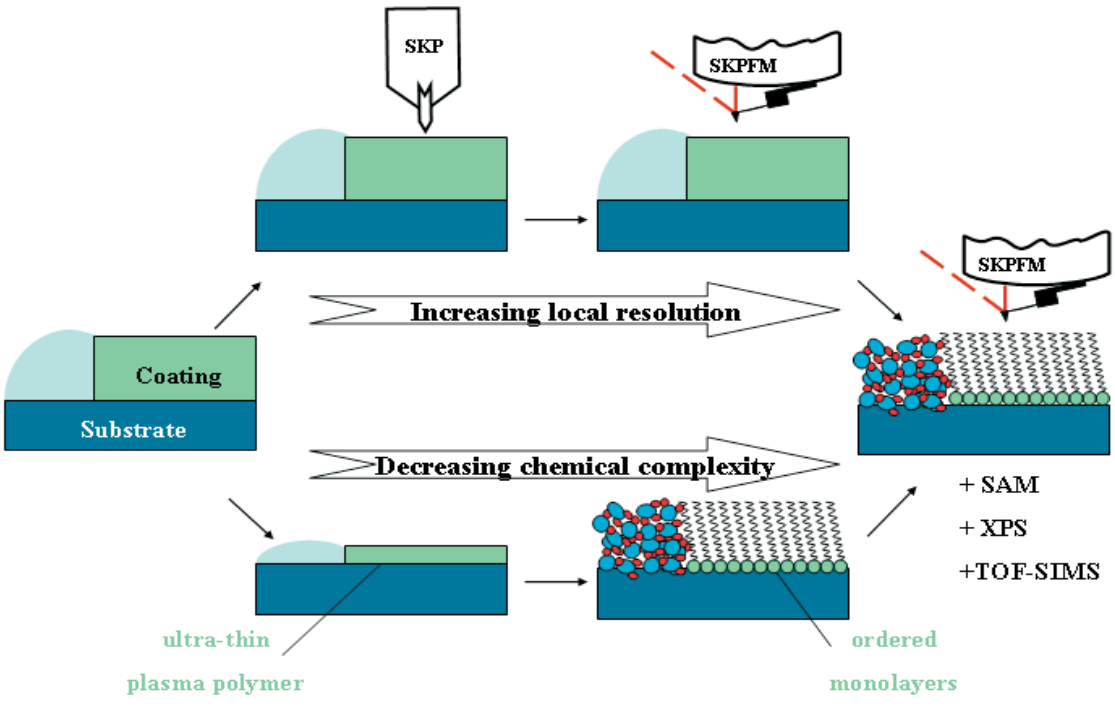


Fig. 1: *Scientific scope for the experimental investigation of elementary de-adhesion reactions at metal/polymer interfaces.*

of the mechanically stretched interface and therefore allows deep insight into elementary processes of adhesion and de-adhesion [2]. Particular emphasis has also been put on the improved local resolution. The conventional scanning Kelvin probe is limited to a local resolution of approximately 10 µm, further improvement is possible in particular using the scanning Kelvin probe force microscopy (SKPFM) [4, 5]. The first delamination studies using this technique have succeeded within the department recently, showing a significantly improved resolution but also a number of physical limitations which are subject of actual studies. Besides these novel electrochemical techniques in-situ spectroscopic studies of buried interfaces have made considerable progress. Ongoing studies concentrate on the migration of D_2O into interfaces starting from local defects and detected by in-situ Fourier transform infrared microscopy (FTIR-microscopy), on the spectroscopic analysis of reactions in conducting polymers being present at buried interfaces and on in-situ spectroscopic studies of the growth of ultrathin inorganic and organic films which are later used as corrosion inhibiting films below organic coatings.

Based on well defined experiments the *understanding* of deadhesion reactions has improved significantly. Key issue is the link between the molecular defect structure of the interfacial polymer, the electronic properties of the oxide scale and the rate of electrochemical reactions at the interface. The corrosion driven deadhesion has been studied on model samples like iron, zinc and aluminium. Based on these studies general de-adhesion mechanisms have been proposed (Fig. 2) [6]. Ongoing studies concentrate on the simultaneous mechanical and electrochemical attack of interfaces as is typical for adhesives being used in out-door applications. The studies are based on the concept that changing locally the free volume within the polymer will enhance the incorporation of ions and therefore electrochemically driven de-adhesion reactions. Furthermore mesoscopic defects being created during forming and their influence on the local stability of the interface has been subject of actual research.

Based on the understanding of de-adhesion reactions *new and highly stable interfaces* have been designed. To mention only few: the department is investigating the formation of zinc layers on iron based alloys with superior mechanical properties, which usually suffer from poor wettability, new zinc-alloys based on the Zn-Mg system are subject of active research [7], we have shown that the electronic properties of oxide scales formed on zinc alloys give rise to highly stable interfaces, cooperation with the chemical industry aims to the development of new and ultrathin conversion coatings based on inorganic/organic hybrid systems, ultrathin and chemically and structurally graded plasmapolymers [8] have been very successfully investigated showing a close link between the defect morphology of these layers at the interface and their electrochemical stability and last but not least film formation from aqueous dispersions are being investigated again with the emphasis on the local defect chemistry at the interface.

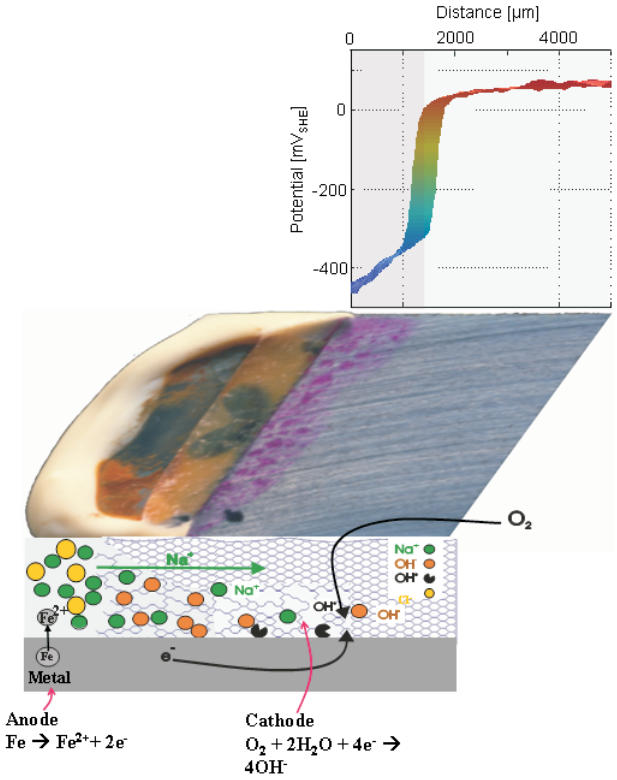


Fig. 2: *Generalised model of cathodic delamination*

a) upper part: Potential profiles as measured by the Kelvin probe

b) central part: View of a delaminating interface, the delaminated part being visible due to phenolphthalein pigments

c) lower part: reaction model.

Corrosion of metals

The area of corrosion science is of particular interest for the department [9]. Future work will concentrate in particular on *elementary and mostly atomistic reactions during alloy dissolution and alloy oxidation* [10, 11]. During the last two years these activities could not be pursued as wished due to the lack of STM facilities with atomistic resolution under conditions of the temporary labs of the department. Actually a complex UHV laboratory is being built up including highly advanced STM facilities. The laboratory should be in operation at the end of 2002 and will then allow the in-situ studies of atomistic corrosion reactions as described above.

Elementary reactions during flow induced localised corrosion of passive metals are actually investigated in an experimental set-up which is designed for the analysis of single and well defined substrate-particle interaction. The *high-temperature corrosion* activities concentrate on alloys in conventional and advanced energy systems (waste- and biomass fired plants, molten carbonate fuel cells) with special emphasis on the thermodynamics and kinetics of salt melt induced corrosion of alloys with the aim to model high-temperature corrosion processes in a given environment, on localized reactions of salt particles with alloy- and oxide surfaces and on degradation mechanisms of oxide scales in the presence of HCl.

Functional layers

Besides increased interfacial stability it is the clear aim of ongoing research activities to increase the functionality of surfaces on structural materials. Most of these activities have just been started but will prove increasing significance in the coming years.

Functional layers include *highly corrosion resistant zinc alloys* (based e.g. on Zn/Mg), *active repair systems* based on conducting polymer pigments making use on their potential dependant ion exchange properties, *scratch resistant polymeric surfaces* based on plasma-polymer nano-composite layers with embedded nanocrystalline inorganic particles and surfaces with well defined wetting properties. For the latter property, plasma polymer films have been successfully developed with a tailored roughness and chemical composition based on organosilicon and fluorcarbon monomers.

Collaboration with the steel industry

Some research activities are closely linked to actual industrial developments. These activities include investigations on *the corrosions resistance and surface protection of Trip and Twip steels* taking into account their high concentration of alloying elements which give rise to unique metal/gas reactions during annealing, to considerable difficulties during hot dip galvanising due to partial wetting of the surface by zinc and to corrosion properties which differ strongly from the homogeneous corrosion of low alloyed steels. Partly linked to this research are investigations on the *kinetics of metal/gas reactions and the subsequent hot dip galvanising of high strength steels*. In order to study scale formation and scale properties for wetting a unique experimental system has been developed which allows to control the scale formation under well controlled conditions, to monitor the contact angle between liquid zinc and the oxide scale using a high speed video system and simultaneously to remove the liquid zinc layer by fast spinning of the sample in order to lay free the reaction zone between the substrate and zinc. Furthermore the *influence of small amounts of salt particles on the corrosion of polymer coated steel* is investigated together with industrial partners. Last but not least the *atmospheric corrosion* of novel surfaces and alloys ("patina on steel") is of considerable industrial interest and subject of ongoing research activities.

Modelling

Modelling activities are actually limited to thermodynamic calculations in the area of metal/gas reactions. It is however anticipated to increase research efforts in this area significantly with particular emphasis on the structure of metal/polymer interfaces, the diffusion of small molecules and ions into the interfacial zone and on reactions triggered by the presence of water and ions close to the interface. From an experimental view such model interfaces are being prepared quite successfully, however, they have to be understood also theoretically. Therefore the still open C3-position within the department should be devoted to a theoretical chemist being able to investigate these fields e.g. by DFT or molecular modelling in close collaboration with existing experimental groups.

Adhesion and Thin Films
G. Grundmeier

The group was founded in July 2001 by Guido Grundmeier. The activities build a bridge between the aspects of molecular structuring and the development of functional thin films with properties including adhesion promotion, corrosion resistance, tailored surface energy and hardness. The scientific focus of the "Adhesion and Thin Films" group is on the understanding of adhesion and deadhesion at polymer/metal interfaces as well as on the deposition of functional inorganic and organic films with high stability in hostile environments. Thus, thin film engineering on metals and polymer substrates is done to provide model surface and interface structures for fundamental studies and to develop thin films with tailored functionality such as wear resistance or anti-adhesive properties. Thin films are deposited mainly from the gas phase (e.g. plasma enhanced chemical vapour deposition) and from aqueous solutions (colloidal dispersions of inorganic species and organic nanoparticles). The technical focus is on the development of high strength adhesives and thin functional films for engineering materials. A principal approach in all projects is to analyse the technical system via a step-wise simplification of its complexity. Optical spectroscopy and UHV surface analysis techniques are combined with film deposition cells to enable the in-situ analysis of nucleation and growth of the films. In the following

three sections the actual research carried out in 2001 and 2002 is presented with regard to the topics: fundamentals, film formation from aqueous solutions, and film formation from the gas phase.

Fundamentals of adhesion and deadhesion

Adhesive joining becomes a more and more interesting technology for the automotive industry. The use of high strength steels, material combinations or pre-primed metal sheets promote the use of adhesives. High strength joints with excellent initial properties are already available on the market. Still, there is a lack of understanding of the fundamentals of the adhesion forces and the mechanisms that lead to a weakening of the interfacial bonds with time of exposure in corrosive environments. Mainly, the simultaneous mechanical stress and corrosive attack leads to a faster degradation of adhesive joints which is not understood so far. A project entitled *"Long term stability of high performance structural adhesives"* (*K. Wapner, BMBF*) considers the mechanisms of deadhesion of high performance adhesives in corrosive environments in combination with mechanical stress. The idea is that the stretching of organic molecules of the adhesive might lead to an accelerated diffusion of electrolyte into the interface which then leads to an increased oxygen reduction at the delamination front. This hypothesis is competing with the idea that the mechanical force leads to an energy release during the deadhesion which lowers the activation energy for the disbonding reaction. Applied techniques are the scanning Kelvin probe, microscopic techniques, spatially resolved spectroscopic techniques and the blister test. The blister test is combined with the scanning Kelvin probe for delamination measurements under mechanical load. Analytical techniques are applied on suitable model samples. The aim is to form the adhesive joint under very defined and reproducible conditions which are of technical relevance.

For example, by a new sample preparation technique, it is possible to cure thin films of adhesives in a joint and afterwards uncover the joint from one side to enable the application of local electrochemical, spectroscopic and microscopic techniques. In this way, the chemical and morphological structure of the inner adhesive layer could be revealed. For example, nanoscopic voids could be detected at the inner interface when joining was performed above a certain humidity and below a certain pressure (Fig. 3). The influence of such voids on the transport and electrochemical kinetics at interfaces will be studied.

To tailor the interface with layers that withstand the corrosive and mechanical attack metal, surfaces are modified with ultra-thin polymer monolayers (*collaboration with Prof. Rühe, University of Freiburg*), thin plasma polymers, and gradient layers. For iron, it was found that an interfacial gradient layer which inhibits electron transfer reactions and, moreover, forms covalent bonds to the adhesive is necessary to provide optimised adhesion in corrosive environments [12]. To support the electrochemical measurements, spectroscopic techniques are applied to detect interfacial covalent bonds and to locally study the water diffusion in joints. The latter is achieved by the formation of model joints with silicon wafers which are transparent for infrared radiation and thus enable the use of an infrared microscope.

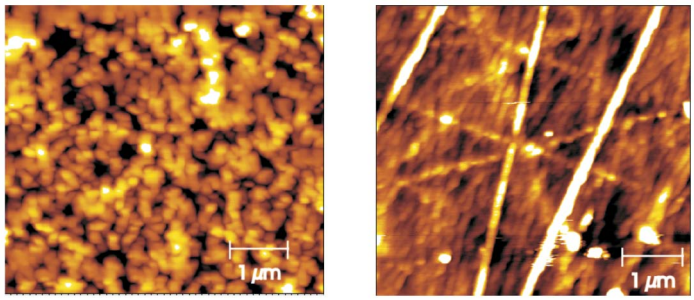


Fig. 3: *AFM topography measurement of the adhesive interfacial surface after removal of the metal substrate of an adhesive/metal joint hardened without mechanical pressure:*

a) Adhesive applied in humid air (Z-range 120 nm)

b) Adhesive applied in dry air (Z-range 35 nm).

Advanced thin inorganic and organic films from aqueous solutions

Conversion layers play a dominant role in providing excellent adhesion and corrosion resistance of polymer/metal interfaces. Processes that are established today have a long history of scientific and empirical development. However, there is a strong need to replace the classical conversion coatings such as phosphatation and chromatation in a short period of time by new environmentally friendly systems. Amorphous thin films deposited from aqueous solutions have a high technological potential since they make use of a smart green chemistry while enabling the further use of established production facilities. In a project called *"New thin conversion coatings on automotive metal sheets"* (*M. Dornbusch*) the formation of amorphous thin inorganic layers is studied on zinc substrates as model systems for galvanised steel.

The project aims at the understanding of the correlation between the composition of the colloidal solution (inorganic species and complexing organic molecules) and the inorganic film formation. The coordination of Zr-species by the complexing organic compounds was observed by means of UV/VIS

spectroscopy and NMR spectroscopy (performed at the University Düsseldorf). A mechanism of the film formation was developed which serves as a basis for the optimisation of the chemistry in the film forming solution. The organic macromolecules complex the inorganic species in solution which would otherwise not be stable at the given pH. By an increase in the pH at the surface induced by the dissolution of the metal the complex is destroyed, the metal ion is released, converted to the metal hydroxide and deposited on the substrate. Due to the simultaneous dissolution of the metal an intermixing layer is formed which leads to excellent adhesion of the conversion coating.

For the corrosion protection of steel constructions such as bridges organic coatings are directly applied to the metal surface without the conversion of the oxide layer. These films have to withstand an extremely corrosive attack. The formation of environmentally friendly latex coatings from aqueous solutions is now studied as an alternative to conventional coatings based on organic solvents in a current project entitled *"Corrosion mechanisms of thin latex films on steel"* (*B. Roßenbeck, BMBF support, in collaboration with BASF and the MPI for Polymer Research*). In the current investigations we focus on the characterisation of thin films and the role of pigments. By the step-wise modification of the iron surface with adsorbed ultra-thin phosphate layers and one to three monolayers of the latex film, the effect of the phosphates on the stability of the interface can be studied. It could be shown by means of electrochemical impedance spectroscopy (EIS) that the phosphate covered surface leads to a significant improvement of the corrosion stability. Model Zn-phosphates will be prepared by the MPI for Polymer Research and model dispersions are prepared by the BASF to tailor the interface.

Plasma polymerisation of thin functional films

Plasma Polymerisation was used as a technology to deposit thin and ultra-thin tailored films on metal substrates. These films can act as corrosion resistant interfacial thin films, ultra-thin adhesion promoters, model polymer coatings for forming operations and ultrahydrophobic aesthetic coatings. Within a project entitled *"Tailored thin plasma polymers for surface engineering of galvanised steel"* (*P. Thiemann, J. Carpentier, V. Barranco, BMBF funding and MPG scholarship*) spectroscopic and microscopic aspects of film formation were studied with the emphasis on fundamental understanding. During the deposition of plasma polymers homogeneous nucleation occurs in the plasma bulk that leads to the formation of plasma polymer nanoparticles which adsorb on the substrate surface. The size of these nanoparticles can be adjusted by the right choice of the plasma process parameters such as the overall pressure and the oxygen partial pressure [13, 14]. The adsorption of nanoparticles leads to voids at the metal/plasma polymer interface and thereby to a decrease in the maximum interfacial contact area. The corrosion protection properties of tailored plasma polymers were investigated by means of EIS. The impedance analysis could be correlated with the morphology of the plasma polymer. Films with voids at the interface led to low barrier properties with a fast increase in the interfacial capacitance due to the incorporation of water at the metal/polymer interface. Thus, the tailored plasma polymer films can be used to study the influence of interfacial defects on the stability. Plasma process parameters can be adjusted so that the void size varies from zero to several ten nanometers.

The highest corrosion protection could be achieved, when gradient layers were deposited where the layer next to the metal is silica like without any interfacial voids and the top layer contains organosilicon groups. The silica layer was produced by an oxygen post plasma treatment. All steps of surface modification were studied by means of surface optical spectroscopy and atomic force microscopy. Optimised layers were tested in cooperation with the steel industry (*Dortmunder Oberflächencentrum, ThyssenKrupp Steel*) and technical conversion layers could be matched in their corrosion protection. Research now focusses on the forming behaviour of the thin plasma polymers on suitable model substrates. In a close collaboration with the group of Dr. Rohwerder the forming aspects of the thin plasma polymers are correlated with their morphology and thickness (*B. Baumert, ECSC funding*).

The wettability of plasma polymer surfaces was studied concerning the chemical composition and topology of the surface. The deposited films were based on organosilanes, fluorinated aliphates and copolymers as well as gradient layers of both. The combined effect of chemical composition and the roughness led to ultra-hydrophobic thin films especially when the nanoscopic roughness of the plasma polymer was superimposed with the microscopic roughness of the substrate electro-galvanised steel (Fig. 4, next page).

New research projects on plasma surface tailoring of metals and polymers

In a new ECSC research project (*L. Yang*) the nanomechanical properties of thin plasma polymers which act as scratch resistant and dirt repelling films on stainless steel and coil coated metals are investigated by means of the nanoindentation technique. A close collaboration will be established with the department of Prof. Raabe with regard to the interpretation and simulation of the results.

In a new BMBF project (*J. Raacke*) pulsed atmospheric cold plasmas are used to tailor the chemistry of polymer surfaces with ultra-thin plasma polymer layers with a high retention of functional groups of the monomers. Here the scientific focus is on the surface reactions during film growth and the correlating density of functional groups on the surface.

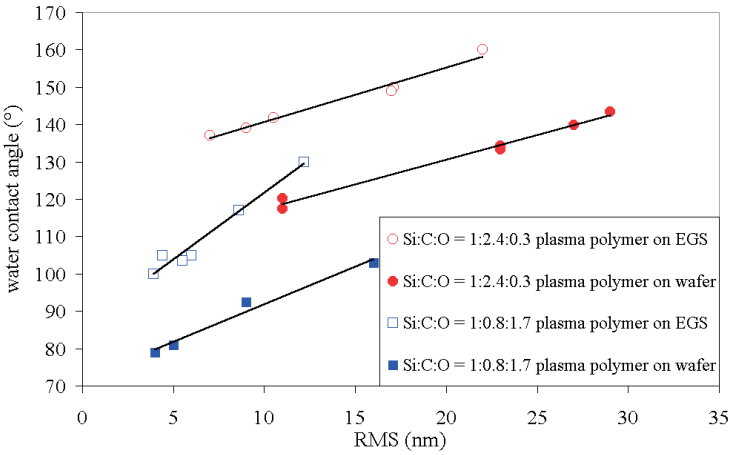


Fig. 4: *Comparison of the static water contact angles on organosilicon plasma polymer surfaces with different chemical composition and surface roughness on polished wafer and electrogalvanised steel (EGS).*

Electrochemistry and Corrosion
A.W. Hassel

The main focus of the „Electrochemistry and Corrosion“ group lies on microelectrochemistry related to tribology and corrosion.

Aqueous corrosion

The use of materials with inherent corrosion protection and repassivaton ability such as weathering steel is cost efficient and environmental friendly [9]. Under environmental conditions they form protective rust layers which hinder further corrosion. The formation of this patina is the result of a complex reaction mechanism being influenced by the compositions of material and environment and by the cyclic climatic conditions (temperature, humidity) during formation. In case of a mechanical destruction, this material will react with the environment to reform the protective layer. The formation of a protective layer fails under marine conditions. In Japan, where the weathering steel is an important structural material for bridges and other constructions new types of low alloyed steels containing 3 % nickel are developed. To prove the higher chloride tolerance (*S. Tsuri, Kawasaki Steel)* compared their behaviour under cyclic corrosion conditions, the natural form of exposition [15]. These investigations where performed in different time domains. For the first cycles (1-10) corrosion potentials and corrosion rates where monitored simultaneously during wet/dry transitions in a special Kelvin probe combined with an oxygen consumption measurement. Midrange tests (10-150 cycles) were performed in a newly constructed dip and dry machine that allows to record potential transients as well as impedance spectra during immersion of the sample. The influence of the chloride content in the wetting electrolyte was investigated in order to simulate the influence of marine environments on the performance of the rust layer formed. In chloride containing environments potential fluctuations where observed during drying which are attributed to pitting corrosion within the scale. Most important is however that a close relation exists between the final corrosion potentials of the dry surface, the time of activation after wetting and the corresponding electrode potentials and the maximum corrosion rate: the higher the electrode potential in the wet stage the smaller the maximum corrosion during drying would be. This was true for all samples.

The mechanism of cathodic delamination of polymers from zinc coated steels is of high industrial relevance. Understanding the rate determining step and how it can be influenced is addressed in a joint project on passive/active transitions in cyclic corrosion tests (*S. Bonk and M. Wicinski, ECSC Partners: Thyssen Krupp Steel, Irsid France, Voest Alpine Linz Austria*). The surface of technical zinc coatings (galvan, galvannealed, hot dip galvanized) is modified by pre-treatments prior to coating with clear coat. The performance of these materials is appraised on the basis of accelerated corrosion tests which include salt spraying and cyclic climatic conditions. Under these corrosion conditions the surface will be alkalised and zinc forms basic chloride containing corrosion products such as simoncolleite which where identified by means of Raman spectroscopy. Scanning Kelvin probe measurements in the active state (wet cycle) show a zone of high potential –300 mV SHE underneath the intact polymer and a much lower potential of –800 mV SHE in the delaminated zone where oxygen reduction takes place. As drying proceeds the potential in the delaminated zone increases indicating the active/passive transition except for a small zone at the delamination front which remains at the low potential. This is attributed to differences in the passive film structure which are determined by pH differences between the head of the delamination front and the delaminated zone behind it. The potential measured after drying reflects a sufficient band bending which limits the rate of electron transfer reactions and this band bending is shifted to more cathodic potentials for a more alkaline pH.

Even though chromate as a corrosion inhibitor is to be replaced for environmental reasons it is still an

excellent object for studies due to its unbeaten performance. The mechanism of filiform corrosion (FFC) and the protective effect of the chromate were studied on Al (*J. Vander Kloet*). Aluminium for use in aircraft industries is susceptible to a thread like local corrosion called filiform corrosion. Second phase particles resulting from alloying elements such as copper are known to play an important role in this special corrosion type which takes place only under specific conditions in the presence of chloride ions and only in a certain range of humidity. Kelvin probe measurements of the active head of filiform threads have shown that anode and cathode are separated [16]. While the anodic metal dissolution takes place in the head of the corrosion thread, the cathodic oxygen reduction is localised in its tail. An extensive SEM, XPS and ToF-SIMS study revealed the fundamental differences between Cr from conversion layers and from pigments in the polymer [17], the latter being by far more effective.

In cooperation with Dr. Rohwerder, SKPFM was successfully applied to this problem. Aluminium model samples with artificial copper dots where coated by an ultrathin polymer. Under FFC conditions SKPFM demonstrated that on the Cu dots a change from more positive to more negative potentials only occurred where the Cu dots were already incorporated into the filament (Fig. 5). These findings show that the Cu dots have no long-range influence on the FFC path.

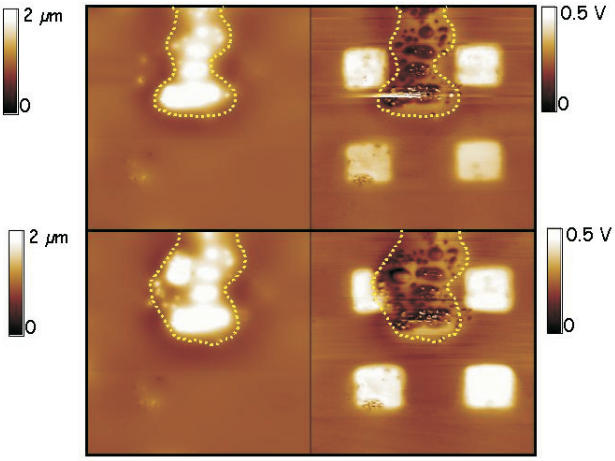


Fig. 5: *The figure shows two successive topography/potential snapshots of an advancing filiform head on an Al model sample, structured with a pattern of evaporated Cu dots in order to simulate inclusions in technical alloys. The scan size of the images is 80×80 µm². Through the thin spin-coated polymer film the Cu dots do not show in the topographical AFM images. In the corresponding potential image they show a several hundreds of mV higher potential than the surrounding matrix. Even if the filiform head touches a Cu dot its potential remains high, only when a part of a Cu dot is incorporated into the head the potential of this part is lowered accordingly.*

Microtribology

Tribocorrosion gained more and more interest in recent years. The disastrous effect results from the synergism of tribologic attack and corrosion. During passivation of aluminium or stainless steel a decent amount of metal is used to form the passive layer. Under tribocorrosive conditions however the continuous removal of the passive film combined with the high dissolution rate of the bare metal leads to an unacceptable mass loss. The two major questions are what is the critical mechanical energy which the passive layer can withstand without formation of a defect and how fast is a defect repaired by repassivation. To answer these questions in the case of *particle induced flow corrosion* it is necessary to separate events from each other in the time domain. For this studies a sophisticated slurry jet was developed during the last 2 years (*E. Akiyama, JSPS*). It is used to study discrete particle impacts on a microelectrode. The experimental challenge to separate impacts of single SiC particles with a diameter of 100 µm was mastered. The high measurement frequency of 1 MHz allows studying the fine structure of each micro transient. After an activation which lasts 50 µs the surface repassivates within 5 ms. In a comparison between pure Al and an Al alloy with 1 % Si, the alloy surprisingly repassivated faster than the pure metal [18]. In the next step the number of impacts will be further decreased to allow experiments with single particle impacts. Subsequent SEM investigations shall link the microtransients to the plastic deformation of the surface.

Microelectrochemistry

Electronic properties of passive films are extremely important for the application of metals. They depend on the formation conditions and for some materials also significantly on the crystallographic orientation of the substrate. Polycrystalline materials show therefore a strong heterogeneity. Microelectrochemical investigations on single grains of a titanium poly crystal where performed to demonstrate that a continuous change of properties can be realised by stepwise anodisation. A pattern of 100 µm anodic oxide spots with thicknesses of 6-16 nm where formed potentiodynamically on a single grain of a titanium polycrystal by means of a scanning droplet cell. Subsequent polarisation to 10 V for 20 min yields a 20 nm oxide film on the entire surface [19]. By means of an imaging ellipsometer it is possible to link the local optical properties to an image of the local electron transfer reactivity demonstrating that the defect concentration and hence a desired reactivity can be realised by a proper chosen anodising procedure. A detailed knowledge of the substrate microstructure namely its grain orientation is necessary. Microstructure and crystal orientations can be most completely determined by automatic

crystal orientation mapping (ACOM) based on automatic evaluation of electron backscatter diffraction (EBSD) patterns in the SEM. These measurements are performed in collaboration with S. Zaefferer (Dept. Microstructure Physics and Metal Forming).

Functional materials with smart properties such as shape memory alloys will drastically gain in importance in the next years. Mechanical micromachining for the production of miniaturised shape memory alloy devices with their exceptional mechanical properties such as superelasticity or pseudoplasticity faces immense problems. The material can reversibly change its form under the mechanic forces during machining or phase transition temperatures can be altered by the strong heating of the surface zone. *Electrochemical micro- and nanomachining* is a promising alternative but faces other difficulties. Nitinol is an intermetallic. During reaction with water nickel dissolves from the surface while the remaining titanium forms a stable oxide layer. The initiation as well as the processing must take these properties into account. A method that uses ultrashort pulses of a few nanoseconds is now being implemented (*K. Fushimi*). To overcome the disadvantage of one dimensional machining a novel type of tool is developed in cooperation with A. Schneider (Dept. Materials Technology). It is based on an intermetallic that is able to form a SONS (self-organized nanostructure) of thin (200 nm) parallel and evenly distributed noble metal fibres.

Microbiologically influenced corrosion

In contrast to corrosion of metals with oxygen (aerobic), corrosion without oxygen (anaerobic) is mainly a microbial process, and sulfate-reducing bacteria play an important role e.g. in oil plants and pipelines. A single genus, *Desulfovibrio* is commonly monitored in industrial plants that are prone to corrosion. All laboratory studies have been carried out with strains of this genus. It is easy to cultivate and shows an effective hydrogen uptake which supports one hypothesized corrosion mechanism. Prof. Widdel and Mrs. Dinh from the Max-Planck-Institute for Marine Microbiology in Bremen successfully applied a non standard isolation approach and separated a novel type/genus of sulfate-reducing bacterium that is more corrosive than *Desulfovibrio*, but that has been overlooked in microbiological monitoring. Kinetic growth experiments support a hypothesized direct electron transfer rather via H_2 from the metal to the attached cells. This shall be confirmed by a direct measurement of the current resulting from the oxidation of iron. Up to now the bacteria were already successfully cultivated on high grade iron electrodes. They appear as fibres together with FeS the main metabolism product on the surface as seen in Fig. 6.

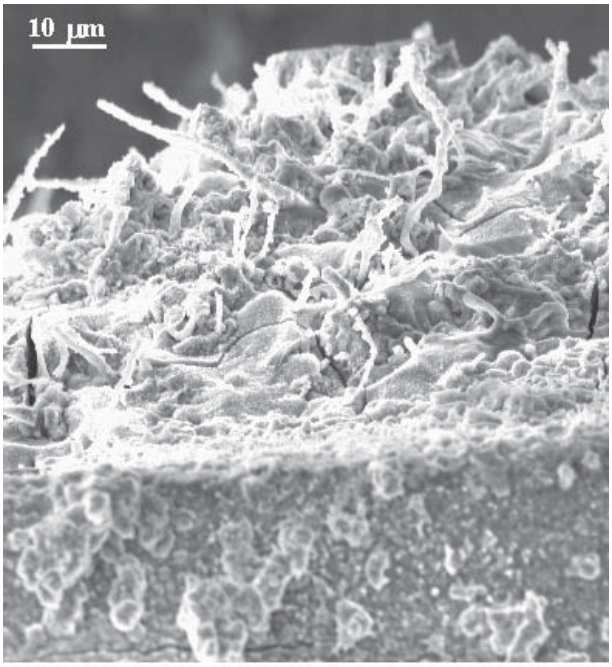


Fig. 6: *SEM image of a new aggressive type of sulfate reducing bacteria (long fibres) on top of their corrosion products. The precipitate consists mainly of FeS and $FeCO_3$ and covers the entire surface.*

Molecular Structure and Surface Modification

M. Rohwerder

The group “Molecular Structure and Surface Modification” has its main focus on the correlation of structure, reactivity and chemical stability of interfaces and surfaces. The projects are organized into the following topics: atomic and molecular structures (nanoscopic aspects of corrosion and delamination at metal/electrolyte resp. metal/polymer interfaces), elementary reactions at buried interfaces, and the effect of mesoscopic structures on these.

The fundamental character of these topics right at the central research interests of the department brings about a strong cooperation with the other groups within the department. Especially regarding the nanoscopic and UHV techniques special experimental set-ups for their application for corrosion and delamination studies are designed and installed, which are of interest for the whole department.

Atomic/molecular structures

Of special importance for the projects on *nanoscopic aspects of corrosion and delamination at metal/electrolyte resp. metal/polymer interfaces* is the ability to apply nanoscopic methods such as

STM and AFM or SKPFM in-situ under the adequate environmental conditions. An electrochemical scanning tunnelling microscope (EC-STM) may allow to perform simple electrochemical experiments in electrolyte, but complex electrochemical sample preparation usually requires the use of more conventional electrochemical equipment. A high-performance glove-box (as low as 1 ppm O_2 partial pressure) will make possible the transfer of samples between different electrochemical set-ups without exposing them to oxygen. Since UHV-based surface characterisation of the prepared samples and also of the samples at intermediate steps of corrosion experiments is of eminent importance, the samples can be transferred via a special UHV-transfer vessel to a complex multi-technique UHV-system, that is currently being built-up. Part of this system will be a variable temperature STM (VT-STM), a small spot ESCA, a ToF-SIMS, a LEED/AES, a plasma chamber, a molecular beam epitaxy (MBE) unit, and a high-temperature oxidation chamber. The set-up, which will be finished at the end of this year, will be used to study elementary reactions during the high-temperature oxidation of binary alloys (*B. Hüning, DFG support*) and during the electrochemical dissolution of single crystal alloys [10] following up research activities, which had to be stopped two years ago because of the reconstruction of the building.

Furthermore reactions at metal/polymer interfaces are of central importance for the research activities of the department. Research over the last years has shown, that de-adhesion reactions are driven by such electrochemical reactions whose rate in turn is determined by the electrode potential of the buried interface. These potentials have been measured by the scanning Kelvin probe, however a fundamental understanding of this technique is limited up to now to the delaminated interface, where usual electrochemical concepts apply. The potential of the intact interface, which seems be quite important for the initial stages of delamination, is not yet understood. In a recent research effort on the *origin of the Volta potential measured on coated metal surfaces* (*H. Ehahoun*), the effect of the molecular structure at inner interfaces on the measured Volta-potential is studied. In order to do so, charged Langmuir-Blodgett (LB) films are transferred to a charged metal surface under well controlled conditions (glove box) and the Volta-potential is measured as a function of the charge density, dipole moment and the interaction of the charged interface with molecular oxygen. The study also should clarify the relation between the charge distribution at the interface and the rate of electron transfer reactions such as the oxygen reduction.

Different LB molecules are being used, thus allowing to modify dipole moments, molecular order and fixed charge at the interface. So far a detailed characterisation of a number of different LB films has been carried out, including the dependence of ion incorporation on the composition of the sub-phase. The interpretation of the results is based largely on the kosmotropic and chaotropic character of the different ions: charged surfactant monolayers at the air/electrolyte interface exhibit high counterion selectivity. The selectivity obeys the Hofmeister ranking observed in biological systems: $ClO_4^- > I^- > Br^- > Cl^-$ and is ruled by the ability of the ions to destroy the ordering of water at the monolayer/subphase interface. The selectivity towards hydroxide ions obeys a more specific rule: surfactants capable of H-bonding exhibit a dramatic preference for OH^- whatever the other competing anion is. During transfer of the films for uncharged monolayers the substrate/electrolyte ionic double-layer is trapped between the substrate and the monolayer. However, a film of neutral subphase is simultaneously trapped by hydrodynamic entrainment, due to the structure incompatibility of the water at the monolayer/electrolyte interface (high order) and at the substrate/electrolyte interface (low order) (subphase entrainment about 10 nm). For charged monolayers no subphase entrainment occurs, due to the destruction of the water ordering at the monolayer/electrolyte interface by the chaotropic perchlorate or halide counterions. In this case the electrochemical double layer seems to get discharged during the transfer.

SKP and SKPFM are of outstanding importance for the investigation of reactions at buried interfaces, the former not only for model systems but also on technical systems under complex climatic conditions [20]. SKPFM, the combination of SKP and AFM, was applied on special model samples in order to study for the first time *sub-microscopic aspects of delamination* (*E. Hornung, X.-W. Yu*) [4, 5]. It could be shown that SKPFM gives indeed comparable information as the SKP. The increased resolution allows to monitor the actual reaction frontier, as could be confirmed by additional high-resolution ToF-SIMS investigations of the ion distribution at the interface: in AFM/SKPFM the electrochemical reaction frontier preceeds the de-adhesion frontier by about 7 μm, while ToF-SIMS shows exactly in this zone a first incorporation of sodium ions. However, the high electric ac fields in SKPFM might cause problems for a quantified interpretation of SKPFM [2]. This is the subject of a project on the *imaging mechanism of SKPFM applied on ultra-thin polymer coatings* (*J. Vander Kloet*). Concerning the model samples, special attention is paid to samples with well-ordered LB-films (*H. Ehahoun*, see above) and well ordered self-assembled thiol films at the interface (*X.-W. Yu*). The electrochemical behaviour of the thiol monolayer, for instance, seems to govern the delamination behaviour of the overall system.

Mesoscopic structures

In the view of the trend to ever thinner alternative surface pre-treatments the electrochemical reactivity of nanoscopic and mesoscopic defects in ultra-thin films will be of great importance. Plasma polymers hold a central position in the departmental research (see "Adhesion and Thin Films", G. Grundmeier). In the project "*Investigation of defect formation and behaviour in plasma polymer films under tensile loading*" (*B. Baumert, ECSC*) the effect of the mesoscopic structure of the metal surface on the formation of the defects has been investigated in detail. The defects can be classified as follows: flaking (i.e. de-adhesion), cutting (e.g. caused by break-through of slip planes) and cracking. The occurrence of these different kinds of defects depends very much on the mesoscopic structure of the substrate and the thickness of the films. For extremely thin films or more elastic films only the latter two are of importance. Interestingly, it was observed that nanoscopic cracks, even though they go through to the substrate, are electrochemically inert (Fig. 7). The effect of the different defects in the film on the delamination velocity is object of the current research. The results of these investigations are used to further optimise the plasma-polymer films (see "Adhesion and Thin Films", G. Grundmeier).

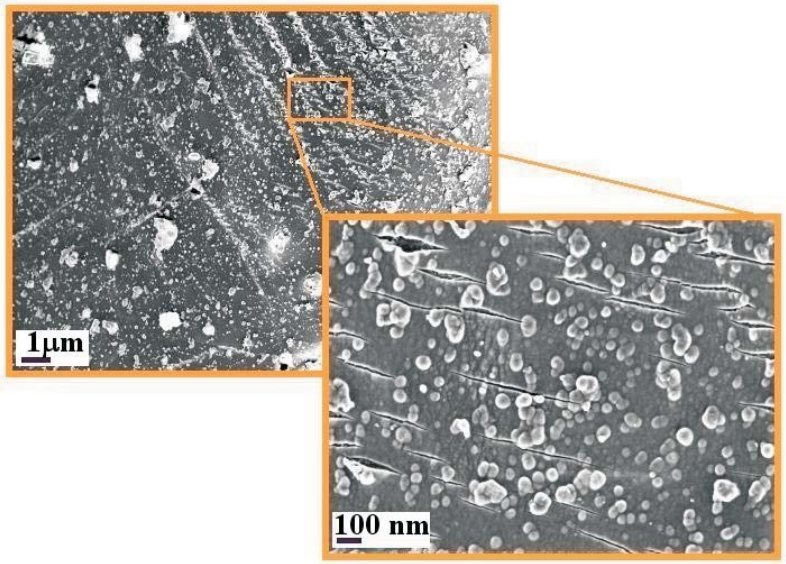


Fig. 7: *9×7 µm² FEG-SEM overview (background) of an electrochemically polished steel sample coated by an ultra-thin plasma-polymer film, after tensile stretching. The slip bands at the surface are clearly discernible. In the zoomed image in the front the nanoscopic cracks on the slip lines are shown in high resolution. These ultra-small cracks proofed to be resistant against copper deposition.*

Another project focuses on the *wetting behaviour of A/B-patterned surfaces and its fundamental aspects of hot dip galvanizing of high strength steels* (*S. Frenznick, ECSC*). One central hypothesis of this project is that the effective surface energy of the submicroscopically metal/oxide-patterned surface is not obtained by averaging the respective values of metal and oxide, but that the line tension at the boundaries plays an important role for the wettability by liquid zinc. The corresponding analysis will be carried out on the basis of contact angle measurements obtained on suitably patterned model samples, as well as the pure oxides and metals as reference. A special galvanising set-up has been constructed which allows the time-resolved study of the galvanising process and the preparation of ultra-thin zinc layers. Thus a high-resolution study of the interfacial reaction zone is made possible, taking into account the effect of the interfacial reaction on the wettability. At the centre of the current research is the search for the most suitable and versatile method for the preparation of patterned metal/oxide surfaces. The most promising candidates in the moment are self-assembled or LB-organized highly-ordered hexagonal close packed layers of Latex spheres, which serve as evaporation masks, colloidal nano-pattering and interferometric lithography. The main problem is that the ordered structure has to be free of defects over about a tenth of a square centimetre at least.

Mesoscopic distributions of submicroscopic salt crystals on the delamination behaviour are at the focus of the new ECSC project "*Salt contamination at the buried interface*" (*E. Müller-Lorenz*). Here, critical size and density for a significant detrimental effect on the interface stability are of interest. SEM, SAM and SKPFM are the most important techniques used in this project.

Buried interfaces

Of central importance for the reactivity at buried interfaces is their electronic structure. Especially, novel zinc alloy coatings are very promising candidates for the preparation of ultra-stable interfaces. For a detailed understanding of the effect of magnesium in novel Mg-containing zinc coatings on the delamination of polymers from such coatings a thorough understanding of the origin of the Volta potential on coated systems is necessary (see also the project by H. Ehahoun). This necessity is one of the main results derived from the project "*Structure of corrosion protective coatings: the Mg-effect in Mg/Zn-alloys*" (*R. Hausbrand, BMBF*). For these alloys the corrosion potential at the defect site is higher than the potential at the intact polymer/alloy interface. This results in a complete inhibition of the fast cathodic delamination process (Fig. 8). Consequently, polymer coatings on these alloys are delaminating extremely slowly. This behaviour of the

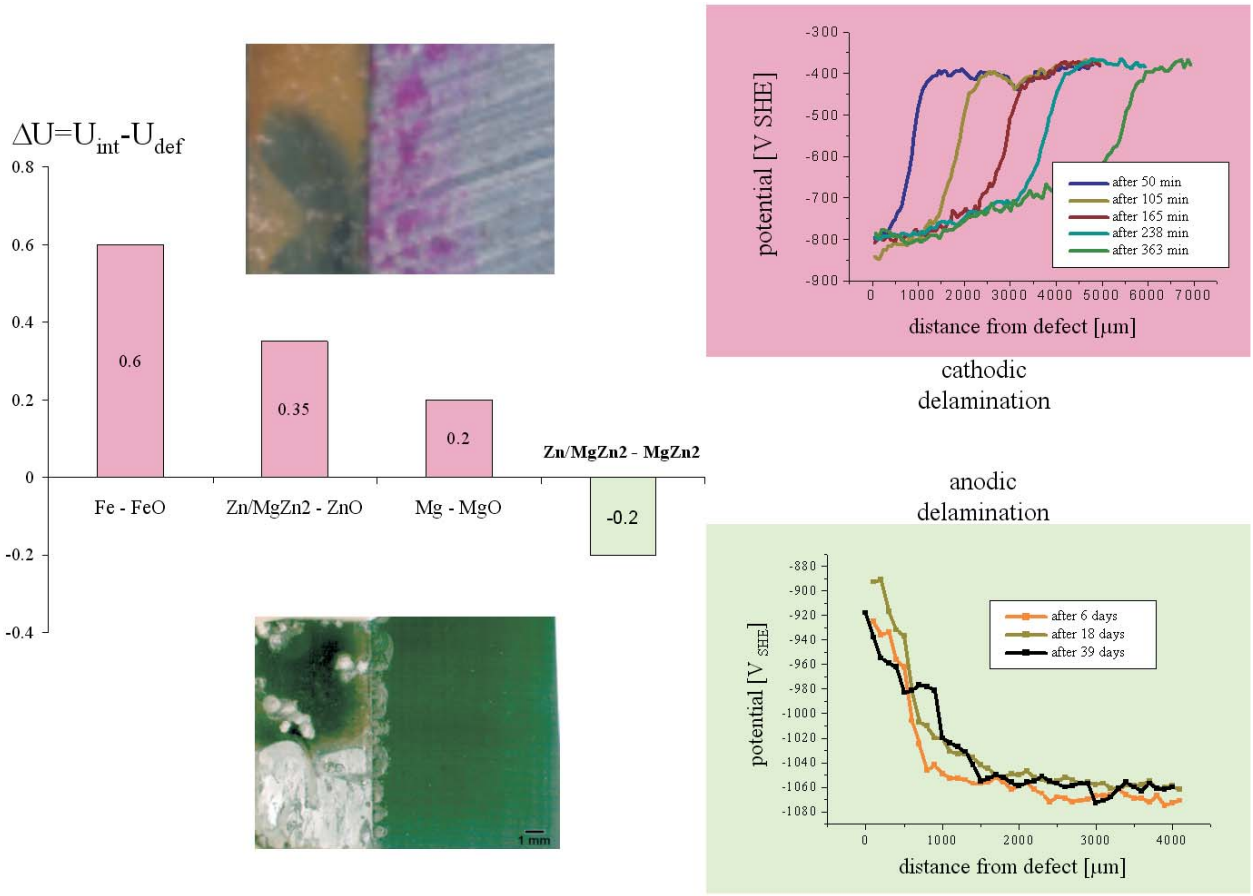


Fig. 8: *Whether fast cathodic delamination is possible or only slow anodic delamination depends on the sign of the difference between the electrode potentials at the intact interface and at the defect site. If it is positive, fast cathodic delamination is possible and will determine the velocity (see delamination curve in the upper right hand corner). Usually, then the delamination proceeds homogeneously (see photo at the top). For a negative sign, as observed for $MgZn_2$, the cathodic delamination is not possible and only a very slow anodic delamination can occur, which looks very inhomogeneous (see lower delamination curve and photo).*

Zn-Mg alloy system, studied on the $MgZn_2$ model alloy, is unique and first of all due to the fact that while the measured electrode potential of the oxide of $MgZn_2$ is situated between the potentials of the pure oxides (Fig. 8), the corrosion potential of the intermetallic phase is not. Instead it is equal to that of pure zinc, which is due to the selective dissolution of Mg [7]. It is expected, that for the optimised coating the difference in potential between defect and intact film should be zero. This requires a suitable adjustment of the potential of the alloy oxide surface. First results on the effect of different pre-treatments for $MgZn_2$ show that the potential can be adjusted within a wide range. This will be correlated to the electronic band structure of the differently prepared oxide scales. That is why a new project on the *properties of novel zinc alloys* has been proposed (RFCS), with a special emphasis on electronic properties of the surface oxides covering these alloys and their effect on the electron transfer reactions (ETR).

But this alone is not sufficient. In most cases the oxides change under high humidity conditions, accompanied in some cases by drastic changes in the potential. This is especially true for oxides prepared under high-temperature conditions (cooperations with Dr. Spiegel). While the values measured directly after oxidation are much too positive, which would promote fast cathodic delamination, the potentials decrease under prolonged exposure to high humidity. This is why a thorough understanding of the correlation of electronic structure, potential and also the stability of the prepared oxides is of ultimate importance for a systematic approach to optimised systems. It is the aim of the new project to provide well defined layers (using the MBE and surface analytics of the UHV complex) and to correlate their structural, compositional and electronic properties with the measured potentials.

Besides modifying the metal side the polymer side of the coated system is also important for improving

the delamination behaviour. In the last few years a lot of attention has been devoted to conducting polymers, although no convincing proof for their superior behaviour has been given yet. On the contrary, the results reported in the literature are very controversial. In most cases it is not clear whether the claimed positive effects are caused by the conducting polymer (CP) or other components of the coating or the surface pre-treatment, which is usually necessary to improve the adhesion. That is why a project was started to investigate the *potential of conducting polymers for corrosion protection* (*A. Michalik, ECSC*) on a more fundamental scale. The approach is to use pure CP coatings or CP pigments in simple matrix polymers (in cooperation with Akzo Nobel) on iron without any pre-treatment. The preparation of such systems, that are stable enough for delamination studies, proved to be very difficult and is still object of ongoing studies. However, for one of the possible working mechanisms of the CPs, the potential delocalisation of the oxygen reduction from the interface to the whole of the polymer thickness, which would decrease the amount of aggressive side products at the interface, first experiments using heavy oxygen O(18) and the ToF-SIMS as analytical tool could be carried out. For a pure polypyrrole/iron model system only a limited delocalisation of the oxygen reduction inside the polymer could be observed. As a next step pigmented systems will be investigated.

Conducting polymers also play on important role in the new project on *release systems for self-healing polymer/metal interfaces* (*G. Paliwoda, BMBF*). The aim of the project is to use the polymer as a reservoir for agents which are able to perform self-repairing activities when delamination sets in. Extensive screening for suitable agents has been performed. The development of these coating systems will be carried out in close cooperation with partners from the TU Dresden, with Chemetall and BASF.

High-Temperature Reactions
M. Spiegel

The "High-Temperature Reactions" group was established in 1.1.2000, proceeding the scientific work on high-temperature corrosion of the former department of Physical Chemistry (Prof. H.J. Grabke). Currently the group has two scientific focuses, one on *surface modification by high-temperature reactions* and the other one on *high-temperature corrosion of metals and alloys.*

Surface modification by high-temperature processes

By metal/gas reactions at high temperatures, solid state reaction products are formed on the metal surface, significantly changing the surface properties with respect to wettability, corrosion resistance and other physical properties like electronic conductivity, etc.

An example for improvement of wettability by metal/gas reactions is the ECSC-funded project "*A mechanistic study of wetting and dewetting during hot dip galvanizing of high strength steels*" (*I. Parezanovic*). During annealing of HSS-steels, surface oxides are formed which have to be removed in a reduction annealing step (5 % H_2-N_2, dew point -30 °C, 820 °C) before galvanizing. However, oxides of alloying elements (Si, Mn, Al, Cr) are not reduced (Fig. 9) and failures in the zinc coating by bad

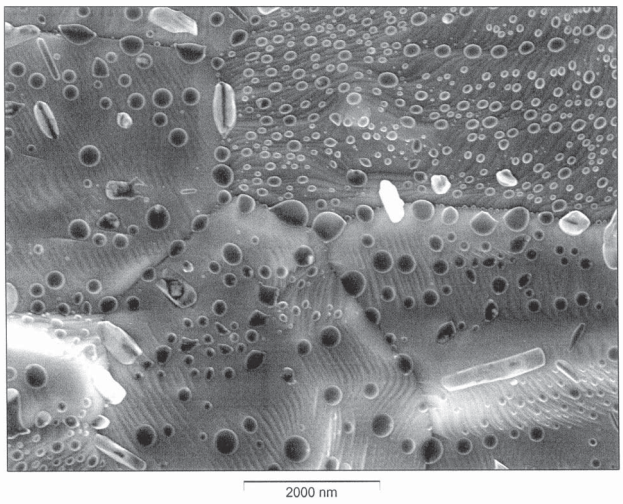


Fig. 9: *Oxide particles (globular, needle shaped) on THM 220 after reduction annealing in N_2-5 % H_2 at a dew point of -30 °C.*

wettability of the surface oxides occur. The technical aim of this work is to improve the wettability of HSS surfaces by improving the reduction annealing conditions. This can be done by changing the H_2/H_2O ratio and, thereby, the oxygen potential. For example, laboratory annealing of DP 500 and Ti-IF steels in the 5 % H_2-N_2 atmosphere with different dew points of -60 and -30 °C at 820 °C for 60 s show that on DP 500 at a dew point of -60 °C BN is formed and the selective oxidation of Mn, Si, and Al is suppressed. Under the same conditions the Ti-IF steel surface is covered with an externally formed Al oxide. At a dew point of -30 °C Mn- and Cr-oxides are detected on the surface of DP 500 and Fe- and Mn-oxides on

the Ti-IF steel. Hence, annealing at low dew point conditions (-60 °C) of both steels is inconvenient because BN and also Al-oxide show bad wettability by zinc.

Scientific aims of the project are:

a) Fundamentals of oxide particle formation: As oxidation of the samples occurs in air, it is non-selective and mainly iron-oxide is formed. At the metal/scale interface, however, oxides of the less noble elements Si, Al, Cr, and Mn are thermodynamically stable. On preoxidized Fe-5% Si model alloys it was found that the iron-oxide is removed after reduction and more Si-rich oxides remain on the surface. It is not clear up to know, whether this oxides are already formed during oxidation or during the reduction treatment only. Oxide particles on the surface of a preoxidized and subsequently reduced Fe-5%Si model alloy are different in shape and chemistry from those only formed in the reduction treatment.

b) Investigation on external/internal oxidation: By varying the H_2/H_2O ratio the oxidation of the less noble elements during reduction can probably be shifted from external to internal, depending on the diffusion coefficient and the concentration on the element to be investigated. Internal oxidation would result in a more reactive surface for the galvanizing process. The oxidation/reduction work will be carried out on model alloys containing Mn, Al, Cr.

c) Interfacial reaction between surface oxides and liquid zinc: Supplementary to the thermal treatments, experiments on the wetting behaviour and the interfacial reaction of the oxides with liquid zinc, applied by the spin coater will be carried out. For this purpose, a zinc droplet is set on the sample surface, subsequently removed by spinning the sample after different reaction times. The surface will then be investigated with surface analytical techniques.

An example for surface modification with respect to electronic conductivity is the development of stainless steels which form slow growing corrosion layers during service with good electronic conductivity necessary for electron transfer within the fuel cell (*I. Parezanovic, N. Sämann, E. Strauch, ECSC support*) [21, 22]. This can be achieved by alloys based on Fe-17% Cr containing Mn, Co, and Mo as multivalent elements forming spinel layers $(Fe^{2+},Co^{2+},Mn^{2+})(Fe^{3+},Cr^{3+},Mn^{3+})_2O_4$ with a good electronic conductivity by electron transfer between the multivalent elements. Laboratory corrosion tests and in-situ conductivity measurements at 650 °C in synthetic air containing 15 vol.% CO_2 in contact with the Li_2CO_3-K_2CO_3 eutectic mixture have revealed that spinel layers containing Mn and Co are formed, as expected. The Ohmic resistivity of all spinel forming alloys was lower than of high Cr steels, especially the addition of 9 wt.% Co was beneficial (80 $m\Omega/cm^2$). In order to optimize the currently used 1.4404 stainless steel, further resistivity measurements are conducted by varying the amount of the elements Mn and Co.

In close collaboration with the working group "Molecular Structures" of Dr. Rohwerder the mechanisms of formation of thermally grown surface passivating oxide films on Zn-Mg alloys are studied (*Y.S. Li)*.The characterization of the thermally grown surface layer is one of the key issues of this part of the project. Up to now, the air oxidation behaviour of an $MgZn_2$ compound by a high frequency induction furnace at 200-300 °C with different exposure duration was studied. The results indicate that both oxides of Zn and Mg can be nucleated at the initial stage and thus form an oxide mixture of Zn and Mg. With increased temperatures or after longer oxidation time an enrichment of Mg in the surface layer dominants the oxidation process. Especially at 300 °C, the surface scale even consists exclusively of MgO. This is explained by a sufficient supply of towards-migrated Mg atoms with which oxygen reacts preferentially in comparison to Zn, in view of the highly negative stand free energy of formation of MgO. In addition, Mg atoms diffusing toward the surface may also convert initially formed nuclei of ZnO to MgO by virtue of the displacement reactions.

Basic research in the area of surface modification by oxidation is done in the MPG and Uni-Potsdam-funded project "*In-situ measurements on growing oxide scales with synchrotron radiation*" (*D. Vogel*). The work is supplementary to the basic STM/AFM studies in the working group "Molecular Structures" of Dr. Rohwerder on atomistic processes of alloy oxidation in hot gases, where the very early stages of alloy oxidation (oxide nucleation) are studied. The focus of this project are studies on basic solid-state reactions within the oxide scale and at the internal metal/oxide interface after continuous scale formation. X-ray reflectometry is sensitive to electron density gradients within layered systems on a nm scale and, therefore, provides information about diffusion and scale forming processes at internal interfaces. Such reaction occur for example by oxidation of Fe-Cr-alloys after a continuous outer Fe-rich scale has formed. With time, chromium will diffuse from the alloy into the external scale and a second layers is formed underneath. In combination with structural investigations by grazing incidence X-ray diffraction it is possible to follow morphological and structural changes in the scale during heating.

For the synchrotron measurements, a heating furnace was installed at the EDR (energy-dispersive reflectometry) beamline of the University of Potsdam and the MPI für Kolloid- und Grenzflächenforschung. First experiments were conducted on pure iron,

because this is easily oxidized, even at lower temperatures. The time resolved (5 min) isothermal and temperature dependent spectra were recorded during oxidation up to 300 °C. Reflectometry spectra could be recorded from the sample surface and the thickness of the resulting layer was measured to 5 nm (*D. Vogel, W. Leitenberger*). Furthermore, additional dynamic changes in the spectra show that next to scale growth also density changes occur in the scale during heating, which have to be investigated in detail. This first experiments show that, in principle, it is possible to acquire short time resolved information on the scale thickness, density and structure upon heating. Further work on pure iron will include the analysis of the scale structure by grazing incidence diffraction. The improvement of the furnace for different atmospheres and studies on simple alloy systems like Fe-Cr are envisaged.

High-temperature corrosion of metals and alloys

The focus of this topic is to understand corrosion processes in energy producing systems such as waste- and biomass fired power plants as well as high-temperature fuel cells [21, 22]. This includes corrosion by hot flue gases (CO_2, HCl, SO_2,..) and molten ash deposits [23-25]. Special emphasis is paid to localized corrosion reactions like selective metal dissolution in molten salts, reaction of oxide scales with salt particles and in the presence of HCl-gas. One new topic is the thermodynamic and kinetic modelling of high-temperature processes on the basis of laboratory data. Furthermore, alloy development and testing for the steel and power industry is also an important aspect [26].

The positive influence of the alloying element like Cr and Al is well established in high-temperature gas phase corrosion. The aim of the MPG-funded project "*High-temperature corrosion of Al-containing alloys and intermetallics beneath molten KCl-$ZnCl_2$*" (*Y.S. Li, M. Spiegel*) is to evaluate the influence of the alloying element Al beneath molten KCl-$ZnCl_2$. Surprisingly it was found that Al is selectively dissolved in the salt melt as very stable $AlCl_3$, reducing the $ZnCl_2$ component of the melt to metallic zinc. Nickel-aluminides behave rather stable because nickel is enriched in the metal and porous nickel-matrix remains after dissolution of Al. Iron-aluminides behave different and iron is also dissolved in the melt next to aluminium. A reaction mechanism for nickel- and iron-aluminides is developed considering the kinetics of Al-dissolution, $ZnCl_2$ reduction in the melt and enrichment of nickel in the alloy.

The EC-funded project "*Optimization of in-service performance of boiler steels by modelling high-temperature corrosion*" focuses on mechanisms and modelling of high-temperature corrosion processes in waste combustion power plants (*A. Ruh*). For the computer simulations, kinetic data on salt melt induced corrosion are collected from experimental investigations and phase diagrams are constructed with the computer program FACTSAGE on salt-metal-gas systems and compared with the experimental results. Up to now, the kinetics of salt melt corrosion of pure iron was investigated and can be described by 3 rate laws, a complex incubation period, a linear period and a logarithmic rate law. The different rate laws are attributed to the different reaction steps melt formation and iron dissolution (incubation time) and oxide particle precipitation (linear region) and surface coverage (logarithmic region). By reducing the oxygen partial pressure, the time period for the incubation period increases and the linear rate constant decreases. It is anticipated to describe the complex reaction product with the use of phase diagrams. Further work is planned on Cr, Ni and binary alloys as well as on the influence of gas phase composition on the reaction kinetics.

More mechanistic studies on corrosion are carried out in another EC-funded project "*Mitigation of formation of chlorine rich deposits affecting superheater corrosion und co-combustion conditions*" was started on corrosion mechanism in biofuel power plants (*S. Cha*). The focus is on studies of localized high-temperature reactions between solid chlorides particles NaCl and KCl, the metallic substrates and preformed passivating oxide scales. This reactions will lead to local failures of the passivating oxide and more harmful gases like HCl and chlorine can penetrate the scale, leading to chloride formation at the metal/scale interface. For this studies, small salt particles are deposited on metallic and preoxidized substrates by thermo-phoretic deposition. Work has started on temperature dependent in-situ FE-SEM investigations in a heating chamber with salt covered samples.

Linked to this project *thermogravimetric investigations on the corrosion of Fe, Ni and Cr beneath solid KCl-NaCl* are carried out (*M. Sanchez, CONACYT*). The main focus in this project is on alloy induced melt formation by gas-salt-alloy interactions at 320 °C. If, for example HCl is present in the gas atmosphere the alloy components are reacting to metal-chlorides, forming low melting metal-chloride-alkali-chloride eutectics on the metal surface. The kinetics of melt formation is then strongly depending on the partial pressures of HCl and other oxidizing gas compounds. Work has started with base metals up to now, simple alloy systems will be investigated further.

Physical Chemistry
H.J. Grabke, Prof. emeritus

Many studies started in the former department Physical Chemistry were finished in the years 2001/2002. One important topic was the *improvement of steels for use at elevated temperatures in aggressive environments*, which was studied in an ECSC project, together with French, Spanish, and Swedish partners [27, 28]. By varying the Mn, Ti, Nb, Si, and Al contents and by coldworking or surface treatments, the high-temperature corrosion resistance of 18Cr-10Ni steels was improved, as confirmed by tests in carburising "metal dusting" conditions and oxidising and chloridising "waste incineration" conditions (*E. Müller-Lorenz, N. Sämann*).The surface working, by grinding or sandblasting, introduces fast diffusion paths, i.e. dislocations and grain boundaries in the steel surface, and this accelerates Cr-diffusion and favours the formation of a protective oxide scale. The initial Cr-oxide formation and Cr-diffusion was studied in detail (*C. Piehl*) by oxidation of six 9-20%Cr steels at 600 °C in three atmospheres with different oxidation potentials. The rapid growth of a Cr-rich protective scale was clearly favoured with enhanced surface deformation. Concentration profiles in and beneath the scale were recorded by secondary neutrals mass spectrometry (SNMS). Non-deformed steels show a deep Cr-depletion in a narrow region beneath the scale, high deformation, i.e. many fast diffusion paths lead to a shallow Cr-depletion, extending far into the interior. From the Cr-profiles, bulk diffusivities and grain boundary diffusivities were obtained for the electropolished and polished specimens, whereas for the strongly deformed, sandblasted specimens effective diffusivities result, increasing with deformation to values corresponding to grain boundary diffusion [29-31]. Surface working is a good remedy against *metal dusting of steels and Ni-base alloys*, i.e. a disintegration into graphite and metal particles occurring in carburising atmospheres, such as synthesis and reduction gas ($CO-H_2$) at $a_C > 1$ and elevated temperatures about 400-800 °C. Sulfur from the atmosphere is an additional remedy suppressing metal dusting on steels, by stabilizing cementite, the intermediate of the metal dusting mechanism on iron and steels. The *role of sulfur in carburisation, carbide growth, metal dusting and coking* has been studied in depth [32-34], also by LEED and AES (*D. Moszynski*). In many chemical processes, sulfur cannot be added due to possible poisoning of catalysts, and the most metal dusting resistant alloys have to be used, i.e. Ni-base alloys with >25% Cr and 2-3% Al. However, it was shown now (*E. Müller-Lorenz*) that weldments and also etched samples of these materials are somewhat susceptible to metal dusting.

Most interesting studies of *metal dusting mechanisms on iron*, which change with temperature and gas composition are continued in cooperation with other departments of the institute (*G. Inden, A. Schneider, J. Zhang*). On Fe-Ni alloys, where the mechanism changes with alloy composition, new results are obtained in cooperation with the MPI für Mikrostrukturphysik (*J. Woltersdorf, E. Pippel*).

Reoxidation of direct reduced iron (DRI) can lead to ignition, and thus endangers depots and transports of DRI. To study the kinetics of reoxidation and ignition a joint ECSC project (with RWTH Aachen, VDEh and CRM Liège) was finished [35]. At the MPI a way for protection of DRI was conceived and confirmed – presence of vapour phase inhibitors, e.g. organic amino- and ammonium compounds, retards and suppresses reoxidation and in this way also ignition of DRI [36].

Hydrogen in steels was a long-term topic in the department. A study on the *role of hydrogen in hot-rolled steel strip on zinc coating* was conducted in cooperation with CRM Liège and TKS Dortmund [37]. Hydrogen uptake during pickling and its desorption during annealing was investigated for several steels (*T. Schlüter*). A review paper on *hydrogen in micro-alloyed steels*, summarizing many years of research using the electrochemical permeation method on H-permeation, solubility, diffusion and trapping was written up [38]. This description of the interaction of H with precipitates (TiC, TiN, Mo_2C, VC, VN, NbC, NbN) in such steels and its effects on mechanical properties was honoured with the "Best Paper Award 2001" of the journal 'steel research'.

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Department of Materials Technology

G. Frommeyer

Scientific Concepts

The scientific and technological research activities in the department are focused on the following four major subjects:

Innovative Steel Research

Rapid Solidification Technology

Development and Characterisation of New Materials

Intermetallic High-Temperature Alloys

One important aim is the development and characterization of new high performance steels with improved mechanical properties, like higher strength, excellent formability and lower density, for advanced design concepts of modern transportation systems, architecture and engineering structures. Besides this specific physical properties, such as electrical resistivity, hard or soft magnetic behaviour of high grade electric steels or iron based alloys used for transformers, electric motors and generators etc. are considered.

Innovative structural steel concepts are based on iron-aluminum -chromium and/or iron-manganese-aluminum-silicon systems. Medium or high-alloy steels possess ferritic, duplex, or fully austenitic microstructures. Metastable austenitic-ferritic steels exhibit extraordinary deformation and strengthening behavior by multiple *Transfomation-Induced Plasticity* (m-TRIP) effect. Highly stabilized austenitic steels with defined stacking fault (SF) energy show supraductility by *Twinning-Induced Plasticity* (TWIP) effect. High manganese or cobalt containing austenitic TWIP steels reveal extensive mechanical twinning resulting in moderate strength, supraductility and extremly high energy absorption under impact loading. The metastable TRIP steels exhibit high work hardening rate and tensile strength. The ongoing research is focused on structural characterization and on investigations of deformation and fracture mechanisms of these classes of novel steels.

The main advantages of the newly developed ferritic iron-aluminium (chromium) steels are the reduction of specific weight by about 10 %, caused by Aluminium and its effect on dilatation. These light-weight steels exhibit increased tensile strength caused by effective solid solution hardening of the b.c.c. iron due to dissolved aluminium in higher concentration and sufficient ductility in comparison with conventional deep drawing steels. The corrosion resistance is remarkably improved by addition of chromium. Micro alloying elements strongly affect grain growth and recrystallisation texture, enhance deep drawing and stretch forming properties.

A long term research work deals with the analysis of the deformation mechanisms of high-strain-rate superplasticity in microcrystalline high-carbon-aluminum steels of superior strength properties. These steels contain large volume fractions of κ-carbides, cementite or borides finely dispersed throughout the fine grained ferritic matrix.

Duplex or super duplex stainless steels consist of fine grained α/γ-microstructures which are the prerequisite for superplasticity at medium or higher deformation temperature. The governing superplastic deformation mechanisms are grain boundary sliding and diffusional accommodation at the triple point of the grains. Superplastic ultra-high strength steels are suitable for near net shape precision forming of parts with complex geometries using blow or die forming operations.

The steel research in the Department of Materials Technology is partly funded by the government: Bundesministerium für Bildung und Forschung (BMBF) and Bundesministerium für Wirtschaft und Technologie (BMWI), and by the Deutsche Forschungsgemeinschaft (DFG).

Another subject of the research activity in the department deals with rapid solidification and related RSR technologies. Rapid cooling of liquid metals and steels leads to a quasi partitionless solidification and refined microstructures due to the occurrence of highly undercooled and rapidly solidifying melts. In combination with near net shape and continuous casting processes, the economic production of thin slabs, sheets, ribbons or foils, wires and fibres of high quality standard with extraordinary physical and mechanical properties will be realized. These advantages also pertain to the technologies of melt atomisation and spray casting for the fabrication of advanced PM steels and superalloys.

Considerable achievements have been realized in research and application of rapid solidification technologies and continuous casting. The ongoing research activities at MPIE are focused on the process analysis and modelling of rapid solidification within the chill zones of innovative casting technologies, such as planar flow casting, shape flow casting as well as laser welding processes. The difficult access to experimental data of rapid solidification and process parameters is concurring with the substantial need of them to understand and describe these processes quantitatively. As consequence, the development of powerful macroscopic solidification models are highly required.

Another objective is to improve existing and to develop new models - generalized enthalpy and multi phase method - accompanied with suitable investigations on microstructures and physical (including thermophysical) properties of rapidly solidified materials.

Another focus is pointed to directional solidification which takes partly place in rapid solidification. The second term of interest is the synthesis of micro- or nanocrystalline materials with superior properties, such as the enhancement of ductility by nano-size ordered domains, exceptional hard or soft ferro magnetisms and superplasticity.

A currently running project is the modelling of solidification and microstructures at the interface of laser welded seams, funded by the DFG.

Advanced generations of highly efficient energy conversion systems, such as stationary gas turbines of power plants, internal combustion and jet engines (instationary gas turbines) with reduced emission of exhaust gases require high performance materials of low density exhibiting excellent high-temperature creep strength and hot gas corrosion resistance.

Modified ordered alloys with superlattice structures and intermetallic compounds like iron aluminides Fe_3Al(Cr, Mo, V, Nb), nickel aluminides NiAl(Cr, Mo, Re), and titamium aluminides γ-TiAl/α_2-Ti_3Al(Cr, Mo, Cu, Si) fulfill the technical requirements.

The research work performed in joint projects with the department of Physical Metallurgy, Interface Chemistry and Surface Engineering, Microstructure Physics and Metal Forming is a major objective. Alloy development, and the characterization of physical and mechanical properties in correlation with microstructural changes and specific deformation mechanisms is the important task of these challenging joint projects, supported by the BMBF, DFG and various industrial companies.

Innovative Steel Research

N.N., G. Frommeyer

Development and characterisation of supra-ductile and high-strength manganese-TRIP/TWIP steels for high energy absorption purposes

An important aspect, concerning new design concepts for advanced light-weight and crash resistant automotive components is the development of high strength and supra-ductile steels possessing enhanced energy absorption and reduced specific weight .

High-manganese steels containing 15 to 25 mass% Mn and additions of silicon and aluminum of about 2 to 4 mass% exhibit high strength and exceptional plasticity due to extensive twin formation under mechanical load (TWIP effect: Twinning Induced Plasticity) or via multiple martensitic transformations (TRIP effect: Transformation Induced Plasticity) [1, 2].

The TRIP and TWIP effects promote specific mechanical properties. The high-manganese TRIP steel exhibits a pronounced strain-hardening behaviour with maximum stress exponents of n = 0.8, high tensile strength of about 1100 MPa, and improved elongations to failure of ε_{tot} = 55 %, respectively.

The TWIP steel shows a relatively low flow stress of $R_{p0.2}$ = 280 MPa and a moderate tensile strength of 650 MPa. A extremely high elongation to failure of ε_{tot} = 95 % is achieved, even at high deformation rates.

The bar diagram (Fig. 1) represents the specific energy absorption $E_{spec.}$ of the TWIP steel in comparison with selected conventional deep drawing steels, such as IF-steels (FeP04), bake hardening steels (Z St E 180 BH) and thermomechanically

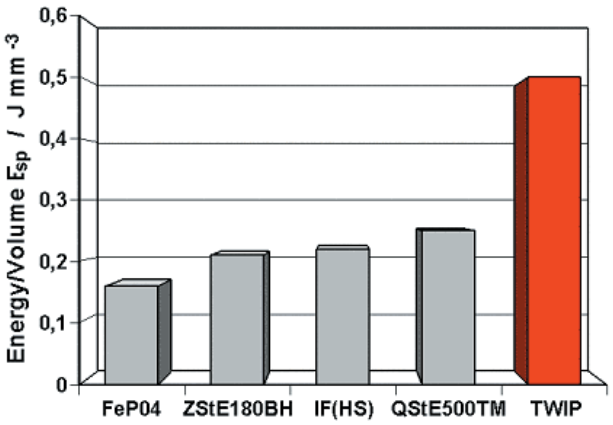

Fig. 1: *Specific energy absorption $E_{spec.}$ values of conventional deep drawing steels in comparison with the Fe-25Mn-3Al-3Si mass% TWIP steel; test temperature: 20 °C, strain rate: $10^2 s^{-1}$.*

processed steels (Q St E 500 TM), respectively. It is shown in the diagram that the specific energy absorption value of the TWIP steel is about 0.5 J/mm^3 and the conventional deep drawing steel qualities possess energy absorption values between $0.16 \leq E_{spec} \leq 0.25$ J/mm^3, which are half of that of the very crash resistant supra-ductile TWIP steel or even less.

Another property, which characterizes the impact behavior of deep drawing steels for automotive bodies and frame structures is the temperature dependent charpy impact toughness. In charpy impact tests the TWIP steel behaves completely ductile, even at low temperatures (-196 °C) [3].

Investigations on the deep drawing behaviour of microalloyed iron-aluminium light-weight steels

In consideration of fuel saving and the decrease of exhaust gases the development of innovative car body concepts requires a substantial weight reduction and the application of high strength sheet steels in combination with light-weight motor components.

The first priority for developing iron aluminium based light-weight steels is the reduction in specific weight, which is associated with a very effective solid solution strengthening and sufficient ductility. However, at higher aluminium contents of more than 7 mass% Al preordering phenomena and long range ordering are occurring, which promote detrimental effects on the formability. These structural features are restricting the application of iron aluminium alloys as deep drawing quality steels to about 6.5 mass% Al. According to this Al content a reduction in density of approximately 8 % (from 7.87 to 7.25 g/cm^3) will be achieved [4].

The tensile ductility of iron aluminium solid solution is also influenced by small amounts of carbon due to the formation of κ-carbide precipitates on grain boundaries. At higher carbon contents cleavage fracture mode partly emerges in impact tests at room and lower temperature using charpy V notched test bars.

In order to avoid the ductility reducing influence of carbon in iron aluminium solid solutions its carbon content must be kept as low as possible, e.g. less than 100 ppm. The excess carbon has to be reduced by adding strong carbide forming microalloying elements, such as titanium and niobium to the iron aluminium steels.

Moreover microalloying elements are influencing the recrystallization texture due to their selective effects on nucleus formation and grain growth of the recrystallizing samples [5-7]. Microalloying elements have a strong effect on the deep drawing properties of Fe-Al-steel sheets, which is illustrated by forming limit curves. An adequate addition of Ti, Nb and B remarkably improves the deep drawing and stretch forming properties.

The characterisation and optimisation of high-strength light-weight steels are current objectives of diverse research projects in cooperation with the steel and automotive industry, supported by the BMBF, BMWI, and DFG (*U. Brüx, K. Brokmeier*).

Physical and mechanical properties of high strength steel

The physical and mechanical properties in correlation with the martensitic and age-hardened microstructure of ultra high-strength maraging steels are under investigation.

In order to achieve the ultra high-strength state via martensite transformation with subsequent age hardening the maraging steel (X1 Ni Co Ti16 10) was heat treated – solution annealed and austenitized at 830 °C for 180 min with subsequent quenching in ice water below the Ms temperature. The transformed martensite is of massive morphology possessing high dislocation densities of >10^{14} dislocations/cm^3. The orientation relationship is:

$$(111)_\gamma \,||\, (011)_\alpha \text{ and } [\bar{1}\,\bar{1}\,0]_\alpha \,||\, [\bar{1}\,\bar{1}\,1]_\gamma.$$

The observed platelet interfaces are rather wavy and the habit plane was indexed to $[0.46; 0.53; 0.708]_\gamma$ tilted about 10° away from $(111)_\gamma$.

The age-hardening treatment was performed at 525 °C/120 min in order to achieve the appropriate hardness of 49 to 50 HRc by precipitation reaction of η-Ni_3Ti. However. in lower nickel content maraging steels γ'-Ni_3Ti precipitates have been detected by X-ray analysis and by electron diffraction in the TEM.

The elastic modulus of the investigated steel is in the age-hardened state of about 195 GPa. The internal stresses cause an increase of about $\frac{\Delta E}{E} \approx 0{,}04$ compared with the soft annealed and stress relieved state.

The linear thermal expansion coefficient is of the order of $\alpha_{RT} = 10.5 \cdot 10^{-6}$ K^{-1}. The achieved high yield stress of $R_{p0.2} = 1575$ MPa and ultimate tensile strength of about $1650 \leq R_m \leq 1850$ MPa is accompanied with high fracture toughness - stress intensity factor of $85 \leq K_{IC} \leq 110$ MPa $\cdot$ m$^{1/2}$.

Creep deformation of the nickel-cobalt-maraging steel type alloyed with small amounts of titanium showed in the temperature regime from 650 to 750 ºC stress exponents of $4 \leq n \leq 5$ which indicates dislocation creep as the dominant deformation mechanism.

The creep strength at the strain rate of 10^{-7} s^{-1} is about 100 MPa (test temperature 700 ºC). The deter-

mined activation energies yields Q = 175 ± 5 kJ/mol which indicates grain boundary diffusion of iron and solutes (Ni, Co) controlled dislocation creep.

An interesting feature is that the ultra high strength maraging steel exhibits severe shear band formation under impact loading at strain rates of $\dot{\varepsilon} = 10^3\ s^{-1}$, represented in the TEM image (Fig. 2).

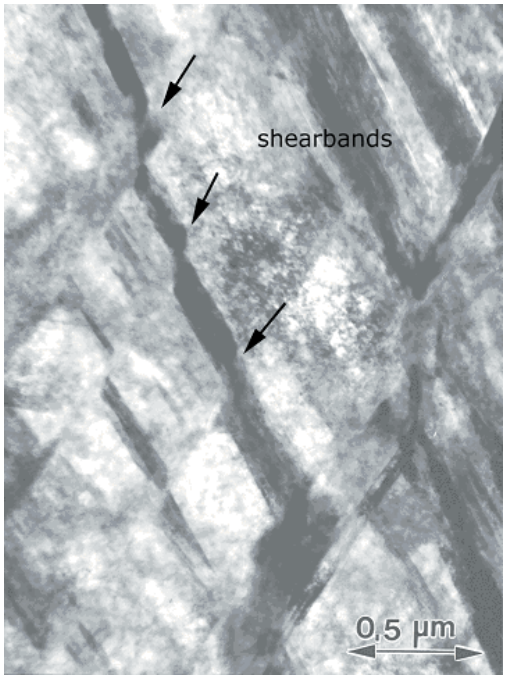

Fig. 2: *TEM bright field image showing extensive shear band formation in the microstructure of the deformed maraging steel (strain rate: $\dot{\varepsilon} = 10^3\ s^{-1}$, T = 20°C, plastic strain: $\varepsilon_p = 8$ %.*

Creep and superplasticity in duplex stainless steels

The high temperature deformation behavior of the modified micro duplex stainless steel IN 744 has been studied in tension tests at temperatures ranging from 850 to 1100 °C. The microstructure of the starting material consists of elongated austenitic grains in a ferrite matrix. Microstructural analysis of the deformed samples showed that the material recrystallizes during heating at the test temperature, and the austenite transforms into fine colonies of grains of 10-15 µm in size. The results of the mechanical tests in the temperature range 850-1100 °C show a high elongation to failure (up to 300 %) and a low value of *n* (2 to 3) for strain rates up to $10^{-3}\ s^{-1}$. This result suggests that the controlling deformation mechanism is grain boundary sliding, GBS. Microstructure and texture evolution studies of deformed samples confirmed the importance of GBS. The colonies of austenitic grains decrease in size during superplastic deformation and minor changes in the texture was observed in the deformed region. At high strain rates an increase of the stress exponent to 5 is recorded, which suggests the transition to slip-creep [8].

Rapid Solidification Technology

J. Gnauk

The ongoing research activities at MPIE are focused on the process analysis and modelling of rapid solidification within the chill zones of innovative casting technologies and laser welding processes. The development of powerful macroscopic solidification models are highly required due to deficient experimental data of rapid solidification inclusive process parameters. Existing conventional and newly developed models will be confirmed and refined by experimental results obtained in suitable experimental investigations on microstructures and related physical and thermophysical properties of rapidly solidified materials.

Another focus is pointed at directional solidification which explains and rounds out a process step during rapid solidification. In addition the synthesis of micro- or nanocrystalline materials with superior properties, such as the enhancement of ductility by nano-size ordered domains, hard and soft magnetisms, superplasticity, etc. performed by high cooling rates is term of interest.

For improving the surface quality of continuously cast steel products it is important to understand the (rapid) solidification at the very initial stages of the casting process exactly. In the project “On-line early solidification control in the continuous casting of peritectic steel grades”, initiated by the “Centro Sviluppo Materiali” (CSM), Rome/Italy, the MPIE research group takes part in the development of a model of phase and microstructure formations inside the mould. Also some other companies and institutes from Scandinavia and southern Europe are taking part in this project, which is expected to be promoted by the “European Research Fund for Coal and Steel” (the former ECSC).

The modelling will be carried out using a “thermodynamic library“, which was developed at MPIE. The calculations are based on the *generalized enthalpy method* [9]. This is a single domain method for the heat and mass transfer, which facilitates the computation of non-equilibrium multiphase solidification processes, such as of peritectic steel grades, in contrast to conventionally used multi-domain methods like the boundary-layer model [10], or the integral method. Quite recently, the multiphase field method as a multidomain method enables to treat complex phase formations. The developed enthalpy method is

extended to non-equilibrium phase formation by incorporating higher cooling rates, away from the assumption of local equilibrium at the solid-/liquid respectively solid-/solid interface.

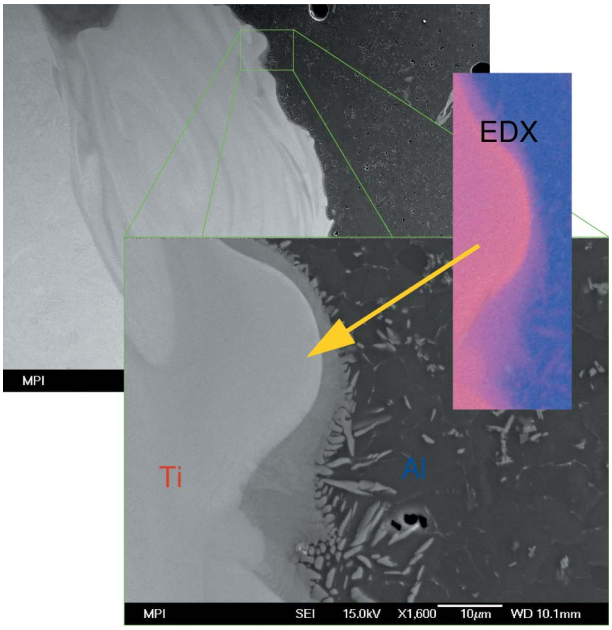

Fig. 3: *Example of a phase seam between laser welded aluminium and titanium. A plane EDX scan and detailed examination of Kikuchi diagrams were performed to determine the distribution of intermetallic phases.*

An ongoing research project is the simulation of aluminium/titanium weldings. Because the joining of these two metals with entirely different physical and thermal properties holds its specific difficulties, especially the formation of intermetallic phases, which might embrittle the joining seam. This complicates or inhibits the application of conventional joining techniques (Fig. 3). The "Bremer Institut für angewandte Strahltechnik" (BIAS) is cooperating in processing and supplying the laser welded samples and providing the necessary process data.

Laser welding of systems like titanium aluminides possessing a variety of different composition with superlattice structures is a challenging subject so far. The demand for titanium and aluminium to save weight and improve performance of the constructions is constantly growing in the automotive and aerospace industries. Therefore great potential applications will be expected.

In order to develop a model for laser welded joints, it is important to examine the thermodynamic and thermophysical properties as well as the formation kinetics of the coexisting phases, like α_2-Ti_3Al, γ-TiAl, $TiAl_2$ and Al_3Ti. Especially a quantitative treatment of the different kinetic processes is important to understand the phase formation under prevalent non-equilibrium conditions. The described project, funded by the DFG is carried out within the scope of the "Schwerpunktprogramm 1139".

Martensitic phase transformation in high-purity iron due to rapid quenching

Martensitic phase transformation in thin high-purity iron foils of 25 µm in thickness produced by rapid quenching either from the liquid or from the solid state (γ-phase regime) have been demonstrated. The interstitials impurities, such as carbon and nitrogen were determined to be c_C = 22 ± 4 ppm and c_N = 12 ± 3 ppm.

Thin splats of 20 to 30 mm in thickness experienced cooling rates during splat quenching - liquid to solid transformation - of the order of $>10^6$ K/s and during the solid state $\gamma \rightarrow \alpha'$ transformation by quenching in ice brine of about $5 \cdot 10^4$ K/s.

As the cooling rate is increased, the transformation temperature of 911 ºC is reduced to about 550 ºC when lath martensite appears.

There are three strong evidences for martensitic transformation in high-purity iron: the martensite relief appears on the surface and the shape of primary austenite grains including annealing twins are present. This is shown in the micrograph of Fig. 4.

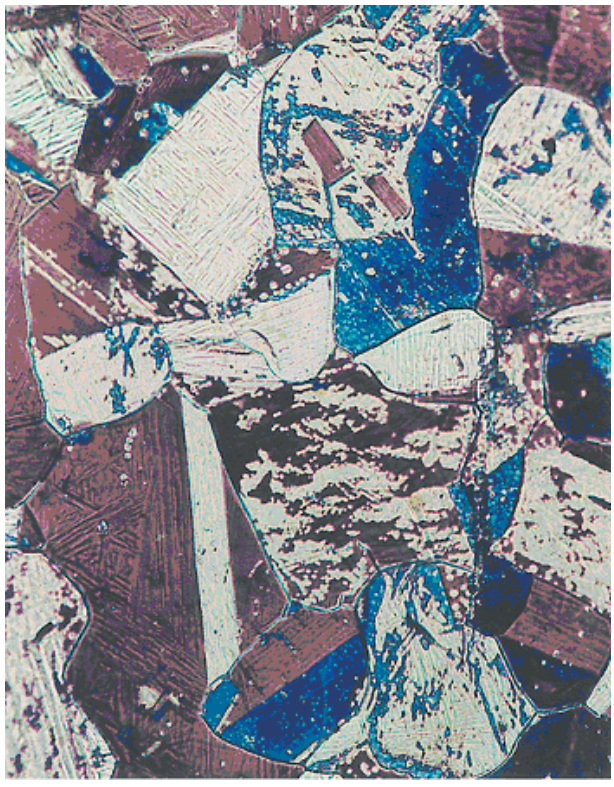

Fig. 4: *Martensite relief and primary γ-annealing twins in a rapidly quenched high-purity iron foil; quenching rate: > 5 · 10^4 K/s.*

The morphology of the martensite is either of lath type with high dislocation density or of twin type with almost no dislocations depending upon the cooling rate.

The Bain strain of $\varepsilon_{[110]} = 0.124$ (dilatation in the [110] direction) and $\varepsilon_{[001]} = -0.205$ (contraction in the [001] direction) is completely released by shear or twinning formations. The diffraction patterns do not show any evidences of a tetragonally distored b.c.c. lattice and some peak broadening is indicated accompanied with a suppression of the intensity of the (200) reflex. The orientation relation between γ and α' is different from the Kurdumow-Sachs relation and was determined to be $(111)_\gamma \parallel (110)_\alpha$ and $[211]_\gamma \parallel [121]_\alpha$.

Development and Characterisation of New Materials

A. Schneider

The main topics of the research activities are focused on the development and characterisation of novel materials for structural and functional applications. An important task of all projects is the understanding of the correlation between microstructures and mechanical properties. The evolution of the microstructure is investigated experimentally by microscopic and nanoscopic methods and predicted by computer simulations using the software Thermo-Calc and DICTRA. The materials under investigation are ordered alloys, such as Fe_3Al- and NiAl-based alloys, and ferritic steels. The development of ferritic steels based on computer simulations of thermodynamics and kinetics of phase transformations is performed in cooperation with the Department of Physical Metallurgy [11, 12]. Structural materials developed for the petrochemical industry are tested for their high temperature corrosion properties under metal dusting conditions.

Development and characterisation of new Fe-Al-based alloys with strengthening precipitates for high temperature applications

Fe-Al-based intermetallic alloys for high temperature application in power plants and petrochemical plants are under investigation (*L. Falat*). The industrial application of Fe_3Al-based alloys is limited to temperatures up to 650 °C. This limitation is related to the requirement of sufficient strength properties at elevated temperatures [13]. In order to increase the high temperature strength alloys with 15 at.%Al and 26 at.%Al with MC-carbides such as TiC, NbC, TaC and VC are investigated. Fundamental studies concerning phase equilibria in heat treated alloys are performed by means of electron-probe microanalysis (EPMA) and X-ray diffraction [14]. Computer simulations on phase equilibria and transformations serve as predictive tools in order to reduce the number of experiments.

Metal dusting of precipitation-hardened Fe-Al-based alloys

The high temperature corrosion process metal dusting attacks conventional high temperature alloys in petrochemical plants. It is suspected that iron aluminium based alloys might possess a higher resistance to metal dusting because of the formation of a protective Al_2O_3 layer. Fundamental studies on the metal dusting of iron (*J. Zhang, G. Inden, H.J. Grabke, DFG*) [15-19] support the understanding of the more complex metal dusting corrosion of iron aluminium alloys.

The kinetics of the carbon transfer was measured using thermogravimetric analysis (TGA). Samples of Fe-15Al-2M-1C (M = Ti, V, Nb, Ta) (in at.%) were exposed to a flowing carburising atmosphere at 650 °C. The carbon activity of the 15 CO - 84.3 H_2 - 0.7 H_2O gas mixture was calculated to be $a_C = 28.4$. The phases of the carburised samples were determined by X-ray diffraction (XRD) and scanning electron microscopy (SEM) equipped with EDX analysis. The TGA results lead to the conclusion that a binary Fe-15Al alloy is not resistant against metal dusting. The alloying additions Nb, Ta and V with C retard the onset of metal dusting but not efficiently. The Fe-15Al-2Ti-1C alloy seems to be the most resistant one because metal dusting could not be observed even after 24 h of carburisation.

Computer simulations using the software Thermo-Calc have been conducted in order to predict the formation of the carbides M_3C, MC and κ-carbide (Fe_3AlC_x) during carburisation. The calculations showed that for the Fe-15Al-2M-1C (M = Ti, V, Nb, Ta) (in at.%) alloys a sequence of MC-carbide, κ-carbide and cementite can be expected for carburised samples.

Flow stress anomaly and order-disorder transitions in quaternary Fe_3Al-based alloys

The binary stoichiometric compound Fe_3Al shows an unusual increase of the yield strength at intermediate temperatures followed by a sharp drop around 550 °C. At this temperature a structural phase transition from $D0_3$ to B2 structure occurs. Alloying elements may strongly influence the critical temperatures. Therefore, the stress-strain behaviour as a function of temperature as well as the critical temperatures of the $D0_3 \leftrightarrow B2$ and $B2 \leftrightarrow A2$ structural transitions were studied on quaternary Fe_3Al-based Fe-26 at.% Al-4 at.% Ti-X alloys containing 2 or 4 at.% of X = V, Cr, Nb, or Mo (*F. Stein*). The microstructures of the alloys were characterized by light optical

microscopy, transmission electron microscopy (TEM), and electron-probe microanalysis (EPMA). The alloys containing V, Cr, or Mo are of single phase solid solutions, whereas the additions of 2 and 4 at.% Nb result in the formation of Laves phase precipitates. The comparison of the temperatures of the maximum of the stress anomaly and the $D0_3 \leftrightarrow B2$ transitions determined in compression tests and by differential thermal analysis (DTA) clearly reveals that there is no correlation between the stress anomaly and the degree of ordering. The effect of the different alloying elements on the order-disorder transition temperatures of the quaternary alloys is discussed and compared with that of respective ternary alloys without Ti [20].

Nanostructural characterization of NiAl-X (X = Cr, Fe, Re) alloys using atom probe field ion microscopy (APFIM)

For quantitative correlations between micro-/nano-structure and mechanical properties of the investigated materials, transmission electron microscopy (TEM) and atom probe field ion microscopy (APFIM) are used in order to analyse the dislocation structure and interactions of lattice defects with interfaces (antiphase and phase boundaries). APFIM investigations which allow imaging and chemical analysis of the samples on an atomic scale, are conducted in order to determine the atomic distribution in the ordered phases, e.g. for Nb in TiAl [21]. With the knowledge of e.g. the site occupancies of antistructure atoms or of ternary alloying elements in the ordered matrix lattice, the ductility or brittleness of an alloy can be understood regarding the antiphase boundaries (APBs) involved in the deformation process.

Alloys based on the intermetallic B2-ordered NiAl are under investigation in view of high temperature applications. Its major shortcomings are their poor room temperature ductility due to the high antiphase boundary (APB) energy which inhibits the activation of a <111> slip and the insufficient creep resistance. Atom probe field ion microscopy investigations have been performed on NiAl alloyed with Cr, Fe, and Re in order to elucidate the influence of the alloying elements on the microstructure and thus on the mechanical properties. The site preferences of the ternary elements were determined by layer resolved atom probe surveys.

The Cr content of NiAl - 2 at.% Cr alloys after heat treatment at 1200 °C/100 h amounted to ≤ 1 at.% in solid solution and decreases with increasing Al/Ni ratio. The latter is due to the site preference of Cr atoms for the Al sublattice [22]. An APB of a<111>{123} orientation showed strong Cr segregation with Cr also occupying Al sites [23]. A <111> super dislocation showed no dissociation into partial dislocations [22, 23]. Chromium precipitates in these alloys were also imaged and analysed [24, 25].

In contrast, $Ni_{47.5}Al_{47.5}Fe_5$, heat treated at 1200 °C/100 h, possessed Fe additions in solid solution. The Fe atoms were almost equally distributed between the Ni and Al sublattices as 54 ± 8 % were located on Al sites [26].

The solid solubility and precipitation of Re were investigated in the hypoeutectic intermetallic alloy $Ni_{49.5}Al_{49.5}Re_1$ in the as-solidified state and after various heat treatments: 1000°C/10, 30,100, 1000 h and 1300 °C/1000 h. Re precipitates inside the primary NiAl crystals were observed in FIM with atomic resolution (Fig. 5), from small Re-clusters consisting of a few Re atoms in the as solidified samples up to Re crystallites of about 100 nm after the heat treatment 1000 °C/100 h. These are responsible for the high creep strength of $Ni_{49.5}Al_{49.5}Re_1$. Layer resolved APFIM measurements on the (001) pole reveal a strong site preference of Re atoms for the Ni sublattice of the B2 ordered NiAl superlattice [27]. The concentration of Re atoms dissolved in the NiAl solid solution matrix varies between 0.18 at.% $\leq c_{Re} \leq$ 0.6 at.% with an average solubility of c_{Re} = 0.3 at% in the as solidified state and decreases during the heat treatment at 1000°C to a Re concentration of $c_{Re} \approx$ 0.18 at.% (*R. Fischer, J. Deges*).

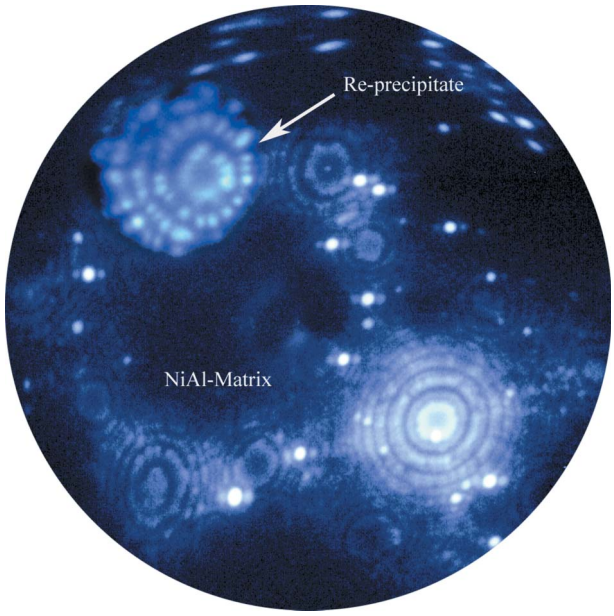

Fig. 5: *FIM image of a $Ni_{49.5}Al_{49.5}Re_1$ sample after a heat treatment for 10 h at 1000 °C. Note the small Re precipitate of about 10 nm in diameter exhibiting atomic resolution in FIM.*

Investigation on site occupancies of the ternary alloying elements Cr, Fe, and Re in NiAl

Atom probe field-ion microscopy (APFIM) with atomic layer resolution was employed for investigating the site preferences of the transition metals Fe, Cr and Re dissolved in stoichiometric NiAl. APFIM

analysis sustained by X-ray diffraction shows that there is a strong site preference of Cr atoms for the Al sublattice. The NiAl host lattice possesses excess Al atoms and vacancies in the Ni sublattice. This leads to a considerable contribution of aluminum to solid solution strengthening of NiAl(Cr) primarily due to structural vacancies.

Fe atoms are almost equally distributed within the Al and Ni sublattice exhibiting a weak preference for Al sites (APFIM) in contrast to the weak preference for Ni sites determined by ALCHEMI. Therefore, no pronounced constitutional lattice defect concentrations are present in NiAl(Fe) solid solutions. The effect of Fe on the lattice expansion of NiAl is caused by the larger Fe-Al bond length compared to the more tight Ni-Al bond. Consequently, Fe additions to stoichiometric NiAl promote solid solution softening.

Re solutes in NiAl possess a strong site preference for the Ni sublattice despite the fact that the atomic radius of Re atoms is considerably larger than that of Ni atoms. Generated Ni antistructure atoms are distributed in the Al sublattice and will slightly contribute to a lattice contraction. However, the resulting lattice dilatation of the NiAl(Re) solid solution is due to the dominant size effect of the Re atoms. Another aspect is the elastic contribution of Re atoms to solid solution strengthening of NiAl [26] (*R. Fischer, J. Deges*).

Atom probe field-ion microscopy analysis of iron aluminides

The field-ion image formation and field evaporation sequences of ordered Fe_3Al with DO_3 superlattice structure was studied performing the atomic plane counting technique and the analysis of field evaporation sequences of different atomic plane sets. The controlled field desorption experiments were supported by atom probe microanalysis. The results show that Al atoms are the brightly imaging species in field-ion micrographs and provide the main contribution to the field-ion image formation of Fe_3Al. However, Fe atoms contribute also to the formation of field-ion images. They appear visible with atomic resolution in topmost atom layers on poles of the ordered (222) and (226) plane sets. This characteristic imaging behaviour allows to perform a layer by layer detection in FIM. Fe atoms seem to be the preferentially field evaporating species. The ordered (222) plane set exhibits double layer (Al + Fe) field evaporation. The remaining two iron layers underneath are suitable for imaging with atomic resolution. In contrast the ordered (226) plane set of the same stacking sequence as that of the (222) plane exhibits the threefold layer (Al + Fe + Fe) field evaporation mechanism due to the closer interplanar spacings in the [113] direction of the (226) pole. The preferential field evaporation of Fe atoms and the strong field-ionization of Al atoms are the important physical contributions to the field-ion image formation of DO_3-ordered Fe_3Al [28] (*Z. Liu, J. Wesemann, G. Frommeyer)*.

Intermetallic High-Temperature Alloys

G. Frommeyer

Superplasticity in Fe_3Al(Cr)

Superplasticity of an Fe_3Al based intermetallic alloy with 3 at.% chromium has been investigated in the strain-rate range from 10^{-5} to 10^{-2} s^{-1} at test temperatures between 700 and 900 °C. The composition of the iron aluminide was Fe-28 at.% Al-3 at.% Cr with additions of titanium and carbon. After thermo-mechanically processing the material possessed a coarse-grained microstructure with an average grain size of 55 ± 10 μm. Strain-rate exponents of $0.33 \leq m \leq 0.42$ were recorded at strain rates of the order of 10^{-5} to 10^{-3} s^{-1} in the temperature range of 750 to 900 °C. Maximum elongations to failure of 350 % and more were achieved. From thermal activation analysis of superplastic flow an activation energy of 185 ± 10 kJ/mol was derived. This is comparable with activation energies of superplastic flow in Fe_3Al(Ti) alloys. However, in unalloyed Fe_3Al the activation energy is higher, about 263 kJ/mol [29].

Optical microscopy showed grain refinement to about 30 ± 5 μm in size in superplastically strained tensile samples. Transmission electron microscopy gave evidence of the formation of subgrains of 0.3 to 0.5 μm in size. Superplasticity in this iron aluminide is mainly due to viscous dislocation glide, controlled by solute drag in the transformed B2 lattice at the actual deformation temperatures.

Constitution of NiAl-Re alloys

The constitution of the quasi-binary eutectic NiAl-Re section of the ternary Al-Ni-Re phase diagram was reinvestigated by using differential thermal analysis, metallography and scanning electron microscopy [30]. The melting temperature of stoichiometric NiAl was determined to 1674 ± 3 °C and is in good agreement with the thermodynamically calculated temperature of 1676 °C.

Due to the extremely high melting temperature of 3180 °C of the h.c.p. refractory metal rhenium the eutectic of the NiAl-Re system is expected to be at low Re concentrations and high temperature. As-cast eutectic NiAl-Re alloys exhibit anomalous and irregular eutectic solidification microstructures. At concentrations higher than 1 at.% Re non-equilibrium microstructures occur consisting of NiAl-Re eutectic and Re and NiAl crystals. The eutectic point of the quasi-binary section of the NiAl-Re system is

determined to be at 1.25 at.% Re and at 1668 °C. The maximum solid solubility of Re in NiAl is 0.6 at.% at the eutectic temperature and 0.2 at.% at 1300 °C [30]. Due to the high eutectic temperature which is only 6°C below the melting temperature of NiAl and the relatively high Re solid solubility at the eutectic temperature the NiAl-Re eutectic reaction is close to a peritectic one.

A well developed fibrous morphology was produced by unidirectional solidification of a NiAl-$Re_{1.25}$ eutectic using the Bridgman technique at a growth velocity of 30 mm/h, see Fig. 6 (*R. Rablbauer*).

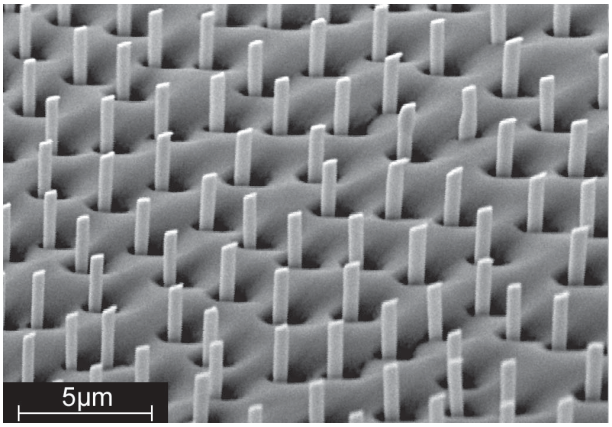

Fig. 6: *SEM image showing the microstructure of an unidirectionally solidified NiAl-$Re_{1.25}$ eutectic. The Re fibres are aligned parallel to the rod axis. The NiAl matrix is preferentially etched out.*

Mechanical properties of NiAl-Re alloys

As-cast NiAl-Re alloys exhibit improved plastic deformation in compression tests at room temperature in comparison to polycrystalline as-cast single-phase NiAl. The plastic deformation of as-cast alloys exhibits a maximum at the hypoeutectic NiAl-Re_1 composition. Enhanced ductility is due to crack stopping mechanism at primary NiAl crystal and eutectic boundaries.

The as-cast NiAl-Re_1 ingots with fine and coarse microstructures, as well as the unidirectionally solidified NiAl$Re_{1.25}$ eutectic exhibit yield strengths higher than 90 MPa at 1200 °C.

The solid solution hardening effect of Re is negligible because of its low solid solubility at room temperature. However, effective second phase strengthening in the as-cast NiAl-Re alloys occurs by the superposition of two different particle size dependent mechanisms: solubility driven and finely dispersed Re particles in NiAl primary crystals and coarser Re fibres of about 0.5 µm diameter in the eutectic.

The high eutectic temperature of 1668 °C accompanied with the occurrence of effective particle strengthening and fibre reinforcement mechanisms imply high thermal stability and improved creep strength of these materials. These superior properties are promising new perspectives for the development of high-temperature structural material based on NiAlRe (*R. Rablbauer*).

TiAl-based alloys - physical and mechanical properties

Gamma titanium aluminides are one of the most promising high temperature light weight materials alternative to conventional heat-resistant steels and superalloys in high-performance automotive and aircraft engines. TiAl base alloys consisting of the intermetallic phases γ-TiAl and α_2-Ti_3Al possess attractive properties for applications under high thermal and mechanical load, e.g. low specific weight of about 3,8 - 4.1 g/cm^3, good oxidation and burn resistance at temperatures up to 800 °C, high elastic stiffness and enhanced high temperature strength. Comprehensive investigations by performing TEM and ALCHEMI have been carried out in order to determine the effects of ternary alloying elements of transition metals, in particular Cr, Mo, Nb and Cu, on lattice parameters, phase morphology and microstructures. Advanced intermetallic Ti-46Al-(Cr,Cu,Mo,Si) alloys of enhanced ductility and strength are under investigation. Improved strength ($R_{p0.2}$) of 800 to 1070 MPa and relatively high room temperature ductility up to ε_p = 4.8 % have been demonstrated. Enhanced ductility is attributed to decreased stacking fault energy and twinning is one major deformation mechanism in these alloys (Fig. 7) [31-36].

The fine-grained intermetallic Ti-46Al-1.5Mo-0.2Si (at.%) alloy with coexisting α_2-Ti_3Al finely dispersed throughout the γ-TiAl matrix microstructure exhibits enhanced superplasticity at strain rates in the range $5 \cdot 10^{-4}$ to $5 \cdot 10^{-2}$ s^{-1} and in the temperature regime of 975 to 1050 °C. The recorded strain-rate-sensitivity exponents are relatively high in the range of $0.55 \leq m \leq 0.78$. Maximum superplastic elongations of $\varepsilon_p \approx 1060$ % were achieved at strain rates of $8 \cdot 10^{-3}$ s^{-1}. Quasi superplastic deformation behavior occurs also at higher deformation rates up to 10^1 s^{-1} and at temperatures of about 1180 °C to 1280 °C. This refers to high-strain-rate superplasticity (HSS), which is in particular suitable for near net shape forming operations of high-performance engine parts. The deformation mechanism of HSS will be investigated and described by a deformation model [37, 38] (*S. Knippscheer*).

Processing of titanium aluminides

Manufacturing processes for the production of TiAl automotive engine parts based on centrifugal casting, near-net shape quasi-isothermal forging or superplastic die forming have been developed in cooperation projects [39, 40]. The influence of conventional

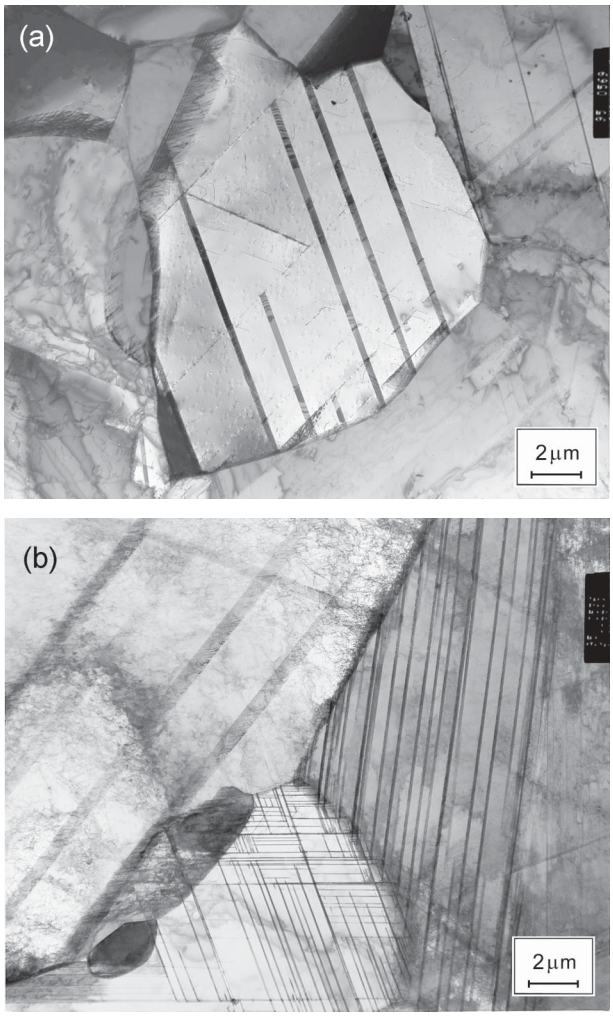

Fig.7: *Bright field TEM images of an extruded Ti-46.8Al-1Mo-0.2Si alloy after tensile deformation of about 3.6 % at room temperature. (a) The slightly deformed grip section of the tensile sample shows very few deformation twins. (b) In the strongly deformed region close to the fracture surface the sample exhibits enhanced mechanical twinning [36].*

machining on the microstructure and mechanical properties of TiAl base alloys is under investigation in cooperation with the Institut für Werkzeugmaschinen und Fabrikbetrieb, TU Berlin. Improved machining parameters and tool technology offer great potential to reduce the production costs and time of high quality engine components made out of TiAl [41].

Al-Al_3Ti metal matrix composites based on multi step extrusion of Al(X)-20Ti (vol.%) powder are under development where extrusion process at low temperature should lead to a controlled formation of intermetallic phases. After a suitable heat treatment of the axisymmetrically deformed bars the microstructure of the MMC material should consists of a solid solution hardened Al matrix interspersed with discontinuous intermetallic Al_3Ti fibers. Materials properties and structural characterization of Al-MMC and Aluminides are reviewed in [42, 43] (*S. Knippscheer*).

High-strength α/β-Ti based alloys - alloy design and characterization

Quasi-eutectoid Ti-10Co-4Al and Ti-12Co-5Al alloys consist of a solid solution α-Ti matrix interspersed with intermetallic Ti_2Co particles. These alloys possess superior superplastic properties in the ultra fine-grained thermomechanically processed state and superior creep resistance in the coarse-grained state as well. Alloy development and characterization of phase transformation, microstructure and mechanical properties of high-strength TRIPLEX Ti-(Al,Cr) alloys exhibiting a microstructure containing α, β and Ω phase is also in progress.

Eutectic α-Ti(Si) cast alloys have been significantly reinforced by about 30 vol.% refractory and extremely hard Ti_5Si_3 precipitates or as-directionally solidified fibers possessing the complex hexagonal $D8_8$ lattice structure. Moreover, the α-Ti(Si) solid solution matrix phase exhibits excellent high-temperature oxidation resistance. The microstructure-properties relations have been studied in greater detail in order to clarify the high temperature deformation mechanisms of the complex intermetallic Ti_5Si_3 compound [44].

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Department of Metallurgy

A.R. Büchner (provisional)

Scientific Concepts

At present the institute is looking for a new head of the department. Therefore, some topics of the Department of Metallurgy are not actively pursued at the moment. It is expected that the valuable research conducted in the past will be followed up in future. Generally the Department of Metallurgy focuses on the fundamentals of steelmaking. The targets are both to optimise conventional process technologies as well as to support new lines of development. All technologies studied at MPI can contribute to reduce the number of process steps and to save cost and energy, and may be part of future concepts for new steel production lines. An important new technology is twin roll strip casting, which has recently been adopted by the industry especially for the production of stainless steels and carbon steels.

Among the methods applied at the department of metallurgy, numerical modelling is a central tool. At the same time, various experimental studies are carried out using laboratory measurements and empirical data supplied by our industrial partners.

Metallurgical Process Models

N.N.

MPI, IRSID, TKS, Dillinger Hüttenwerke and Rautaruukki Steel participate in the ECSC project "Determination of *high temperature surface crack formation* criteria in continuous casting and thin slab casting"; the project is co-ordinated by IRSID. The aim is to find rules for the choice of a "process with reduced cracking risk" for as-cast products. The MPI consequently advanced a previously developed numerical model for the computation of thermally induced stresses and cracking risk. Work on the thermomechanical simulation of continuous casting has been continued at MPI, with an application to Dillingen caster no. 5 and Rautaruukki caster no. 6. The modelling of phase transformation was considered and dilatometric tests have been carried out. Simulation of cyclic tests performed by TKS is in progress in order to determine critical values for the formation of surface cracks (*M. König*).

Near Net Shape Casting

A.R. Büchner

Two twin roll laboratory casters are operated in this group for producing steel strip in the thickness range of 1 to 3 mm [1]. Steel strips with a width of 65 mm can be cast from a smaller machine with a 40 kW induction furnace and a capacity of up to 15 kg. Steel and copper rolls (with and without nickel coating) are available. The rolls on this machine have no water cooling. A gas protection device was constructed in order to reduce oxidation between furnace and tundish. This caster permits inexpensive trials in short intervals and is used extensively for various kinds of tests within projects and also for orders from industry.

With the larger machine 120 mm wide strip is cast using water cooled copper rolls, again with or without Ni coating. This caster is used for all tests which require thermal steady state. A 150 kW furnace is able to deliver heats of up to 250 kg. The furnace can be combined directly with a gas-protected tundish, which can prevent reoxidation more efficiently. A new preheating facility for the pouring spout was constructed.

In-line rolling is of large interest for strip casting. One purpose is to eliminate shrink holes in the strip. Other aims still to be studied are the influences on the strip structure. As a preparation of an extensive study of in-line rolling effects, the facilities of the rolling mill were investigated in detail. Especially the limitations in rolling force, constancy of rolling velocity and of upper torque limit were addressed.

A general study of in-line rolling of twin roll cast strip is underway, where different types of transformations in crystal lattice, such as (δ / γ / α, St14), (δ / α, FeSi), (δ / γ, stainless steel), (γ / α, C60) will be investigated. To carry out the rolling step close to the relevant transformation points, the cooling distance between both units is varied. Occasionally,

very short intermediate times are needed; thus, the guidance of the strip needs to be achieved within relatively short distances. This requires, however, a very exact velocity coordination between rolling mill and caster. The problem is worsened because of different degrees of reduction, which lead to different amounts of material backward slip. To solve this velocity co-ordination problem, a sophisticated software is in preparation. Fig. 1 illustrates the situation of inline rolling (*S. Pötschke, A.R. Büchner*).

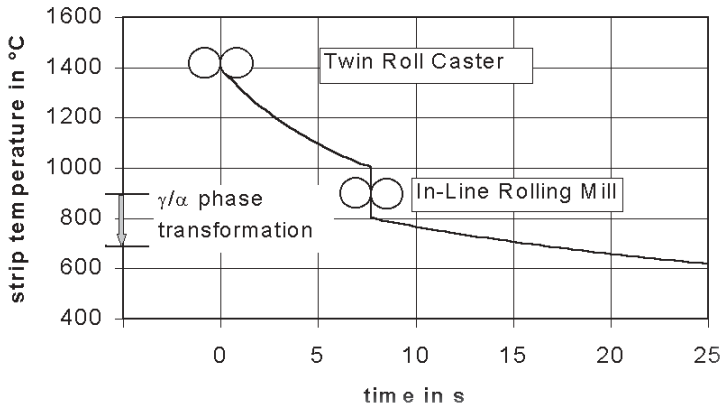

Fig. 1: *Schematic illustration of inline rolling problems.*

An EU-project in cooperation with SIMR in Stockholm and Salzgitter Flachstahl GmbH addresses the *detrimental effects of tramp elements, Cu and Sn*. Concentration limits in the case of a continuous casting line are well known. In addition to that, it will be studied which effects related to tramp elements are important in the fully different casting technique with a twin roller. Due to the change in temperature and time history, all diffusion-controlled effects will be changed. As a preparation of these studies, the casting conditions for Cu- und Sn-containing steels were investigated. Special emphasis was laid on avoiding cracks in order to obtain strip material suitable for mechanical tests.

Several thin strip casting experiments have been carried out under different conditions. They have to be compared with results from laboratory hot shortness tests carried out at SIMR. A detailed study was needed in order to find out how to compare specimens from such different techniques. It seems that the thickness of the scale is the common feature. Scale formation calculations following a previous investigation [2] were applied to both types of experiments (*S. Pötschke, A.R. Büchner*).

An ECSC-project in cooperation with Corus, CSM, Sidenor, and TKS addresses the problem of surface quality of continuously cast semis. The objective is a basic understanding of shell development and growth especially for the very critical peritectic steel type. A model mould was constructed, where metallic walls of different type come into contact with a rising melt. Thermocouples in the walls give information on heat contact and heat flow. These data will be analysed in connection with microscopic studies. As a result of first experiences, the model mould was fully redesigned; the size was enlarged in order to stabilize the melt movement; the preparation of the thermocouple holes had to be improved in order to assure a well defined thermal contact.

The steps necessary to calculate the relevant quantities for the forming of the structure, namely cooling rate $\dot{T}$ and temperature gradient grad T at the phase front, are the following: Temperature measurement in the wall; correction of temperature for time delay failures; calculation of heat flux at the wall interface; calculation of $\dot{T}$ and grad T. Large efforts are necessary to establish a reliable temperature measurement by thermocouples in the walls, as the real temperature changes take place faster than the thermocouples can react. The development of the temperature correction calculations and also some corresponding calibration measurements were very time-consuming and have not yet been completed. An electronic device for storing data from up to sixteen thermocouples with sufficient speed was tested and improved. In Fig. 2, the difference between the temperatures in the wall as measured by a thermocouple and the corrected real values is demonstrated (*M. Thiemann, A.R. Büchner*).

The *contact and heat transfer between melt and roll in twin roll casting* is a subject of great importance. The length of pool contact during casting can be observed and renders important information. From the experience that this contact depends strongly on

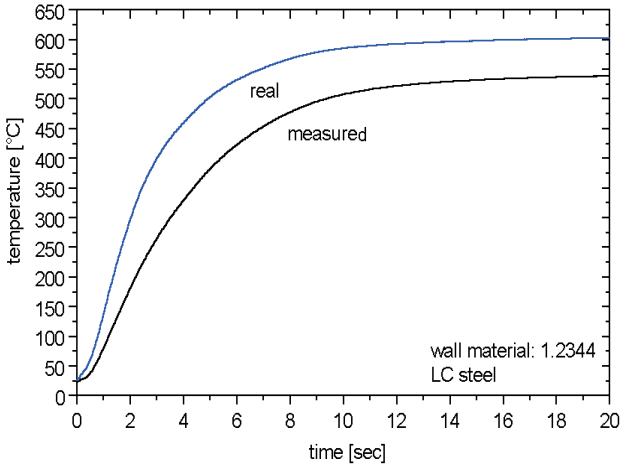

Fig. 2: *Illustration of the need of temperature corrections (temperature measurement 1 mm below the surface; starting at time of melt contact).*

the type of melt and rolls used, it is deduced that microscopic physical effects need to be studied. The model mould mentioned above is used in order to investigate contact angles, wetting behaviour and heat transfer coefficients. The parameters to be varied are four types of mould walls, at least three types of melt, and the gas atmosphere. Fig. 3 demonstrates large differences of temperature time curves depending on wall material (*M. Thiemann, A.R. Büchner, DFG support*).

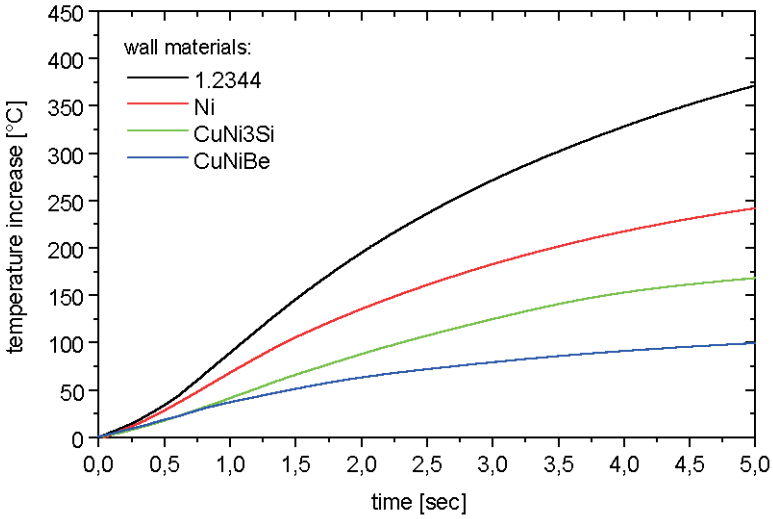

Fig. 3: *Demonstration of the influence of the wall material (temperature measurement 1 mm below the surface; starting at time of melt contact).*

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Department of Microstructure Physics and Metal Forming

D. Raabe

Scientific Concepts

The department for Microstructure Physics and Metal Forming at the Max-Planck-Institut für Eisenforschung GmbH covers a broad spectrum ranging from fundamental aspects such as microstructure physics, materials mechanics, and crystallographic textures to applied topics associated with thin slab casting, large scale hot working, and metal forming of modern materials both at the theoretical and experimental level (Fig. 1).

The department has five scientific groups, namely "Theory and Simulation" (Dr. F. Roters), "Diffraction and Microscopy" (Dr. S. Zaefferer), "Thermomechanical Processing" (Dr. D. Ponge), "Metal Forming" (Dr. W. Rasp) and the new research group "Surface and Microstructure Mechanics" (Dipl.-Ing. M. Sachtleber). In addition to this structure the department has established four service groups, namely, "Scientific Computing and Computational Services", "Metallography", "Materials Testing Laboratory", and "Materials Processing". The basic idea behind this structure of the department is to build a highly interdisciplinary research initiative which aims at merging physically oriented microstructure research and materials processing on the one hand and theory and experiment on the other hand. The aim of the department

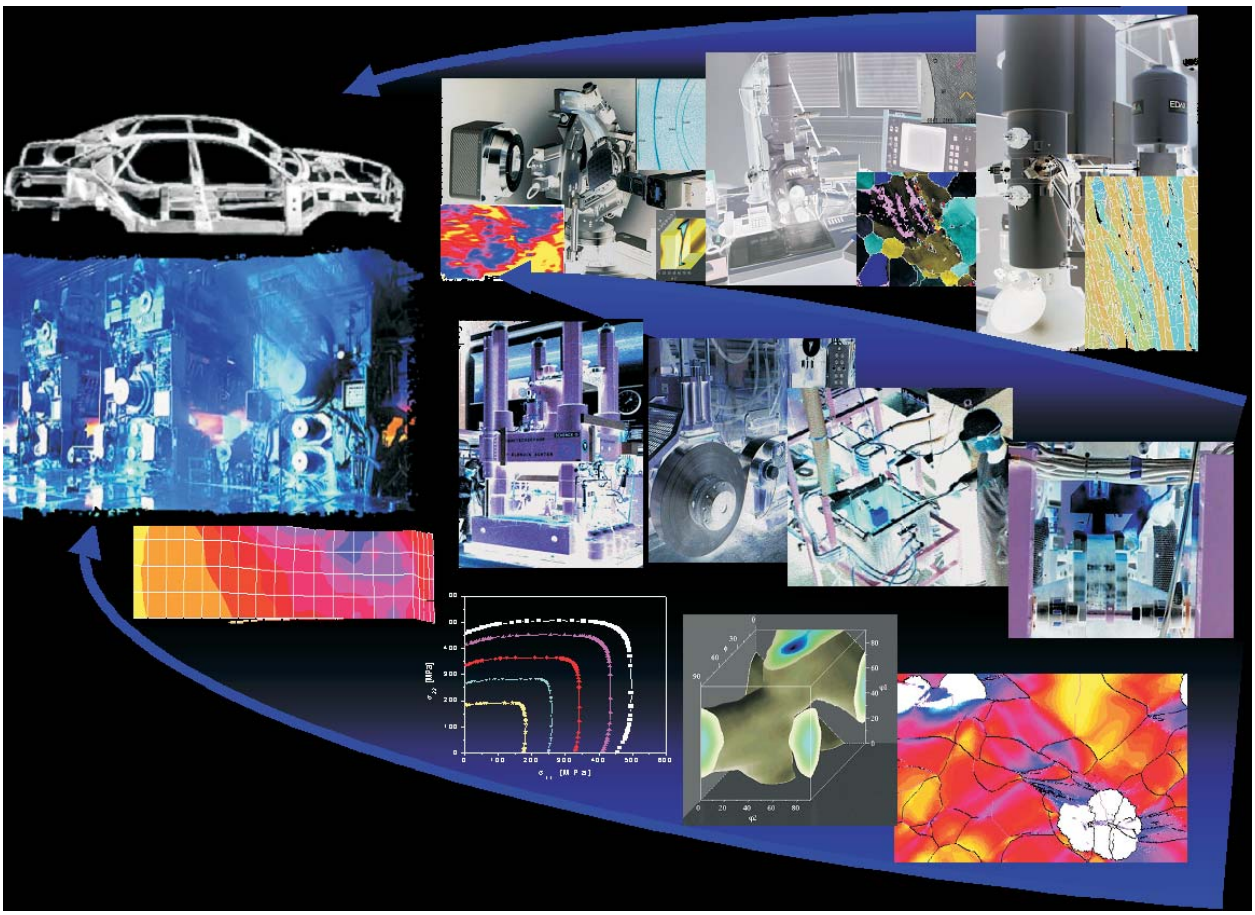

Fig. 1: *The department for Microstructure Physics and Metal Forming covers topics ranging from fundamental aspects associated with microstructure physics, materials mechanics, and crystallographic textures to applied topics such as thin slab casting, large scale hot working, and metal forming of modern materials both at the theoretical and experimental level.*

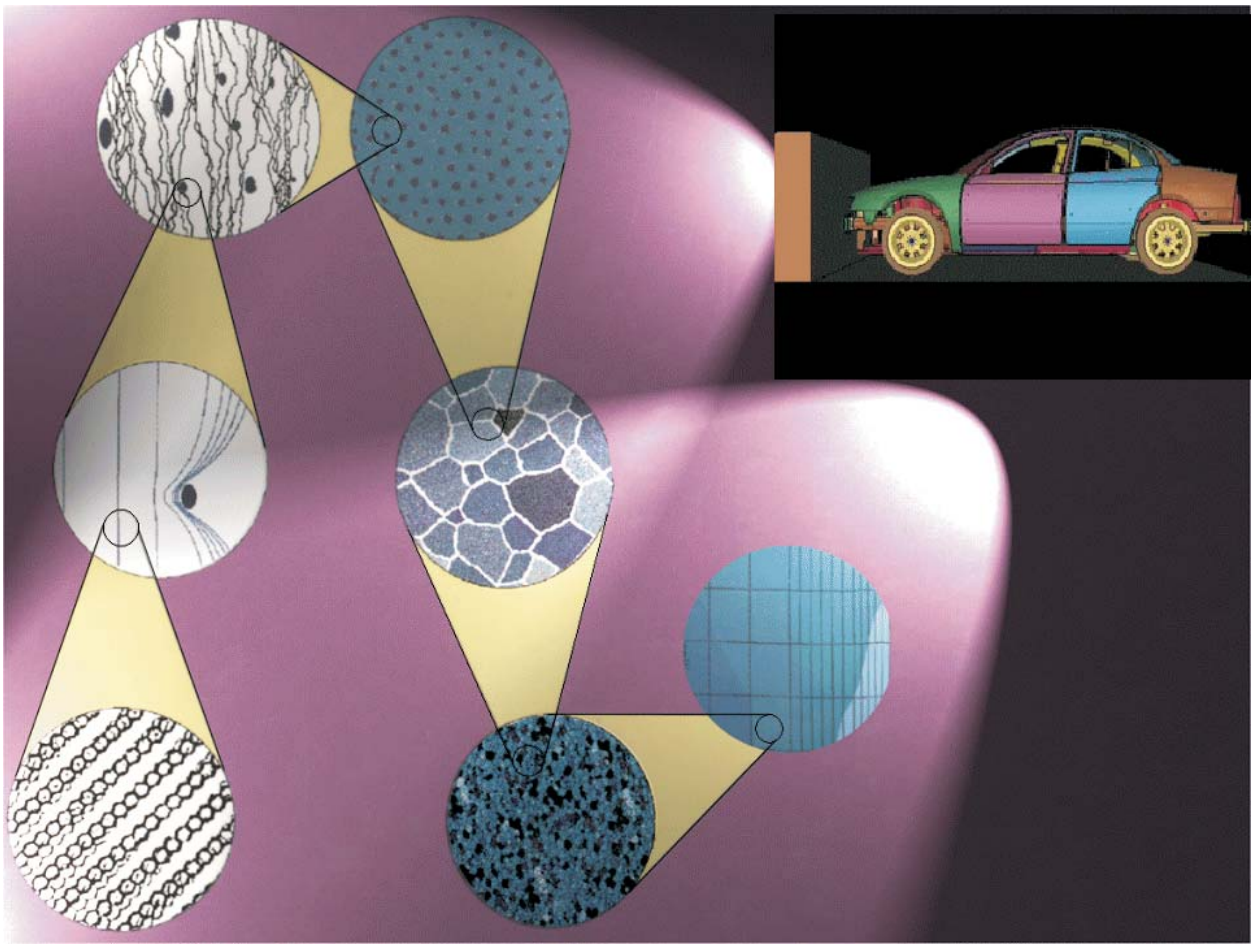

Fig. 2: *In the field of computational materials science the department concentrates on the development of quantitative and scale bridging simulation methods which help to mimic, understand, and design new microstructures, properties, and processes.*

consists particularly in pursuing projects which are based on creating basic knowledge in physical metallurgy for areas which have their roots in the field of application. Among these physically based and microstructure-oriented computational methods, texture and anisotropy, thermomechanical processing, as well as materials surface and interface mechanics are considered particularly challenging topics (Fig. 2).

Correspondingly, these fields were established in the form of interdisciplinary long-term key projects accross the department research groups reaching out also into other departments. In the area of computational materials science an increasing demand exists to develop quantitative and scale-bridging simulation methods which help to mimic, understand, and design new microstructures, properties, and processes. In the area of texture and anisotropy we aim to understand better the mechanisms of microstructure formation from the nanoscopic regime up to the occurrence of macroscopic anisoptropy of engineering parts. This long-term research project is closely connected to various studies in the groups of thermomechanical treatment and theory. In the area of thermomechanical processing our main focus is placed on understanding and optimising microstructure evolution and resulting properties during complex hot working paths. In the area of surface and interface mechanics we concentrate on establishing new joint in-situ experimentation methods and developing new corresponding theoretical approaches for conducting experiments and simulations of forming processes of coated materials. The following sections will give a more detailed outline on these aspects.

All reserach projects are pursued in a highly interdisciplinary and international scientific atmoshpere. Most of our researchers stem from Europe, Asia, and America which establishes a pleasant and constructive research spirit. Scientists in our group typically have a background in materials science, physics, or mechanical engineering.

Theory and Simulation

F. Roters

The activities of the research group "Theory and Simulation" in the years 2001 and 2002 focused on the two topics: the crystal plasticity finite element method (CPFEM) and dislocation density based constitutive laws for the plastic deformation of metals and alloys.

In the field of crystal plasticity finite elements a new method the so-called *Texture Component Crystal Plasticity FEM (TCCP-FEM)* was developed by the group (*F. Roters, Z. Zhao, D. Raabe*) [1, 2]. It is basically a combination of the texture component method with the crystal plasticity FEM. The key problem in incorporating anisotropic material behaviour into industrial scale finite element simulations is to find an efficient way for *representing texture and its development*. The texture component method is very well suited for this purpose. Instead of hundreds of individual orientations, as they are frequently used in other texture discretization procedures, it uses very few components to represent the experimentally obtained texture [3]. The texture component method uses two types of components: the "peak" components and the "fibre" components [4]. The first implementation of the TCCP-FEM uses only "peak" components and the random texture part as input for the crystal plasticity simulation. It was originally implemented using an existing Abaqus UMAT routine for crystal plasticity. In 2002 it was also implemented into the commercial FEM software MSC.Marc (*F. Roters*). For this purpose the original Abaqus UMAT routine was completely rewritten. The new MSC.Marc version uses some advanced Fortran90 features such as dynamic memory allocation and is therefore much more flexible and efficient than the old Abaqus routine. With the new method it is now possible to simulate forming processes such as deep drawing using the full texture information, e.g. the method accounts for both *elastic and plastic anisotropy and* most important *its development*. Fig. 3 shows the simulated earing profile of a deep drawn aluminium cup as compared to the experimental result.

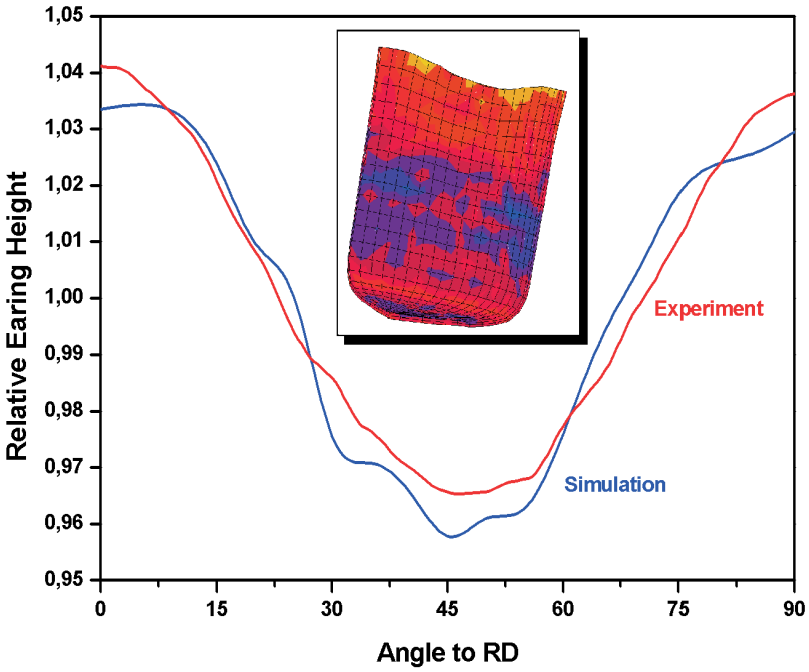

Fig. 3: *Comparison of simulated (blue) and experimental (red) earing profile of Al hot strip. Inlay: FE model of one quarter of the deep drawn cup.*

Parallel to the work regarding the TCCP-FEM the standard CPFEM was used for simulations in a number of research projects of the department.

In the field of fundamental research numerical studies were carried out to study the fragmentation behaviour of single and bi crystals (*Z. Zhao*) [5]. The results were also used to proof the predictions of a simple analytic model for the prediction of grain fragmentation tendency (*D. Raabe, Z. Zhao*) [6]. In the project "*Orientation dependence of micro-mechanical properties*" (*J.-C. Kuo*) simulations of the plain strain deformation of Al bicrystals were carried out (*Z. Zhao*). In 2002 the new DFG project "Experimental and theoretical investigation of strain and orientation gradients in steel at the grain scale" (*A. Ma*) started.

Simulations of *nano-indentation* were carried out (*Y. Wang*) within the project "*Theory of nano-mechanics*" (*C. Klüber*). While it is possible to reproduce the dependence of the shape of the indents on crystal orientation as found in the experiment (Fig. 4), it is still work in progress to directly compare the load displacement curves. There are some problems due to the unsteady changes of the contact area in the FEM simulation which enforce a displacement-controlled simulation in opposition to the load-controlled experiments.

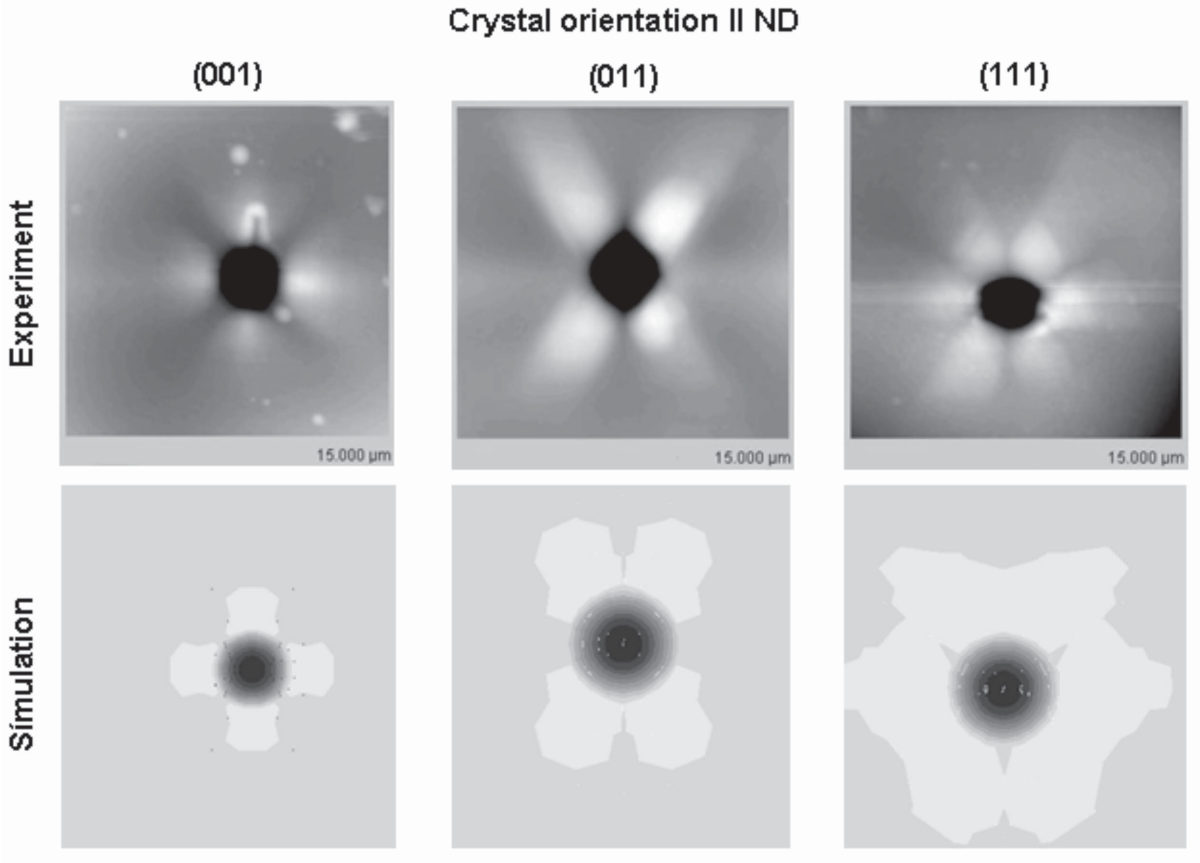

Fig. 4: *Experimental (top row) and simulated (bottom row) height profiles (brighter is higher) of nano-indents in differently oriented Cu single crystals.*

The CPFEM simulations of *foil rolling* (*G. Scheele*) were also continued focusing on the development of the foil surface topography.

The *constitutive law* plays a key role in every forming simulation. Therefore the group works on the development of a *dislocation density based models*. A new concept was developed for the calculation of the mobile dislocation density (*F. Roters*) [7]. Based on formerly developed models work started on a new model for the incorporation into the TCCP-FEM (*F. Roters, A. Ma*). Such model has to be formulated on an individual slip system basis.

One important aspect of using a dislocation density based constitutive law within the TCCP-FEM is that the results can than directly be used as starting input for the simulation of consecutive *heat treatments*. For the simulation of recrystallization a cellular automaton program is used (*H. Lücken*), while grain growth is simulated using a newly developed Monte Carlo code (*N. Chen*).

The group also performs standard simulations of deformation processes using commercial FE packages. One example in this field is the numerical simulation of multi step hot deformation with the hot deformation simulator (WUMSI) of the department (*Y. Wang*).

Microscopy and Diffraction
S. Zaefferer

Task of the group “Microscopy and Diffraction” is the experimental investigation of the microstructure and crystallographic texture in materials and their evolution during different materials processes. The focus lies on the investigation of local processes. Therefore, the experimental methods mainly comprise transmission and scanning electron microscopy (TEM and SEM) and the appropriate diffraction techniques (transmission Kikuchi and spot diffraction in the TEM and backscatter electron diffraction (EBSD) in the SEM). Additionally, X-ray diffraction is used to measure textures, single grain orientations and residual stresses on a macroscopic and microscopic scale.

The group is equipped with a variety of instruments: for EBSD investigations, a high-resolution, high-beam SEM (JEOL JSM 6500 F) with a TSL EBSD system (with DigiView CCD camera) as well as a standard tungsten filament SEM (JEOL JSM 840A) with an HKL Technology EBSD system is available. The latter SEM also has the possibility of inserting a micro tension machine (Kammrath & Weiss) for in-situ deformation tests. For TEM a Philipps CM 20, available in the

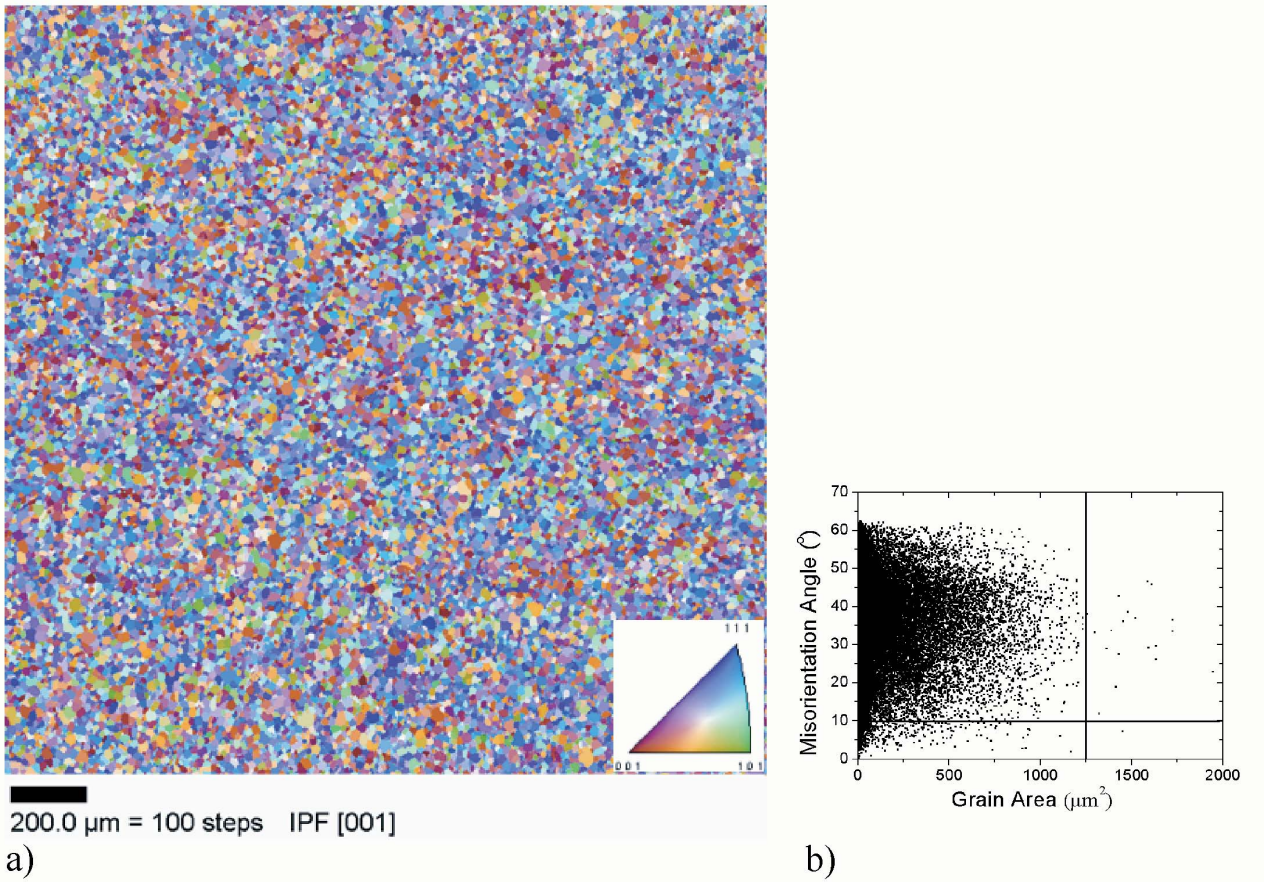

Fig. 5: *a) Microstructure of primary recrystallized Si-steel sheet, as measured by automatic orientation mapping by EBSD in the SEM. The map consists of 1000 × 1000 measurement points. b) Correlation between angular deviation to the Goss orientation and the grain size for all grains of figure a). The lines mark 10° deviation to the Goss orientation and the double average grain size. Grains in the lower right quadrant are possible Goss nuclei.*

department of materials technology, is used. This instrument is equipped with the software TOCA (*S. Zaefferer*) for on-line crystallographic analysis. Furthermore, the latest X-ray goniometer of Bruker Axs, equipped with capillary beam guide for high X-ray intensity and an area detector (GADDS, Bruker Axs) is available. Alternatively, this instrument can be used with a monochromator set-up for high-precision residual stress measurements.

The *origin of the sharp Goss texture (110)<001>* developing during secondary recrystallization of Si steel sheets is a subject of research since more than 60 years. Nevertheless, no really satisfying solution for the problem has been given until now. Particularly, no experimental proofs of the models which have been developed on the basis of statistical observations have been given. In collaboration with Thyssen-Krupp Electrical Steel (TKES) (*N. Chen, S. Zaefferer, D. Raabe, K. Günther*) this problem has been taken up again. The new SEM and X-ray devices acquired in the group, make a more detailed and spatially resolved re-investigation of the origin of the Goss texture possible and large advances could be made. By large area crystal orientation maps the influence of grain topology (grain size and number of next neighbours) on nucleation of abnormally growing Goss grains was tested and some promising results have been obtained. In particular, a “finger-print“ graph showing the relationship between topology and Goss-orientation has been developed [8]. This graph, together with a large orientation map which serves as data basis is shown in Fig. 5. The search for a topological reason of nucleation has lead to the discovery of Goss clusters (clusters of slightly misoriented Goss grains) which might act as nuclei for secondary recrystallization. In order to check their growth behaviour currently an X-ray orientation scanning technique is developed which will allow to detect Goss grains even if they are below the sheet surface.

Various research projects deal with aspects of primary recrystallization in different materials, including IF steels, NiAl and Fe_3Al intermetallics, and aluminium. A study on *the origin of the heterogeneous recrystallization of IF steels* is conducted in collaboration with Thyssen-Krupp Steel (TKS) (*I. Thomas, S. Zaefferer, D. Raabe, F. Friedel*). Using EBSD in the SEM it is tried to understand the

nucleation phenomena in differently oriented crystals in commercial material. This investigations have lead to the discovery of several nucleation sites inside of characteristic orientation gradients [9]. Furthermore, the combination of SEM and TEM investigations has shown large amounts of Ti(N,C) precipitations on grain boundaries. The importance of this particles for the growth behaviour during recrystallization is currently investigated.

In collaboration with Dr. Hirano, NIMS, Japan, an investigation on *the recrystallization mechanisms of heavily deformed NiAl single crystals* is carried out. Here, it seems that preferred growth or secondary recrystallization plays an important role. Furthermore a study on *the origin of the cube texture in aluminium* (*S. Zaefferer*) is conducted. In this case, start material is prepared by equal channel angular extrusion (ECAE) and subsequent recrystallization in order to obtain fine grains and unusual start textures.

Besides investigations on recrystallization mechanisms there are also studies on the micro-mechanisms of deformation. One of these studies (*J.-C. Kuo, D. Raabe, S. Zaefferer, M. Winning*) aims to understand the influence of well defined, symmetric <112> tilt boundaries with various misorientations on the *microscopic and macroscopic deformation behaviour of bicrystals*. To this end a variety of measurement techniques are used: the macroscopic deformation pattern is measured by photogrammetry, mesoscopic and microscopic crystal rotation distributions are determined by EBSD measurements on different scales [10]. It has been found that only very close to the grain boundary (some 10 µm) a change of the bulk deformation behaviour can be detected. In this area, however, a clear difference between large angle and small angle boundaries is visible.

In collaboration with U. Glatzel, Universität Jena, *the phase transformation mechanisms leading to the formation of oxidation protection coatings on Ni-based superalloys* are studied. A crystallographic model for the $L1_2$ - B2 transformation from the Ni_3Al superalloy substrate into the NiAl coating could be proposed based on EBSD measurements. Additional spatially resolved EDX measurements lead to the proposition of a precipitation and diffusion model [11].

A project in collaboration with Prof. Bleck, Universität Aachen, (*J. Ohlert, S. Zaefferer, W. Bleck*) on *the transformation behaviour of TRIP steels* is currently in progress. Here, EBSD and TEM have been applied in order to distinguish the different bcc phases (ferrite, bainite and martensite) and the fcc phase (austenite). It has been found that the ferrite phase can be separated into two groups, one which does not transform at all and one which was created from the austenite during cooling. Bainite can be distinguished by its high content of dislocations, visible in TEM and leading to bad pattern quality in EBSD.

Thermomechanical Treatment

D. Ponge, R. Kaspar

In 2002 three additional projects started so that the activities of the research group of thermomechanical treatment are divided into six projects and some investigations in a direct cooperation with the industry. "*High strength long products with improved toughness and fatigue resistance*" (*S. Detroy, EGKS*) should be obtained by defining a medium carbon steel composition in combination with new processing strategies including full use of a thermomechanical treatment (TMT) by avoiding quenching and tempering. The aim is to achieve a refined multi-phase structure (carbide-free bainite/acicular ferrite with some network of retained austenite) that exhibits the required combination of high strength and improved toughness. After choosing promising steel compositions with increased silicon and manganese contents to obtain a bainitic phase transformation during a continuous cooling special heats will be cast. To guarantee a subsequent industrial application some of the processing parameters (e.g. range of cooling rates) are predetermined by the European industrial partners.

The project "*Optimization the critical contents of trap elements in SiCr high strength steels through thermomechanical processing*" (*A. A. Barani, Arbeitsgemeinschaft industrieller Forschungs-vereinigungen (AiF)*) aims to minimize the negative effects of phosphorous, copper and tin on the final properties of springs used in the automotive industry. Depending on the concentration and the production process the afore mentioned elements can cause two different problems: the temper embrittlement and the liquid copper embrittlement. Within the framework of the project the rolling process and the heat treatment are simulated and the significant parameters are varied to improve the ductility and fatigue properties and maintain the required high strength level. The main interest is to avoid the above addressed embrittlement problems and to correlate the mechanical properties to the tramp element concentration and to the microstructure, i.e. to the austenite grain structure and the martensite morphology. Maximum concentration limits of the tramp elements will be deduced for different processing parameters. Apart from the standard production route the potential of the TMT will be investigated. The goal of the TMT is to achieve a better distribution of phosphorous through the conditioning of austenite in order to avoid its segregation to the grain boundaries.

For the project on "*Optimisation of the process parameters of ferritic rolling of deep-drawable steels*" (*A. Elsner, Verein zur Förderung von Forschungs-arbeiten auf dem Gebiet der Walzwerkstechnik in der Hüttenindustrie (VFWH)*), a commercial IF steel was rolled in the lower ferrite region to produce a "hard" hot strip, exhibiting a strained microstructure. For such

microstructure a supplemental annealing is required to produce a recrystallized strip with good deep-drawing properties. Additionally the two different ferritically rolled products, the "soft" (directly recrystallized in the coil) and the "hard" (additionally annealed) hot strip, were used as an initial strip for a subsequent cold rolling, though using a reduced deformation prior to final annealing. The microstructure and texture investigations together with the measurement of mechanical properties, including the Lankford values, will be used to determine the process parameters necessary to improve the deep-drawing properties of the ferritically rolled strip.

In the project on "*Ultra fine grained steel by innovative deformation cycles*" (*R. Song, EGKS*), microstructures comprising fine ferrite grains and dispersed cementite particles for plain C-Mn steels (with different carbon contents) have been achieved by controlled cooling and heavy warm compression by WUMSI. The final re-coiling has been simulated as an important step in the later large scale industrial production process. The grain boundary character distribution was investigated by using electron backscattering diffraction (EBSD): The fraction of high angle grain boundaries (disorientation angle $\theta \geq 15°$) was found to be approximately 60% in the ferrite. Considering only high angle grain boundaries the mean ferrite grain size was 0.9 µm. If all grain boundaries ($2° \leq \theta \leq 63°$) are considered, the mean ferrite (sub)grain size was 0.6 µm. The hardness increases with decreasing grain size. Dispersed fine cementite particles are useful to prevent ferrite growth. An increase of deformation and/or coiling temperature higher than 600 °C leads to a coarser cementite which is less effective in stabilising a fine grain structure. However, temperatures higher than 650 °C are necessary to obtain a more equiaxed ferrite grain shape and a rather homogeneous distribution of cementite.

In the project "*Transformation behaviour of steel in the in-line hot rolling steel processing*" (*D. Ponge, EGKS*) an efficient laboratory test method for a throughout simulation of the transformation behaviour of steels must be developed for casting and in-line rolling with subsequent cooling. The question whether a remelting of the samples is indispensable or if the as-cast state can be simulated by reheating samples up to high temperatures will be addressed by using the continuous casting simulator and WUMSI. The effect of strain rate on the transformation behaviour will also be investigated.

The project "*Heavy Warm Rolling (HWR) for the production of thin hot strip*" (*L. Storojeva, EGKS*) aims to define processing parameters for the PonyMill concept: a hot strip coil produced on a conventional hot strip mill is to be transferred to a single high reduction rolling stand (PonyMill) where a HWR reduction is to be performed. For optimization of the processing parameters CCT diagrams for the steels with 0.15, 0.35 and 0.67% carbon have been established without and with austenite deformation at the previously optimized temperatures. The study of HWR of steel with 0.35%C was carried out on WUMSI, using different cooling rates prior to HWR as well as two different HWR routes. The optimum processing parameters for this steel were obtained. The microstructure of samples were studied using scanning microscopy and the EBSD method. After the optimum processing parameters the micro-structure consists of spheroidised cementite, which is distributed homogeneously in a recrystallized ferrite matrix with fine grains, Fig. 6.

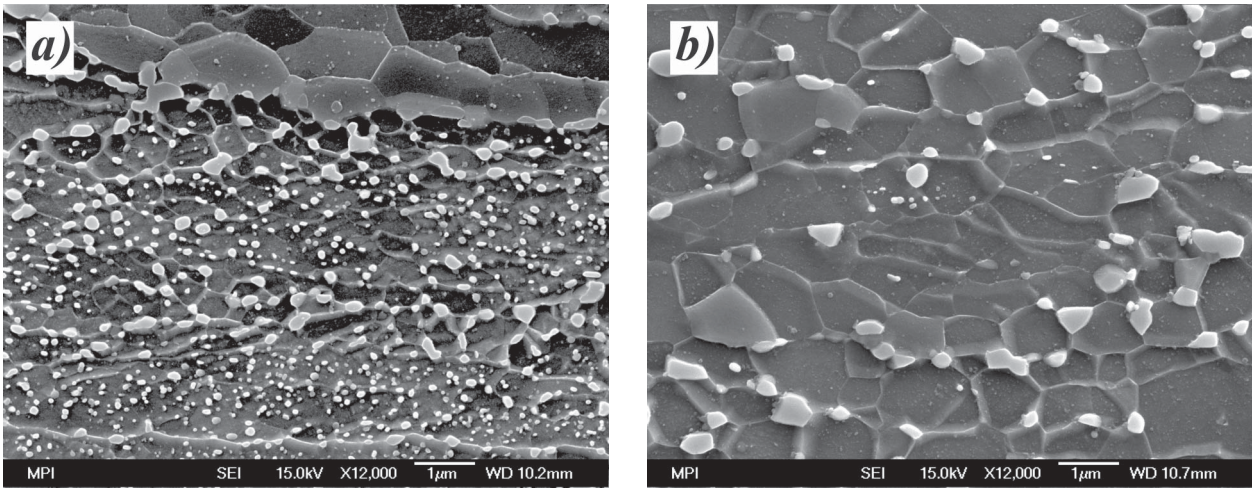

Fig. 6: *Microstructures of the steel with 0.35% C after hot deformation at 900 °C, cooling rate of 10 K/s and heavy warm rolling (HWR) at 550 °C and different re-coiling temperatures:*

a) incomplete spheroidisation of pearlitic cementite and inhomogeneous cementite distribution after re-coiling simulation at 600 °C / 2 h

b) complete spheroidisation and homogeneous cementite distribution in both former pearlite and proeutectoid ferrite regions after re-coiling simulation at 650 °C / 2 h.

Metal Forming

W. Rasp

The final report on the basic concept of *the tribologically asymmetric flat-compression test - a new method to analyse friction during metal forming* (*C. Wolff, DFG, VFWH*) [12] was presented. This test methodology incorporates the high deformation levels, high strain rates, and new surface development, which are present during rolling, into a simple qualitative and quantitative frictional analysis. In this analysis a test specimen is compressed with a drop hammer, using both steel and aluminium test specimens and a series of lubricants with a similar base and varied additives. Different frictional conditions at the upper and lower test specimen-tool interfaces are created by use of different lubricants or different surface preparations. The difference in the frictional conditions between the upper and lower interfaces can be quantified by measuring the out-of-plane-angle, which forms in the workpiece during upsetting. The angle measured during experimentation can be correlated to the actual friction-factor by using either the upper-bound analysis or the finite-element method.

Surface topography, friction, and plasticity form a complex system at the interface of a plastic deformation operation. Surface topography before deformation affects the frictional behaviour at the interface, which in turn affects the level and distribution of plastic deformation. The surface topography after deformation operations is determined by a combination of the initial surface topography, the frictional behaviour at the interface, and the level and distribution of the plastic deformation. This is why the *effects of surface-topography directionallity and lubrication condition on frictional behaviour during plastic deformation* (*C. M. Wichern, EGKS*) [13] are of special interest in the metal-forming group. Friction testing with the Asymmetric Friction Upsetting, AFU, test machine and subsequent surface-topography analysis help to further clarify the relationship between surface topography, interface friction, and plasticity. The frictional conditions during testing are controlled via lubricant viscosity, film thickness, surface roughness, and deformation velocity. High and low viscosity lubricants are used in conjunction with a range of test speeds to produce frictional conditions in the boundary, mixed, and hydrodynamic lubrication regimes. The effect of initial surface topography was examined by preparing five different initial specimen surfaces and recording the surface topographies before testing. Five surface conditions were used: as received, etched, coarsely ground perpendicular to test direction, coarsely ground parallel to test direction, and polished. A correlation between surface topography directionality and frictional resistance was previously observed by testing specimens with grooves machined into the surface at a range of angles. In the current work coarse grinding has been used in place of machined grooves. The scale of the effect from the surface topography directionality has been compared with the scale of the effect of lubricants, arithmetic roughness value, and friction regime. For aluminium specimens, Fig. 7 demonstrates the development of scratches transverse to rolling direction before (top) and after upsetting (middle). The test was performed fairly fast (4.5 m/s) with a lubricant so that the friction regime is dominated by hydrodynamic lubrication. It results in an increase of the roughness and a dramatic change of the slope of the bearing area depicted in the Abbott curve (bottom).

Three-dimensional non-contact surface profilometry was used to measure the voids on the surface of test specimens before and after Asymmetric Friction Upsetting, AFU. By *using the aspect ratio of surface void-volumes to quantify surface-topography directionality* (*C. M. Wichern, EGKS*) [14] the lubrication regime in the die-workpiece interface has been described. A key component in the quantification of the surface voids is the aspect ratio of the individual voids or the Volumetric Aspect Ratio, VAR. The VAR quantifies a surface's ability to draw lubricant into the roll gap and subsequently trap it there. Directionally coarsely ground surfaces were AFU tested using different lubrication conditions and strain rates. Before and after AFU testing the aspect ratios of the surfaces subject to these different testing conditions were calculated. Using the surface topography measurements and the calculated VAR, predictions of the lubrication regime at the die-workpiece interface were made for different process parameters. These predictions regarding the lubrication regime were compared with the friction factor m, measured during AFU testing. Results show that the VAR changes drastically during deformation in the friction regimes tested. The effect of the VAR on the frictional characteristics of the surface varies dependent upon the friction regime. For boundary lubrication the VAR appears to have an effect on the friction properties of the surface, however, during hydrodynamic and mixed lubrication the VAR seems to have very little effect.

The *improvement of formability by superimposed hydrostatic pressure* (*J.-M. Jamet, DFG*) [15] is performed by a simltanuous deformation of a brittle core material and a ductile shell, which surrounds the core. The deformation of the shell equals a foreward-extrusion process. By varying the shell material and the deformation degree the hydrostatic pressure acting against the core material can be increased up to the limits of the tool and the press. With this method the deformability of rather brittle core materials can be studied. Accompanying FEM calculations with the program DEFORMJ allow to get insight into the local strain and stress state of the

process. Whereas the strain state can be verifyed fairly well by experiments, the deformation forces resulting from the stress state are underestimated by far. Reason for that is the uncertain knowledge of the flow curve of the hard-to-deform core material at higher deformation degrees. First attempts to deform intermetallics as representitives of very brittle materials seem to be successful.

The objective of the work *on the forming limit diagram, von Mises equivalent strain, strain path, and surface roughness* (*C. M. Wichern*) [16] is to provide some insight into the complex relationships between formability, strain level, strain path, and roughness in sheet metals. A series of sheet steel specimens were strained along eight different strain paths and the strain and roughness were measured at the identical positions on the specimens. Plots of roughness versus strain provide the roughening rate for the various strain paths. The highest roughening rates occurred closest to plane-strain strain path, which also had the lowest forming limit. For the uniaxial and biaxial strain paths the formability was greater, the rate of roughening was less and the level of roughness achieved was higher as compared to the plane-strain strain path. Swift's analysis is used to show that higher roughnesses can be achieved with the uniaxial and biaxial strain paths because the sheet is less sensitive to thickness related instabilities. Hence, higher forming limits can be reached in uniaxial and biaxial strain paths, with higher roughnesses as compared with the plane-strain strain path.

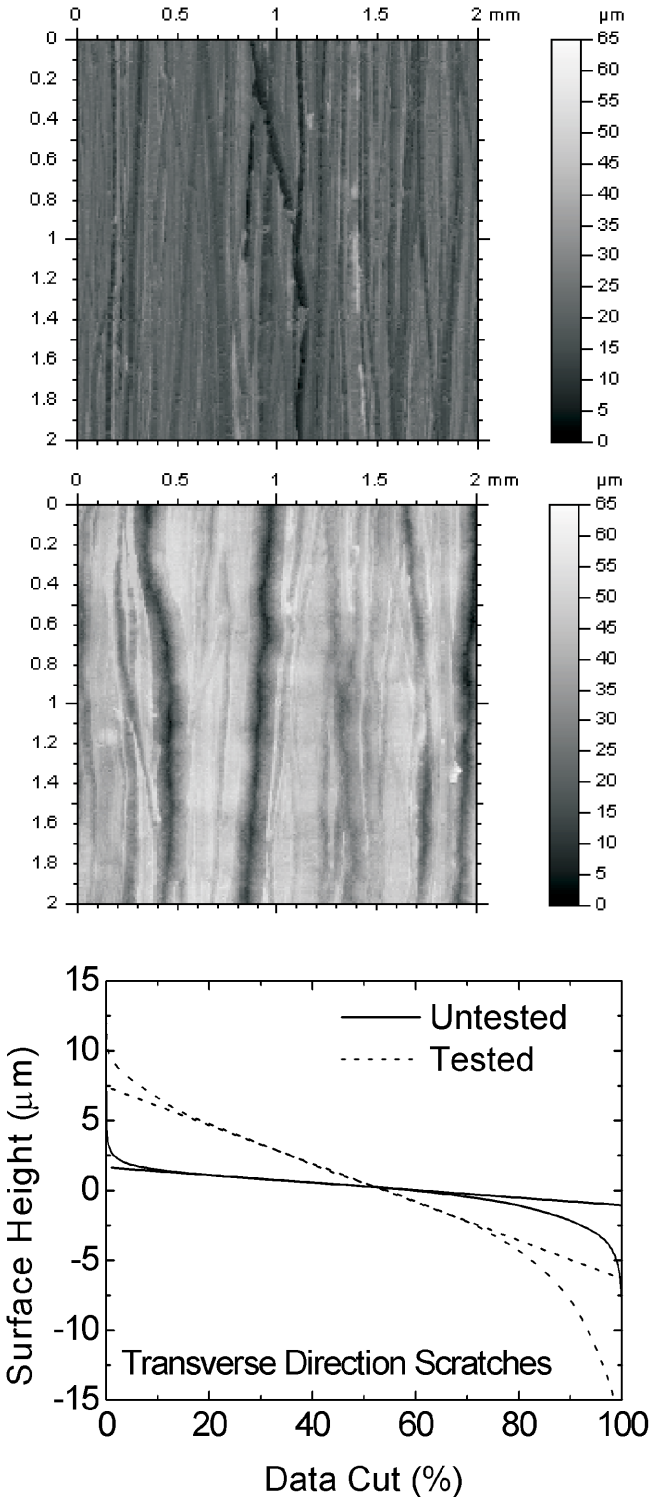

Fig. 7: *Hydrodynamically dominated friction regime of scratches arranged transverse to rolling direction before (top) and after upsetting (middle) with the related bearing-area curve (bottom).*

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Department of Physical Metallurgy

P. Neumann

Scientific Concepts

The macroscopic properties of steels are determined to a large extent by their microstructure down to configurations and defects on the atomistic scale. These property-microstructure relationships are studied in the department.

Steels with complex property profiles require the addition of many alloying elements. The determination of *phase equilibria in such multi-component systems* is an important aspect of our studies. With the help of suitable models for the thermodynamic description of multi-component systems the phase equilibria are calculated numerically with high reliability. In this way the number of experiments, which are necessary to characterise the alloys, can be reduced dramatically. The complexity of metallic alloy systems with their variety of different phases are an almost inexhaustible reservoir for new materials developments. However, such a complexity impedes their quantitative predictability. Therefore it has been and still is an important task of the department to foster the knowledge of the thermodynamic description of the most important metallic alloy systems by using fundamental models of solid state physics as well as by experimental thermodynamic characterisation.

The *kinetics of phase transformations* in multi-component systems is even more complex than phase equilibria. This yields an unbelievable multiplicity of reactions. As an example, the reaction rate can be changed by orders of magnitude by changing the temperature of heat treatment by a few tens of degrees or by adding small amounts of alloying elements below a tenth of a percent. Computer simulations help to predict the development of transformations in time and allow to determine the local distribution of elements and phases down to microscopic dimensions. Due to the multitude of systems, this approach is an important task for the future.

New demands (applications at higher temperatures, light-weight constructions) require continuous development of *new materials*. As an example, the improvement of the thermodynamic efficiency of energy conversion is limited by the high-temperature properties of materials with sufficient toughness. In order to obtain a good combination of the creep resistance of ceramics and the toughness of metals, intermetallic phases are promising candidates. Aluminium or silicon forms dense oxide scales and thus provides a good oxidation resistance of respective alloys. Thus intermetallic alloys on the basis of the aluminides of titanium, iron and nickel and a number of silicides are most attractive for high-temperature applications. Such alloy systems offer a multitude of alloying possibilities which opens a wide field for future research, which is focussed on the design of novel materials with optimisation of strength, toughness and hot-corrosion resistance by control of alloy composition and phase distribution.

Experimental techniques: Alloys to be studied are routinely prepared by induction melting. If high-purity alloys are needed, an inductively heated levitation melting unit is used, and high-melting alloys (up to about 2200°C) are prepared by arc melting. The different methods of alloy preparation and the growth of single crystals are described in the chapter on the materials preparation facilities in the first part of this report.

The evolution of microstructures is analysed by use of electron-probe microanalysis (EPMA), X-ray diffraction (XRD) at ambient and high temperatures, light optical microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). In addition, phase transitions are identified by high-temperature differential thermal analysis (DTA) and oxidation kinetics are observed by high-temperature thermal gravimetry (both up to 1700 °C).

The mechanical behaviour is analysed by computer-controlled testing in compression, bending and/or tension at temperatures in the range -180 °C - +1600 °C in air, inert gas or vacuum at deformation rates in the range 10^{-9} - 10^{-2} s^{-1} with loads in the range 20 - 500 kN. Information on the local deformation behaviour is obtained by computer-controlled microhardness testing, which supplies data not only on hardness of grains and phases, but also on elasticity and ratio of plastic and elastic work. With brittle materials indentation cracking occurs by hardness testing which allows toughness determination.

High-Temperature Materials

G. Sauthoff

The work of the group on the physical mechanisms of phase transformations and phase reactions on the one hand and of deformation on the other hand in multinary multi-phase alloys at high temperatures with particular emphasis on the mutual interaction of phase reactions and deformation processes is continued. Deformation during thermal treatments can be used for controlling nucleation, growth and coarsening of strengthening phases in view of optimisation of microstructure by alloy preparation with thermomechanical processing. Structural high-temperature alloys under service conditions with mechanical loading deform by creep and age with coarsening of grains and second phases. Creep and ageing occurs simultaneously with mutual interaction of the concurrent processes. The aim is to establish a firm physical basis for developing new structural materials for applications at high temperatures above those of existing alloys. The interest is focussed on alloy systems with intermetallic phases, which excel by their high strength at comparatively high temperatures [1-3].

A most attractive intermetallic phase for high-temperature applications is the nickel aluminide NiAl with cubic B2 structure, which excels by its high oxidation resistance whereas the low-temperature ductility and the strength above 1000 °C are insufficient. Proper alloying of NiAl with Ta and Cr produces a three-phase NiAl-base alloy with strengthening second phases which are a coarse brittle Laves phase and a fine Cr-rich phase. An optimum balance of high creep strength above 1000 °C (up to about 1250 °C) and tolerable brittleness at room temperature could be achieved by optimising phase compositions and distributions. This gave rise to the successful development of the novel intermetallic NiAl-Ta-Cr alloy IP75, which shows a high oxidation and hot-corrosion resistance besides attractive mechanical properties and is ready for specific application-oriented developments e.g. for application as combustor panel material in industrial gas turbines (*M. Palm, BMBF support*) [4-12].

The strength of the IP75 alloy relies on the ternary Laves phase TaNiAl, which is one of a long series of hard and brittle ternary Laves phases with transition metals and Al with hexagonal C14 or C36 structure or cubic C15 structure depending on composition and temperature [13]. This variation of crystal structure with composition has been studied in detail in the ternary Fe-Al-Zr system. It was found that the ternary Laves phase $Zr(Fe,Al)_2$ shows repeated C14/C15 phase transitions with variation of the Fe:Al ratio - see Fig. 1. Thus, this Laves phase forms manifold phase equilibria with various disordered or ordered phases which allows the design of multi-phase Fe-Al-Zr alloys with broadly varying mechanical properties. However, the oxidation resistance of such alloys deteriorates with increasing Zr content (*F. Stein, M. Palm, DFG support*) [14-24]. This work has been accompanied by various other studies on the phase

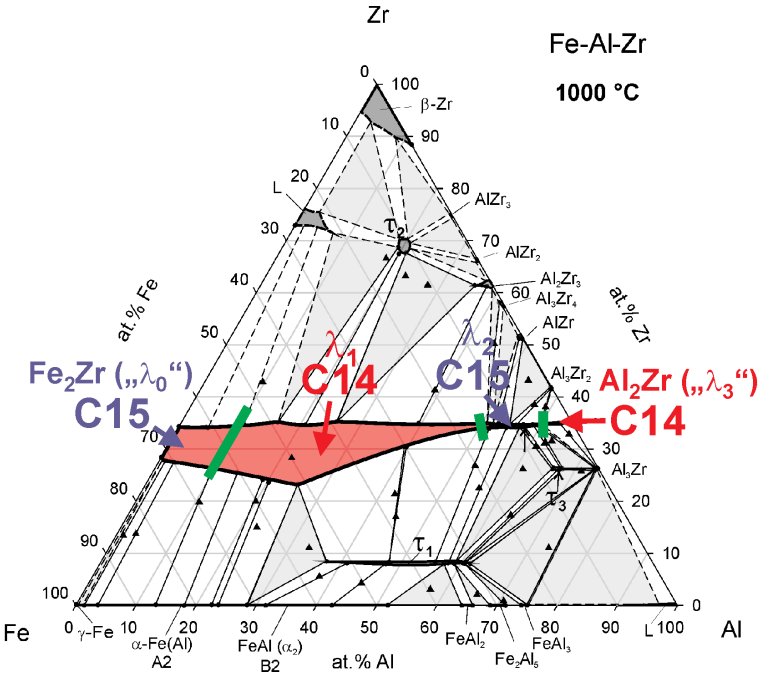

Fig. 1: *Isothermal section at 1000 °C of the ternary Fe-Al-Zr system with focus on the C15/C14 phase transformations in the $(Fe,Al)_2Zr$ Laves phase.*

relations and equilibria in related ternary systems for establishing more reliable phase diagrams and characterising basic mechanical properties [25-30].

Fe-Al-base alloys are of very high interest for high-temperature applications in view of their excellent oxidation and hot-gas corrosion resistance. However, their high-temperature strength is insufficient. Fe-Al alloys may be strengthened by atomic ordering, which, however, affects ductility and toughness [31-33]. Apart from alloying with Zr, ternary Fe-Al systems with other transition metals offer various promising possibilities for strengthening by precipitation of hardening intermetallic phases, which is in the focus of new projects. Appropriate alloying with Ni produces precipitate particles of the intermetallic NiAl phase in a bcc Fe-Al matrix (*Ch. Stallybrass, DFG support*). These precipitate particles are spherical or cuboidal and coherent or semicoherent depending on volume fraction and growth stage. Thus, the microstructure of these Fe-Al-Ni alloys is analogous to that of the

well-known fcc Ni-base superalloys, i.e. these alloys with bcc matrix and B2 precipitate may be regarded as ferritic Fe-base superalloys. Such ferritic alloys are expected to show lower thermal expansion and higher heat conductance than fcc Ni-base alloys and austenitic steels and thus are advantageous for applications in conventional power plants and other energy conversion units.

For Fe-Al alloys with highest possible strength at high temperatures the Fe-Al matrix has to be hardened by precipitation of an intermetallic phase with highest possible hardness. Alloying with Ta produces the ternary Laves phase $Ta(Fe,Al)_2$, which indeed shows the highest hardness of such ternary Laves phases with transition metals and Al [13]. This ternary Laves phase - as well as the analogous phase $Nb(Fe,Al)_2$ - forms stable equilibria with Fe-Al alloys with an extended miscibility gap in the phase diagram, which allows the variation of both the Al content and the Ta content within wide limits. The former controls the atomic order in the Fe-Al matrix and the latter controls the volume fraction of the Laves phase. Both effects are used to optimise the ratio of strength and ductility, which is studied in close cooperation with the Development and Characterisation of New Materials Group of Dr. Schneider (*D.D. Risanti, DFG support*) [34-38].

This work follows the example of the additional strengthening of new creep-resistant martensitic/ferritic 12% Cr steels by precipitation of the WFe_2 Laves phase. The creep resistance of such steels usually relies on strengthening $M_{23}C_6$ and MX carbides and/or carbonitrides, which allow service temperatures up to about 600 °C e.g. in conventional power plants. They coarsen during service, thus deteriorating the creep resistance. Additional Laves phase precipitation is to improve the creep resistance to such an extent that service temperatures of up to 650 °C are possible (Fig. 2). Laves phase precipitation is a slow process compared to carbide precipitation. The kinetics of the various precipitation processes are to be adjusted by balancing the alloying additions and optimising the pre-treatments in close cooperation with the Phase Equilibria and Transformation Group of Prof. Inden in such a way that Laves phase precipitation occurs during service to compensate for the deterioration effect of carbide coarsening (*V. Knežević, DFG support*) [39-43].

Besides strength, ductility and oxidation resistance, low density is an important issue for developments of new light-weight materials [44]. In view of the favourable mechanical behaviour of the familiar intermetallic γ-TiAl + Ti_3Al alloys with lamellar microstructures, work has been focussed on Al-rich Ti-Al alloys, for which a still lower density and a higher oxidation resistance is expected and for which the feasibility of the generation of lamellar microstructures with the Al-rich γ-TiAl phase has been shown [45]. For clarifying the phase equilibria and reactions, Al-Ti alloys with 58 to 73 at.% Al and $TiAl/Al_3Ti$ diffusion couples were investigated in detail using electron-probe microanalysis (EPMA), transmission electron microscopy (TEM), X-ray diffraction (XRD), and light optical microscopy together with differential thermal analysis (DTA) studies of phase transitions. According to the newly established phase diagram there is a continuous solid-solubility range of the γ-TiAl phase

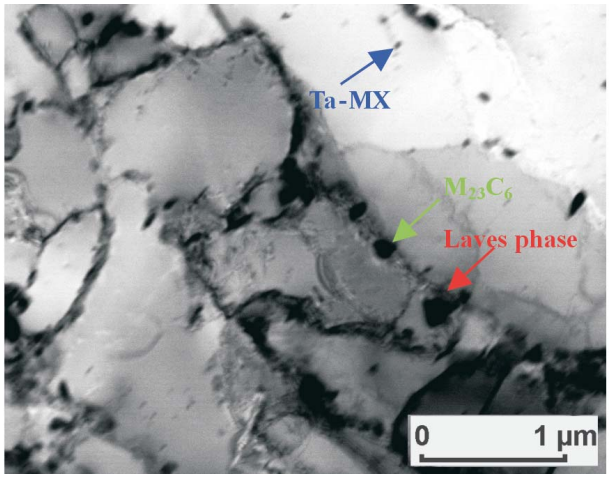

Fig. 2: *Transmission electron microscopy micrograph of a martensitic/ferritic 12% chromium model steel variant with additional 3% tungsten and 0.2% tantalum for precipitating fine Ta(C,N) carbonitride particles and coarse $M_{23}C_6$ carbide and WFe_2 Laves phase particles after creep.*

between about 48 and 71 at.% Al above 1215 °C which is the predominant feature of the Al-rich part of the Al-Ti system [46]. Below 1215 °C the γ-TiAl phase is no longer stable in this composition range and transforms to various Al-rich phases depending on composition with crystal structures which all derive from the $L1_0$ γ-TiAl structure by ordering of excess Al atoms in the (002) Ti layers. TEM and DTA studies on samples with various annealings revealed that Al_5Ti_3 and h-Al_2Ti are non-equilibrium phases [47]. Al_5Ti_3 is a metastable phase at low temperatures, which disintegrates irreversibly on heating between 700 °C and 900 °C depending on composition. h-Al_2Ti transforms to the equilibrium phase r-Al_2Ti by two very slow reactions. The mechanisms for both transformations, a continuous and a discontinuous one, have been identified, the latter leading to the formation of fine lamellar γ-TiAl + r-Al_2Ti microstructures [48]. A first Ti-62 at.% Al alloy with lamellar γ-TiAl + r-Al_2Ti microstructure shows a 0.2% yield stress and a creep resistance which are similar to those of the familiar Ti-48Al-base alloys, and a more favourable oxidation behaviour is observed [49]. Such lamellar Al-rich TiAl alloys are now being optimised with respect to strength, ductility and oxidation resistance (*M. Palm, F. Stein*).

Phase Equilibria and Transformations

G. Inden

The properties of materials are controlled by microstructures. Any materials development and optimisation thus has to deal with the kinetic path a system takes to form a given microstructure. This kinetic path strongly depends on the thermodynamic properties of a system which also define the final state the system will attain at equilibrium. Today this thermodynamic information is made available in terms of databases and software which provide the thermodynamic properties by computer calculation. Complex multi-component systems like steels can only be accessed by numerical techniques.

Although a large amount of information has been acquired during the last two decades there are still enormous gaps, particularly when it comes to new developments such as super heat-resistant ferritic steels for applications in Ultra Super Critical Power Plants at temperatures above 650 °C. In order to reach sufficient strength and creep resistance one has to open the development to new enforcing phases like Laves phases, μ-phases, MX carbonitrides. This in turn introduces new additional elements like Ta, Nb, W, V. For these high melting elements there exists virtually no experimental information about multi-component phase diagrams. In the current databases the thermodynamic description of the mentioned phases does not even contain the element Ta. Consequently, a joint project, funded by Nato, was defined together with Prof. A. Kroupa at the Institute of Physics of Materials, Academy of Sciences, Brno/ Czech Republic, with the aim of developing a database for this class of materials. A first version based on the scarce existing data has been completed (*J. Balun, P. Unucka/Brno*). It is planned to launch a new experimental project in this area to provide new data. These activities are embedded in the German cooperative research project on ferritic steels. One issue of this work is to obtain a thermodynamic description of the phases which is consistent with the crystallographic properties and allows for off-stoichiometry. This is a prerequisite for any application in the field of phase transformations.

In the same spirit an experimental study of the system Fe-Rh-Ti has been performed providing new and very interesting information [50, 51] (*J. Balun, L. Eleno, C.G. Schön/São Paulo*). In this instance all three binary subsystems exhibit strong ordering tendencies leading to the formation of very stable bcc ordered compounds of type B2. Although all three subsystems are of ordering type, i.e. with strong bonding energies between unlike atoms, a wide miscibility gap opens up in the ternary system. This gap is not caused by an atomic separation tendency, it comes from the difficulty of building configurations with maximum number of unlike bonds of each kind. This result is particularly interesting since it allows the formation of heterogeneous microstructures with coherent ordered phases just as in the case of superalloys. Based on these experiments a thermodynamic dataset was established trying to calculate the phase equilibria by two approaches, the CALPHAD approach with fitted thermodynamic functions and a statistical mechanics approach with the Cluster Variation Method (CVM) using cluster interactions. The CVM approach permitted to calculate a series of prototype diagrams in order to find out which parameters determine the position of the miscibility gap [51]. The boundary of the miscibility gap A2+B2 and the slope of the tie-lines are well reproduced by the calculation. The work continues to get similar good agreement at the Ti corner of the system. The energy parameters obtained from this study will then be used to improve the thermodynamic functions of the CALPHAD approach and to put the parameters on a more physical basis.

The statistical mechanics approach including both CVM and Monte Carlo simulations is also applied to magnetic systems [52-55] (*C.G. Schön/São Paulo*). The emphasis is on high spin numbers (up to spin 15/2) relevant in systems with heavy rare earth elements like Gadolinium. At present the effect of external magnetic fields is analysed. The first results yield a surprisingly strong effect on the shape of the magnetic specific heat capacity at both sides above and below the Curie temperature. This offers new possibilities for microstructural modifications using pulsed fields in addition to traditional heat treatments.

The computational thermodynamics made available by the CALPHAD approach is one major input in the numerical simulation of phase transformations. The simulations not only provide an insight into the transformation mechanism, into growth of stable and metastable phases and their competition, they also allow the prediction of long term stability under service conditions [56]. This is again of paramount importance for high temperature materials like ferritic steels which have to maintain good strength and creep properties over years and even decades. The simulation of diffusion controlled transformation is done using the software DICTRA. Microstructure formation can be simulated defining different cells, each of which containing a different growing phase. The cells are coupled by different choices of boundary conditions. An example of competing reaction is shown in Fig. 3 for a ferritic steel Fe-12Cr-0.1C. The growth of $M_{23}C_6$ and M_7C_3 carbide is simulated within two ferrite cells coupled by equal chemical potentials at the cell surfaces. M_7C_3 is not a stable carbide in this system. Yet it grows simultaneously with $M_{23}C_6$ at the beginning of the reaction. It disappears eventually when equilibrium is approached (*A. Schneider*).

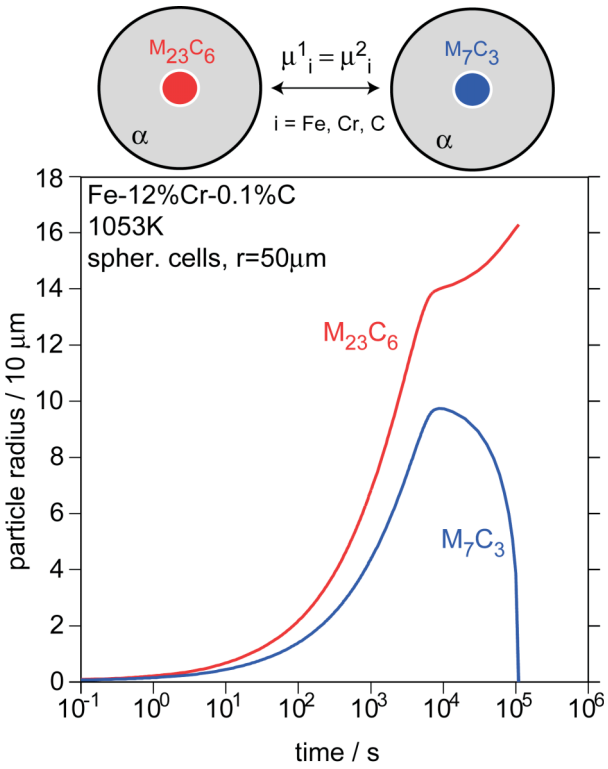

Fig. 3: *Simultaneous growth of $M_{23}C_6$ and M_7C_3 carbide in a ferritic Fe-12Cr-0.1C steel. Growth is simulated by regarding two ferrite cells that are coupled by supposing equal chemical potentials at the cell surfaces. In this system M_7C_3 is only a metastable carbide, which disappears at later stages of $M_{23}C_6$ growth.*

The treatment of diffusion controlled transformations requires boundary conditions at the moving interface. It is common practice to assume local equilibrium which has proven to be fulfilled in many cases of practical interest. However, there are other alternatives possible or necessary when it comes to finite interface mobility, solute drag and solute trapping. Experimental and theoretical studies are currently performed in order to get more information on the conditions adopted by real systems (*Y. Bréchet, C. Hutchinson/ Grenoble*).

There are two major ingredients entering the treatment of transformations: thermodynamics defining the driving forces, and kinetic parameters like atomic and interfacial mobilities. The kinetic parameters have to be derived from experiments like growth kinetics and concentration profiles within growing phases. Experimental problems arise in the case of extremely small precipitates like carbides. A way out of such problems is to control the growth by appropriate gas atmospheres. Experiments are currently performed for cementite using high carburising atmospheres. The effect of alloying elements on the growth kinetics of cementite is studied using diffusion couples. A large range of compositions can then be analysed in one experiment. The effect of gas composition on growth kinetics is also analysed providing, as a side branch, further insight into the effect of metal dusting. Fig. 4 shows the topology of graphite forming at the surface of cementite under high carburising atmosphere. This corrosion phenomenon gains increased actuality due to the intended drastic reduction of sulphur content in fuel oil. By means of alternated carburisation and decarburisation experiments new information on the mechanism of formation and decomposition of cementite was obtained [57, 58] (*A. Schneider, J. Zhang*).

Finally, general topics out of the field of phase equilibria and transformations have been treated, such as atomic ordering [52] or the treatment of surface tension in multicomponent systems [59].

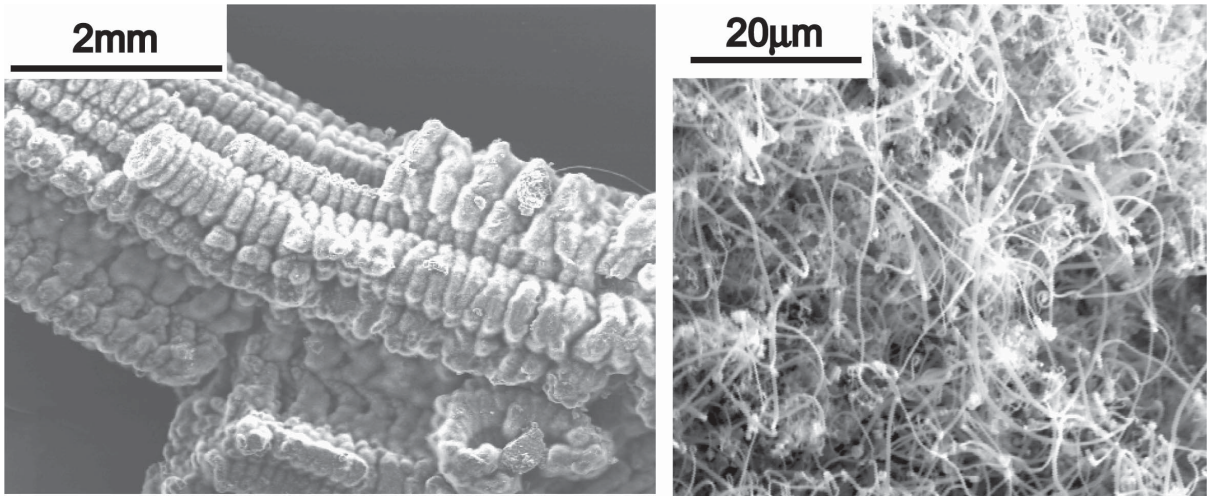

Fig. 4: *Graphite formed after 456h carburisation at 650°C under 0.3%H_2-15%CO-0.7%H_2O at the surface of Fe-26at% Al-2at%X (X=Nb, Ti, Ta, or V):*
a) macroscopic morphology, b) higher magnification: cementite particles become visible at the end of the graphite filaments.

Mechanical Properties
P. Neumann

Grain boundaries are important constituents of the microstructure of most materials. Besides changes in the atomic structure and chemical composition, the mechanical stress state at a grain boundary is different from that in the interior of the grain. Therefore, it is quite common that local damage occurs first at grain boundaries, i.e. there is intercrystalline fracture or corrosive attack. The effects of orientation difference on atomic structure, on grain boundary energy, and on chemical segregation have been studied extensively. However, the peculiar conditions for the stress state at a grain boundary in elastically anisotropic crystals has not been studied analytically because of the complexity of the underlying equations.

Even in cubic crystals, which have the highest possible symmetry, elasticity is not necessarily isotropic, since it is a fourth order tensor. Elastic moduli do vary typically by a factor of two to four in common metals. Therefore, even in the elastic regime differently oriented grains deform differently under the same applied stress. Since compatibility at the grain boundaries is maintained, stress discontinuities will usually occur at most grain boundaries (Fig. 5). Modern finite element programs, which are employed in micro-mechanical studies, are capable of incorporating anisotropic elasticity and can calculate the resulting stress discontinuities. However, no general analytical solution for compatibility stresses at grain boundaries apparently exists in the literature. In the recent year the analytical solution for the important class of 180° twist boundaries, which include all symmetrical tilt boundaries, was obtained by Neumann [60]. The general case is somewhat involved, but for simple cases like the symmetrical twin boundaries in fcc and bcc lattices the solution is of utmost simplicity and can explain many observations of the dependence of fatigue crack initiation at certain grain boundaries on the grain boundary character and orientation.

In texture research the orientation distribution function (ODF) of polycrystals is almost exclusively presented and discussed in the space of the three Euler angles. F.C. Frank, P. Neumann and others have pointed out more than ten years ago that representations of the ODFs in Rodrigues space are superior to those in Euler angle space because of three reasons:

- Each orientation is represented exactly once (in Euler space it is represented three times),

- all fibres and symmetry boundaries are straight lines and planes, respectively (in Euler space both are curved in general),

- the natural distance between rotations R_1 and R_2 (the rotation angle of $R_1^{-1}.R_2$) deviates from their metric distance in Rodrigues space by less than 28% for cubic lattice symmetry (in Euler space the deviation diverges at $\phi = 0$).

It could be shown recently by Neumann [61] that further attractive simplifications can be obtained in Rodrigues space if other essentials of textures are considered like the prevalence of certain orientations („main components“) and fibres, and the determination of averages of orientations. The latter is closely tied in with the peculiar topology of all rotation spaces, which renders certain averages meaningless. These results will help to present texture data in a form that is considerably more transparent than that of Euler angle space.

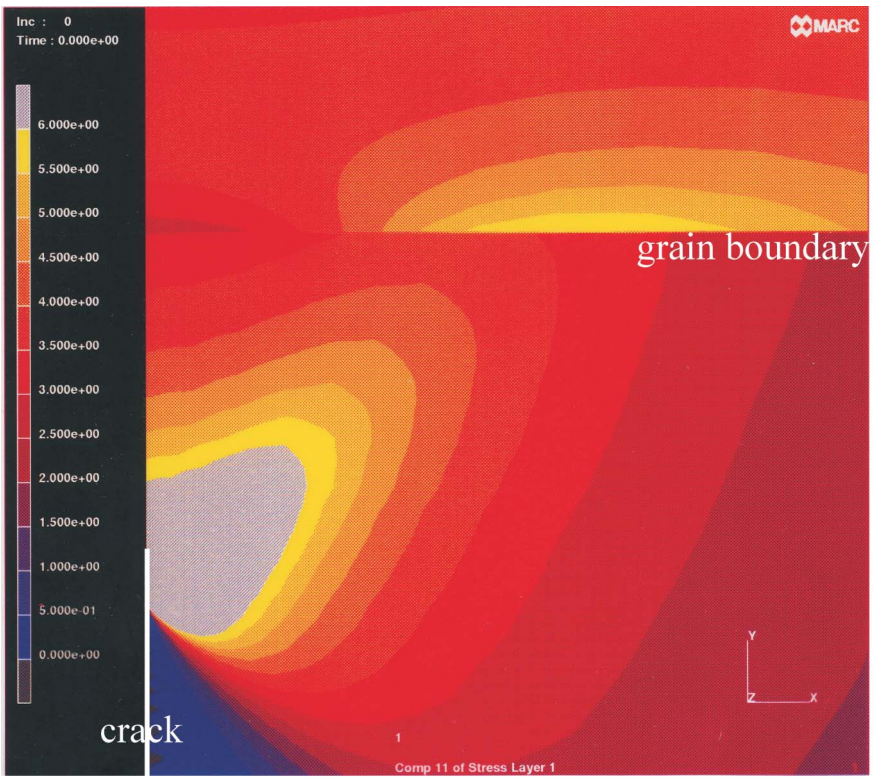

Fig. 5: *The stress field of a crack (loaded by horizontal tension) near a grain boundary. The stresses were calculated numerically assuming anisotropic elasticity. They display a discontinuity at the grain boundary due to the anisotropy of elasticity.*

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PART III.

STATISTICS

(*Note:* The last Status Report only included statistical information up to the end of 1999. To ensure a complete documentation, the following chapter contains the statistical data of the years 2000, 2001, and 2002.)

Supervisory Board

(in 2002)

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RAABE, Dierk, Prof. Dr.-Ing.
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Emeriti:
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PAWELSKI, Oskar, Prof. Dr.-Ing.
PITSCH, Wolfgang, Prof. Dr.

External Scientific Member:
HILLERT, Mats, Prof. Dr., Stockholm/
Sweden

Scientific Honours

Prof. Dr.-Ing. G. Frommeyer was nominated to a member of the ”Japan Prize“ selection committee by “The Science and Technology Foundation of Japan“ in 2000.

Prof. Dr.-Ing. G. Frommeyer became member of the consulting committee of the “International Space Station ISS” in 2001.

Prof. Dr.-Ing. G. Frommeyer and Dr.-Ing. O. Grässel received the “Steel Innovation Award“ for their outstanding work on new classes of TWIP/TRIP steels in 2000.

Prof. Dr. H.J. Grabke received the title “Honorary Member of the European Federation of Corrosion”, in recognition of his outstanding contribution to the achievement of the aims of the Federation, in particular during his thirteen years as Chairman of the Working Party on “Corrosion by Hot Gases and Combustion Products“, on the occasion of EUROCORR 2001 in Riva del Garda, Italy, 01 October 2001.

Prof. Dr. H.J. Grabke received the “Adolf-Martens Medaille” of the “Arbeitsgemeinschaft Wärmebehandlung und Werkstofftechnik” in recognition of his outstanding scientific efforts in the field of heat treatment. The award was handed over at the Härterei-Kongress in Wiesbaden, 10 October 2001.

Prof. Dr. H.J. Grabke received the “Best Paper Award – STEEL RESEARCH 2001” together with *Dr. E. Riecke* and *Dr. F. Gehrmann* for the paper “Hydrogen in Microalloyed Steels”, steel research 72 (2001) 225-235 on the occasion of the 17. Aachener Stahlkolloquium, 23-24 May 2002.

Dr.-Ing. O. Grässel received the “Wissenschaftspreis des Wissenschaftszentrums Nordrhein-Westfalen” for his outstanding doctoral thesis in July 2001.

Dr. G. Grundmeier has been empowered by the Christian Doppler Forschungsgesellschaft in Austria to build and to manage a Christian Doppler Laboratory starting 1 January 2003.

Prof. Dr. G. Inden was elected Honorary Member of the Société Française de Métallurgie et de Matériaux (SF2M) at the Annual Meeting in Paris in November 2001.

Prof. Dr. G. Inden was invited by Fundação de Amparo à Pesquisa do Estado de São Paulo” (FAPESP, “São Paulo State Research Funding Agency“) for a two months research stay at the Escola Politecnica da Universidade de São Paulo/Brazil (Sept./Oct. 2001).

Prof. Dr. G. Inden was invited by CNRS (Centre National de la Recherche Scientifique) for a 6 months stay at the Laboratoire de Thermodynamique et Physico-Chimie Métallurgique at the Institut National Polytechnique de Grenoble/France (2002/2003).

Dr. A. Leng, *Dipl.-Ing. H. Streckel* and *Prof. Dr. M. Stratmann* received the “T.P. Hoar Award” for their publication “The Delamination of Polymeric Coatings from Steel. Part I : Calibration of the Kelvin Probe and basic delamination mechanisms”, Corrosion Science 41 (1999) 547-578, in August 2002.

Prof. Dr. P. Neumann received the “Heyn-Denkmünze” of the German Society of Materials Science (DGM) on 12 July 2001.

Prof. Dr. D. Raabe received the “Materials Science and Technology Price of the Federation of European Materials Societies (FEMS)“ in Rimini/Italy, June 2001.

Prof. Dr. D. Raabe received the “Dr. Meyer-Struckmann-Wissenschaftspreis 2001“ of the Brandenburgische Technische Universität Cottbus, December 2001.

PD Dr. M. Spiegel was honoured with the “Friedrich-Wilhelm-Bessel-Preis” of RWTH Aachen for outstanding scientific efforts on 29 November 2002.

Dr. S. Zaefferer was awarded the “Young Scientists Award“ of the first joint conference on recrystallization and grain growth, Rex & GG 2001, in Aachen.

Dr. S. Zaefferer received the “Masing-Gedächtnispreis 2001” of the Deutsche Gesellschaft für Materialkunde.

Participation in Research Pogrammes

National:

BMBF

Grenzflächenhaftung in technischen Systemen:

“Long-term stability of high performance adhesives“

“Release systems for the self-healing of polymer/metal interfaces”

“Surface tailoring of materials by atmospheric pulse plasmas”

MaTech:

“Novel protective coatings for steel strip: Investigation and preparation of innovative coatings and coating systems”

“Water as a medium in production, processing and use of plastics – novel polymer anti-corrosion coatings for metal surfaces”

“High strength light-weight materials based on iron-aluminium alloys“

“Intermetallic NiAl components for non-polluting energy conversion - Materials characterisation and optimisation”

“Development and characterisation of high strength and supra ductile TRIP/TWIP – light-weight steels based on Fe-Mn-Al-Si”

“The effect of steel making on the distortion of steel components (Vordringliche Aktion 19: Beherrschung von Wärmeprozessen im Fertigungsablauf)”

BMWi

BEO – Förderschwerpunkt Energie:
“Energy-saving production of light-weight steels using DSC-technology”

DAAD

Partnerschaftsprogramm:
“Investigation on superplastic properties of the intermetallic Ti-46AL-1Mo-0,2Si alloy”

DFG

SPP Cellulose und Cellulosederivate: molekulares und supramolekulares Strukturdesign:
“Investigation on the transport behaviour of thin cellulose layers on solid-state substrates”

SPP Hochtemperatur-Oxidation:
“Initial stages of high temperature oxidation: an in-situ STM investigation”

SPP Erweiterung der Formgebungsgrenzen bei Umformprozessen:

"Improvement of the formability of TRIP-, TWIP- and conventional steels by biaxial loading in tension and compression, superposed by torsion"

"Forming by deep drawing and stretch forming of FeMn - TRIP- and TWIP-steels under inclusions of high dynamic deformation conditions and complex diameters"

"Superplastic forming of intermetallics and steels - fabrication and process parameter studies"

"Experiments to improve the formability by superimposed hydrostatic pressure"

SPP Erweiterung der Prozessgrenzen bei der Werkstoffbearbeitung mit Laserstrahlung:

"Modelling of phase formation and solidification structures under non-equilibrium conditions of Ti-Al laser welded seams"

SPP Strukturgradienten in Kristallen:

"Investigations on the structural gradients at face and antiphase boundaries in B2-ordered particle strengthened NiAl (X=Fe, Cr, Mo) solid solution alloys by analytical field ion microscopy (APFIM)"

SPP Modellierung von Größeneinflüssen bei Fertigungsprozessen:

"Investigation of scale influences on texture and anisotropy with regard to the theory of similarity for forming processes"

Normalverfahren:

"Chromium diffusion and initial oxide formation on chromium steels in different surface status"

"Diffusion of interacting lattice gases on heterogeneous surfaces"

"Fundamental aspects of delamination"

"Investigations on chlorine induced high temperature corrosion of model alloys, steels, nickel-based alloys and pure metals"

"Nitration and oxidation of steels in a NH_3H_2O mixture at 300-600 °C"

"Investigations on mechanical properties – strength and plasticity – and characterisation of the microstructures of Fe_3Al based alloys"

"Cementite formation and decomposition during metal dusting"

"Constitution and thermodynamics of the ternary systems Fe-Rh-X (X=Co, Ti)"

"Constitution of cementite"

"Development of ferritic iron-aluminium-tantalum alloys with strengthening Laves phase for maximum creep resistance in corrosive atmospheres"

"Hardening of novel iron-chromium-aluminium-nickel alloys by coherent precipitates"

"Influence of carbide forming elements on the metal dusting process"

"Structure and deformation behaviour of Fe-Al-Zr alloys with strengthening intermetallic phases for high-temperature applications"

"Super heat resistant ferritic steels - alloy development, alloy preparation and long-term behaviour"

"Super heat resistant ferritic steels - thermodynamics and kinetic simulations of precipitation reactions"

"Heat transfer into the rolls in thin strip casting"

"Strip quality and melt supply system in twin roll thin strip casting"

"Influence of machining with geometrically defined cutting on material properties of NiAl and TiAl aluminides"

"Experimental and theoretical investigation of strain and orientation gradients in steel at the grain scale"

"Thermomechanical processing of near-net-shape cast directly rolled steel strips of the as-cast thickness of 5-20 mm"

International:

EGKS

"A mechanistic study of wetting and dewetting during hot dip galvanizing of high strength steels"

"Development of electrically conductive polymer coatings for coil coated steel sheets"

"Investigations of damaging mechanisms of coil-coating steel sheet during forming in order to enhance the performance predictability of finished parts"

"Passive/active transitions in cyclic corrosion tests"

"Soluble salt contamination on blast cleaned surfaces and the effect on the durability of subsequently applied paint systems"

"Stainless steels for molten carbonate fuel cells"

"Study of the reoxidation and ignition behaviour of sponge iron to improve safety in storing and transport"

"Tailored thin plasma polymers for surface engineering of coil coated steel"

"Determination of high temperature surface crack formation criteria in continuous casting and thin slab casting"

"Improving surface quality of continuously cast semis by an understanding of shell development and growth"

"Improving the properties of near net shape cast strip containing copper and thin from scrap"

"Strand reduction in slab casting and its effect on quality"

"The formation and prevention of scale during twin roller strip casting"

"Advanced modelling of lateral flow and residual stresses during flat rolling"

"Assessment and synthesis of the most relevant tribological tests for the characterization of cold rolling lubricants"

"Heavy warm rolling for the production of thin hot strips"

"High strength long products with improved toughness and fatigue resistance"

"Transformation behaviour of steel in the in-line hot rolling processing"

"Ultra fine grained steel by innovative deformation cycles"

EU

Growth:

"European network surface engineering of new alloys for super high efficiency power generation"

"Mitigation of formation of clorine rich deposits affecting on superheater corrosion under co-combustion conditions, target action H – CORBI"

"Optimisation of in-service performance of boiler steels by modelling high temperature corrosion"

Collaborations with Research Institutes and Industrial Partners

Research Institutes

National:

Betriebsforschungsinstitut BFI, Düsseldorf
Dr. E. Neuschütz

Bremer Institut für Strahltechnik BIAS
Prof. Dr. G. Sepold

DECHEMA, Frankfurt
Prof. Dr. M. Schütze

Forschungszentrum Jülich
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Fraunhofer Institut für Angewandte Materialforschung, Bremen
Prof. Dr. O. Hennemann

Fraunhofer Institut für Schicht und Oberflächentechnik, Braunschweig
Prof. Dr. C.-P. Klages

Fraunhofer Institut für Werkstoff- und Strahltechnik, Dresden
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CEIT, San Sebastian, Spain
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Industrial Partners

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BASF AG, Ludwigshafen

Bayerische Motorenwerke AG, München

BÄHR-Thermoanalyse GmbH, Hüllhorst

Buderus Edelstahl AG, Wetzlar

Chemetall, Frankfurt a. M.

CUTEC GmbH, Clausthal

Daimler Chrysler AG, Ulm

Deutsche Nickel AG, Schwerte

Deutsche Titan GmbH, Essen

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dmc² Degussa Metals Catalysts Cerdec AG, Düsseldorf

Dortmunder Oberflächenzentrum GmbH, Dortmund

Doncaster Precisions Castings-Bochum GmbH, Bochum

Ford Werke AG, Köln

Forschungszentrum Jülich GmbH, Jülich

GKN Sinter Metals, Radevormwald

GTT Technologies, Herzogenrath

Gontermann & Peipers, Siegen

Groche & Tilgner GmbH, Kalletal

Hans Goldschmidt Forschungs- und Entwicklungs-GmbH, Essen

H. C. Starck GmbH, Laufenburg

Henkel Oberflächen GmbH, Düsseldorf

Henkel GmbH, Düsseldorf

Houghton Deutschland GmbH, Aachen

Hydro Aluminium Deutschland GmbH, Hamburg

INPROTEC GmbH & Co. KG, Niederkrüchten

Krupp Thyssen Nirosta, Krefeld and Benrath

Mankiewicz Gebr. & Co. (GmbH & Co. KG), Hamburg

Niobium Products Company GmbH, Düsseldorf

Renolit AG, Worms

Robert Bosch GmbH, Stuttgart

Salzgitter Flachstahl AG, Salzgitter

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Profil ARBED, Luxembourg, Luxemburg

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SIDENOR Steel Products Manufacturing Company S. A., Athens, Greece

Treibacher Auermet GmbH, Treibach-Althofen, Austria

Voest Alpine Stahl Linz, Linz, Austria

Voest-Alpine Stahl, Donawitz, Austria

Voest-Alpine Industrianlagenbau GmbH & Co. (VAI), Linz, Austria

VTT Chemical Technology, Espoo, Finland

Patents and Licences

(granted in 2000 to 2002)

Date of Issue	Description	Inventors
14.05.2002	Light constructional steels and the use thereof (US Patent No. 6387192 B1)	Frommeyer, Georg, Prof. (30%) Grässel, Oliver, Dipl.-Phys. (30%) Drewes, Dr., TKS (20%) Engl, Dr., TKS (20%)
07.05.2002	Light weight steels and its use for car parts and façade linings (US Patent No. 6383662 B1)	Frommeyer, Georg, Prof.
15.04.2002	Verwendung von Stahlpulver auf der Basis Fe-Cr-Si für korrosionsbeständige Beschichtungen (Austrian Patent AT 0000215617 E)	Schroer, Carsten, Dr. (30%) Spiegel, Michael, Dr. (20%) Grabke, Hans-Jürgen, Prof. (30%) Sauthoff, Gerhard, Dr. (20%)
03.04.2002	Verwendung von Stahlpulver auf der Basis Fe-Cr-Si für korrosionsbeständige Beschichtungen / Use of Steel Powder Based on Fe-Cr-Si for Corrosion Resisting Coatings (European Patent EP 0933443 B1)	Schroer, Carsten, Dr. (30%) Spiegel, Michael, Dr. (20%) Grabke, Hans-Jürgen, Prof. (30%) Sauthoff, Gerhard, Dr. (20%)
03.04.2002	Anvendelse af stalpulver pa basis af Fe-Cr-Si til korrosionsbestandige belaegninger (Danish Patent DK/EP 0933443 T3)	Schroer, Carsten, Dr. (30%) Spiegel, Michael, Dr. (20%) Grabke, Hans-Jürgen, Prof. (30%) Sauthoff, Gerhard, Dr. (20%)
25.04.2001	Nickel-Aluminium Intermetallic Basis Alloy (European Patent EP 0760869 B1)	Sauthoff, Gerhard, Dr. (50%) Zeumer, Benedikt Alfons, Dr. (50%)
30.11.2000	Verfahren und Vorrichtung zum kontinuierlichen Gießen von Drähten (Process and Facility for Continuous Casting of Wires) (German Patent DE 19757093)	Frommeyer, Georg, Prof. (40%) Frech, W., Dipl.-Ing. (40%) Heyder, Bernhard, Dr.-Ing. (20%)
18.05.2000	Verwendung eines TRIP/TWIP Leichtbaustahls (Use of TRIP/TWIP light weight steels) (German Patent DE 19727759)	Frommeyer, Georg, Prof. (30%) Grässel, Oliver, Dipl.-Phys. (30%) Drewes, Dr., TKS (20%) Engl, Dr., TKS (20%)
10.05.2001	Intermetallitsheskiy splav na osnove nikel-alyuminiya (Russian Federation Patent No. 2148671)	Sauthoff, Gerhard, Dr. (50%) Zeumer, Benedikt Alfons, Dr. (50%)
23.03.2000	Verwendung von α_2/γ-TiAl(Mo,Si) Legierungen (Use of α_2/γ-TiAl(Mo,Si) alloys) (German Patent DE 19748874)	Frommeyer, Georg, Prof. (50%) Wesemann, Jürgen, Dr. (50%)

Institute Colloquia and Scientific Seminars

M. Wenderoth, Univ. Göttingen:
Nichtlineare dynamische Instabilität beim Sprödbruch von GaAs (25.01.2000, Colloquium).

E. Tolksdorf, VGB Essen:
Potentiale und Grenzen der Anwendung der neu entwickelten 9-12% Cr-Stähle für die Kraftwerkstechnik (15.02.2000, Colloquium).

H. Zimmermann, TKS AG Duisburg:
Oberflächenrisse beim Dünnbandgießen von Stahl (04.03.2000, Colloquium).

S. Zaefferer, TU Darmstadt:
Online Semi-Automatische Messung von Einzelorientierungen in hochverformten Metallen im TEM (02.03.2000).

J. Hald, TU Denmark, Lyngby, Denmark:
9-12 %Cr power plant steels – microstructure stability and alloy optimisation (28.03.2000, Colloquium).

B. Zeumer, McKinsey & Company, Düsseldorf:
Wege zu Innovationen in der Stahlindustrie (11.04.2000, Colloquium).

M. Hillert, Royal Institute of Technology - KTH Stockholm, Sweden:
Solute drag, solute trapping and diffusional dissipation of Gibbs energy (12.04.2000, Colloquium).

M. Hillert, Royal Institute of Technology - KTH Stockholm, Sweden:
Martensite (13.04.2000).

M. Hillert, Royal Institute of Technology - KTH Stockholm, Sweden:
Bainite (14.04.2000).

M. Zehetbauer, Univ. Wien, Austria:
Merkmale extremer plastischer Verformung zur Realisierung von Nanostrukturen in Metallen (17.04.2000, Colloquium).

S. Ahzi, Univ. Strasbourg, France:
Texture modeling (02.05.2000, Colloquium).

K. Helming, TU Clausthal:
Texturen in der modernen Festkörperforschung (16.05.2000, Colloquium).

P. Juntunen, Univ. Oulu, Finland:
Optimizing cold rolling reduction and continuous annealing to improve deep drawability of IF steels (29.05.2000).

T. Miyazaki, Univ. Birmingham, UK:
Computer simulation of microstructure formation in real alloy systems based on the Discrete Type Phase Field Method (06.06.2000, Colloquium).

A. Mozalev, Belarusian State University of Informatics and Radioelectronics, Belarus:
Application of porous anodic alumina and valve metal oxides in modern electronics and nanotechnology (10.07.2000).

D. Ponge, Schweißtechn. Lehr- und Versuchsanstalt Nord, Hamburg:
Metallkundliche Konzepte bei Feinkornbaustählen (11.07.2000).

E. Nembach, Univ. Münster:
Experimentelle Untersuchungen der intermetallischen Phase Fe_3Al: Ordnungszustände und Versetzungsprozesse (15.08.2000, Colloquium).

G. Grundmeier, Dortmunder Oberflächen Centrum, TKS AG, Dortmund:
Elektrochemische und oberflächenanalytische Untersuchungen an neuartigen Dünnschichten für die organische Bandbeschichtung von Stahl: SiO_2-Flammenpyrolyse, Plasmapolymerisation, UV-Härtung (24.08.2000).

H. Takahashi, Hokkaido Univ., Sapporo, Japan:
Patterning of the surface of aluminium by laser irradiation and AFM probe processing (08.09.2000).

M. Seo, Hokkaido Univ., Sapporo, Japan:
Surface stress of noble metal electrode and stress of anodic oxide film on metal (08.09.2000).

N. Tsuji, Osaka Univ., Japan:
Ultra-grain refinement of steels and aluminium alloys by a novel intense straining process, ARB – Microstructure formation and mechanical property of ultra-fine grains (11.09.2000).

V. Lanteri, IRSID, Maizieres-lés-Metz Cedex, France:
Scaling phenomena during hot rolling: kinetic and mechanical aspects (12.09.2000, Colloquium).

S. Takaishi, Tokyo, Japan:
Katana - Japanische Schwertkunst (19.09.2000, Colloquium).

L. Kestens, Ghent Univ., Belgium:
Orientation selection during the nucleation and growth of recrystallized grains in cold rolled low carbon steels (17.10.2000, Colloquium).

M. Winning, RWTH Aachen:
Einfluss mechanischer Spannungen auf die Korngrenzenbewegung (24.10.2000).

T. Al-Kassab, Univ. Göttingen:
Die Tomographische Atomsonde (TAP): Nanaoanalytische Spektrometrie in der Materialphysik (31.10.2000, Colloquium).

K.J. Bowman, Purdue University, West Lafayette, IN, USA:
Texture and anisotropy in piezoelectric materials (16.11.2000).

H. Vehoff, Univ. des Saarlandes, Saarbrücken:
Gefüge und Riss – lokale Messmethoden, Modelle, Anwendungen – (28.11.2000, Colloquium).

T. Hirano, National Research Institute for Metals, Tsukuba, Japan:
Fabrication of Ni_3Al thin foil by cold-rolling (06.12.2000).

R. Rapp, Ohio State Univ., Columbus, OH, USA:
Hans Jürgen Grabke (07.12.2000).

E. Mittemeijer, MPI für Metallforschung, Stuttgart:
Festkörperreaktionen in dünnen Schichten (07.12.2000).

R. Schlögl, Fritz-Haber-Institut der MPG, Berlin:
Heterogene Katalyse mit Eisenoxiden (07.12.2000).

R. Mast, Deutsche Nickel AG, Schwerte:
Grenzflächensegregation von Verunreinigungselementen in Eisen und Stählen – Einflüsse auf Materialeigenschaften (07.12.2000).

F. Blekkenhorst, CORUS, Ijmuiden:
Korrosionsbeständigkeit, Ein Fall für zwei Phasen (08.12.2000).

J. Behm, Univ. Ulm:
Struktur und Dynamik der Cu(100)-Korrosion (08.12.2000).

G. Eckstein, Univ. Erlangen:
In-situ STM-Untersuchungen zur selektiven Korrosion von niedrig-indizierten Au_3Cu*(hkl)*- und Cu_3Au*(hkl)*-Legierungseinkristallen (08.12.2000).

H. Möhwald, Max-Planck-Institut für Kolloid- und Grenzflächenforschung, Golm:
Funktionalisierte Oberflächen und Kapseln (08.12.2000).

W. Rühe, IMTEK, Univ. Freiburg:
Bürsten, Bojen, ultradünne Netzwerke – Neue Wege zu maßgeschneiderten Oberflächen von Materialien (08.12.2000).

G. Grundmeier, Dortmunder Oberflächen Centrum, TKS AG, Dortmund:
Neue Wege in der Oberflächenveredelung von Stahlband – Dünnste funktionale Schichten an Grenzflächen (08.12.2000).

M. Schütze, DECHEMA, Frankfurt:
Erhöhung der Oxidationsbeständigkeit von Titanaluminidlegierungen durch den Halogen-Effekt (08.12.2000).

P. Biedenkopf, Forschungszentrum Jülich:
Werkstoffe und Reaktionen an Phasengrenzen in der Energietechnik (08.12.2000).

A. Pyzalla, Hahn-Meitner-Institut, Berlin:
Beugung mit Synchrotron- und Neutronenstrahlen: Neue Möglichkeiten zur Analyse der Mikrostruktur (30.01.2001, Colloquium).

M. Rettenmayr, Univ. Darmstadt:
Prozeßübergreifendes Modellieren der Gefügeentwicklung in Guß- und Knetlegierungen (19.02.2001).

F. Appel, GKSS, Geesthacht:
Elektronenmikroskopische Untersuchungen zu Verformungsmechanismen in Titanaluminid-Legierungen (13.03.2001, Colloquium).

H. Yasuda, Univ. of Missouri-Columbia, MO, USA:
Cathodic dark polymerization and glow polymerisation (26.03.2001).

H.K.D.H. Bhadeshia, Univ. of Cambridge, UK:
Design and manufacture of really strong and tough bainitic steels (24.04.2001, Colloquium).

E. van der Giessen, Univ. of Groningen, The Netherlands:
Discrete dislocation studies of plastic flow in small confined volumes (15.05.2001, Colloquium).

V. Sarma, Institut für Werkstofftechnik, Dresden:
On the development of multiphase (ferrite+bainite+martensite) medium carbon microalloyed steels. (17.05.2001).

A.J. DeArdo, Univ. of Pittsburgh, PA, USA:
Role of stabilization in improving the microstructure and texture of hot band in type 409 ferritic stainless steels (27.06.2001, Colloquium).

W. Bleck, RWTH Aachen:
Herstellung und Charakterisierung von metallischen Schäumen (10.07.2001, Colloquium).

M. Rappaz, Ecole Polytechnique Fédérale de Lausanne, Switzerland:
Modelling of microstructure formation during solidification (14.08.2001, Colloquium).

H. Freund, Fritz-Haber-Institut der MPG, Berlin:
Von Oberflächen im Vakuum zur Katalyse (21.08.2001, Colloquium).

G. Martin, CEA-Saclay, Gif sur Yvette Cedex, France:
Time scales in solid state kinetics (04.09.2001, Colloquium).

S. Seetharaman, Carnegie Mellon Univ., Pittsburgh, PA, USA:
In-situ studies of high temperature materials processes: observing, modeling and understanding (26.09.2001, Colloquium).

J.M. Vitek, Oak Ridge National Laboratory, TN, USA:
Modeling of phase transformation kinetics in welds (27.09.2001).

D. M. Kolb, Univ. Ulm:
Das Rastertunnelmikroskop in der Elektrochemie: Von der Abbildung zur Nanostrukturierung (13.11.2001, Colloquium).

Y. Brechet, Domaine Univ. de Grenoble, Switzerland:
Challenges in materials and process selection: from rational procedure to expert systems (13.12.2001, Colloquium).

B. Bhushan, Ohio State Univ., Columbus, OH, USA:
Micro/nanotribology and materials characterization studies using scanning probe microscopy (12.02.2002, Colloquium).

K. Janssens, ETH Zürich, Switzerland:
Material simulation using cellular automata (25.02.2002).

J.M. Schneider, RWTH Aachen:
Thin film growth in the presence of residual gas (09.04.2002, Colloquium).

M. Acet, Univ. Duisburg:
Magnetismus und Löslichkeit in interstitiellen Eisenlegierungen (14.05.2002, Colloquium).

J.-H. You, Max-Planck-Institute for Plasma Physics:
Fiber reinforced metal matrix composites for fusion reactors - A solid mechanical study (22.05.2002).

F. Mansfeld, University of Southern California, CA, USA:
Microbiologically influenced corrosion inhibition (MICI) (06.06.2002).

T. Furuhara, University of Kyoto, Japan:
Interphase boundary structure in diffusional and diffusionless transformations (07.06.2002).

M. Tanaka, Tohoku University Sendai, Japan:
Defect identification by CBED (17.06.2002).

N. Wanderka, Hahn-Meitner-Institut, Berlin:
APFIM an Ni-Basislegierungen (18.06.2002, Colloquium).

E.P. George, Oak Ridge National Laboratory, TN, USA:
Smaller is (mostly) stronger; recent developments in our understanding of the indentation size effect (02.07.2002, Colloquium).

H. Mughrabi; Univ. Erlangen-Nürnberg:
Festigkeit und Duktilität ultrafeinkörniger Metalle mit besonderem Bezug zum Dauerschwingverhalten (09.07.2002, Colloquium).

W. Mao, University of Science and Technology, Beijing, China:
On-line measurement technology based on two dimensional X ray detector (12.07.2002).

H. Dong, Central Iron and Steel Research Institute, China:
Recent development of ultra fine grained steel (16.07.2002).

V. Radmilovic, Lawrence Berkeley Laboratory, University of California, CA, USA:
TEM and CALPHAD assisted aluminium alloy design (28.08.2002).

C. Olsson, EPFL Lausanne:
In-situ Analyse von Oxidschichten auf Edelstählen mit EQCM (06.09.2002).

F. Barlat, Alcoa Technical Center, PA, USA:
Measurements and modelling of anisotropic plasticity in aluminium alloys (19.09.2002).

B. Nestler, FH Karlsruhe:
Mikrostrukturausbildung in metallischen Legierungen: Modellierung und numerische Simulation (31.10.2002, Colloquium).

N.D. Spencer, ETH Zürich, Switzerland:
Surface functionalization with polyelectrolytes for biosensor and tribological applications (12.11.2002, Colloquium).

P. Fratzl, Montanuniv. Leoben, Austria:
Hierarchische Struktur und mechanische Eigenschaften von biologischen Materialien: Bericht über Untersuchungen an Holz, Knochen, Sehnen usw. (26.11.2002, Colloquium).

P. Marcus, Laboratoire Physico-Chimie des Surfaces CNRS ENSCP, Paris, France:
Growth mechanisms, atomic structure, and reactivity of ultra-thin oxide films on metal surfaces (10.12.2002, Colloquium).

Symposia and Meetings Organized by the Institute

D. Raabe organized and chaired the session “Metals, Alloys, Composites” of the EU Workshop on strategies for future areas of basic materials science, 13-15 June 2000 in Ludwigsburg near Stuttgart, Germany.

The Meeting of the Fachausschuss Computersimulation of the DGM entitled „Modellverknüpfungen und Hybridmodelle in der Werkstoffsimulation“ was organized by F. Roters at the Max-Planck-Institut für Eisenforschung on 20 September 2000. 12 papers were presented to more than 50 participants from 3 countries.

A symposium was organized by M. Stratmann (Department of Interface Chemistry and Surface Engineering) on 7-8 December 2000 at VDEh, which was dedicated to Profs. H.-J. Engell and H.J. Grabke. There were 150 participants from industry and other research organizations. The after-dinner speech “H.J. Grabke” was presented by R.A. Rapp, Ohio State University USA.

G. Sauthoff together with M. Palm organized and chaired the 10th annual meeting of the Intermetallics Committee (Fachausschuss Intermetallische Phasen) of the DGM on 18 January 2001 at the Max-Planck-Institut für Eisenforschung with 35 participants and 7 presentations by researchers from various institutes and industry.

G. Sauthoff co-organized the annual DPG Metal Physics Section meeting on 26-30 March 2001 at Hamburg as member of the programme committee and session chairman.

The Meeting of the Fachausschuss Computersimulation of the DGM entitled „Prozessmodellierung in der Halbzeugindustrie“ was organized by F. Roters at Wieland Werke AG, Vöhringen, on 22 May 2001. 6 papers were presented to about 20 participants.

A seminar on „Moderne Entwicklungen in der Metallurgie“ was organized by the Max-Planck-Institut für Eisenforschung at Schloss Mickeln in Düsseldorf on 30-31 May 2001 with about 30 participants and 5 presentations.

The Meeting of the Fachausschuss Computersimulation of the DGM entitled „Werkstoffmodelle für die Simulation von Umformprozessen, Neue Methoden - Industrielle Anwendungen“ was organized by F. Roters at the Heinrich-Heine-Universität Düsseldorf on 22 November 2001. 12 papers were presented to more than 50 participants from 3 countries.

G. Sauthoff together with M. Palm organized and chaired the 11th annual meeting of the Intermetallics Committee (Fachausschuss Intermetallische Phasen) of the German Materials Society (DGM) on 16 January 2002 at the Max-Planck-Institut für Eisenforschung with 30 participants and 9 presentations by researchers from various institutes and industry.

Within the 53rd Annual Meeting of the International Society of Electrochemistry (jointly organized by GDCh-Fachgruppe Angewandte Elektrochemie) at the Heinrich-Heine-Universität in Düsseldorf on 15-20 September 2002 with 10 Symposia and more than 1200 Participants, the important symposium „Molecular and Microscopic Aspects of Corrosion and Corrosion Protection“ was co-organized and co-chaired by M. Stratmann and A.W. Hassel. With a total number of 7 contributions the Department of Interface Chemistry and Surface Engineering demonstrated its activity in the field of electrochemistry.

The Meeting of the Fachausschuss Computersimulation of the DGM entitled „Simulation von Phasenausscheidung und Ausscheidung“ was organized by F. Roters at Voest Alpine Industrieanlagenbau in Linz, Austria, on 21-22 November 2002. 19 papers were presented to more than 50 participants from 6 countries.

A symposium on hot forming entitled “Warmumformtag” was organized by D. Ponge at the Max-Planck-Institut für Eisenforschung on 5 December 2002 with approx. 100 participants and 6 presentations.

Lectures and Teaching

G. Frommeyer, TU Clausthal: Physikalische Metallkunde und Technologie der Platinmetalle, WS 2000/2001.

G. Frommeyer, TU Clausthal: Physikalische Metallkunde und Technologie der hexagonalen Metalle Ti, Zr und Hf, WS 2001/2002.

A.W. Hassel, Univ. Düsseldorf: Thermodynamische Rechenübungen für Chemiker, SS 2000.

P. Neumann, Univ. Düsseldorf: Versetzungen in Metallen, WS 2000/2001.

P. Neumann, Univ. Düsseldorf: Werkstoffwissenschaftliche Mechanik, WS 2000/2001.

D. Ponge, Schweißtechnische Lehr- und Versuchsanstalt SLV-Nord in Hamburg, Schweißfachingenieurkursus: Legierungen, Phasendiagramme, Eisen-Kohlenstoffdiagramm, 06.03.2002; ZTU-Diagramme, Eisen-Kohlenstoff-Legierungen, Einfluß der Legierungselemente in Stahl, Herstellung und schweißtechnische Verarbeitung von Feinkornbaustählen, 07.03.2002.

D. Raabe, RWTH Aachen: Eine kleine Ethymologie und Geschichte der Metalle, WS 2000/2001, WS 2001/2002.

D. Raabe, RWTH Aachen: Prozeß-und Werkstoffmodellierung, WS 2000/2001, WS 2001/2002.

D. Raabe, RWTH Aachen: Polykristallmechanik – Theorie, Simulation, Experiment, SS 2001.

D. Raabe, RWTH Aachen: Textur und Anisotropie, SS 2001, WS 2001/2002.

D. Raabe, Carnegie Mellon Univ. Pittsburgh, USA: Computer Simulation in Materials Science, WS 2000/2001.

D. Raabe, Carnegie Mellon Univ. Pittsburgh, USA: Introduction to Computational Materials Science, special summer lectures 2001.

D. Raabe, Carnegie Mellon University, Pittsburgh, USA: Advanced Approaches in Polycrystal Plasticity (block course, lecture) SS 2002.

W. Rasp, RWTH Aachen: Neuere Entwicklungen in der Umformtechnik – Strukturierte Bleche für den Leichtbau, SS 2001.

G. Sauthoff, RWTH Aachen: Spezielle Probleme der Metallphysik II/ Theoretische Metallkunde II, SS 2000.

G. Sauthoff, RWTH Aachen: Einführung in die Werkstofftechnik (für Wirtschaftswissenschaftler), Teil 2: Werkstoffkunde der Nicht-Eisen-Metalle, SS 2001, SS 2002.

M. Spiegel, RWTH Aachen: Korrosion keramischer Werkstoffe, WS 1999/2000, SS 2000, WS 2000/2001, SS 2001, SS 2002, WS 2002/2003.

M. Stratmann, Ruhr-Univ. Bochum, Elektrolytische Korrosion, SS 2001.

K.-H. Tacke, TU Berlin: Werkstoffprozeßtechnik: Gießprozesse und Produkteigenschaften, SS 2000.

Publications

2000

Books, Book Chapters, and Editorial Work

Grundmeier, G.; Jüttner, K.-M.; Stratmann, M.: Novel electrochemical techniques in corrosion research. In: Corrosion and Environmental Degradation, Vol. 1, eds. M. Schütze, R.W. Cahn, P. Haasen, E.J. Kramer. Wiley-VCH, Weinheim, Germany 2000, 285-382 (ISBN 3-527-29971-8).

Pawelski, H.; Pawelski, O.: Technische Plastomechanik. Verlag Stahleisen, Düsseldorf 2000, 214 pp. (ISBN 3-514-00659-8).

Sauthoff, G.: Plastic deformation. In: Intermetallic Compounds, Vol. 2: Basic Mechanical Properties and Lattice Defects of Intermetallic Compounds, eds. J.H. Westbrook, R.L. Fleischer. John Wiley & Sons, Chichester, UK 2000, 41-64 (ISBN 0-471-61175-1).

Sauthoff, G.: Plastic deformation - Addendum. In: Intermetallic Compounds, Vol. 2: Basic Mechanical Properties and Lattice Defects of Intermetallic Compounds, eds. J.H. Westbrook, R.L. Fleischer. John Wiley & Sons, Chichester, UK 2000, 64-66 (ISBN 0-471-61175-1).

Publications in Scientific Journals

Ågren, J.; Clavaguera-Mora, M.T.; Golcheski, J.; Inden, G.; Kumar, H.; Sigli, C.: Application of computational thermodynamics to phase transformation, nucleation and coarsening. Calphad **24**, 41-54 (2000).

Dayal, R.K.; Grabke, H.J.: Dependence of the hydrogen permeation in stainless steel on carbon content, heat treatment and cold work. steel res. **71**, 255-260 (2000).

Enders, M.; Spiegel, M.; Albrecht, J.; Putnis, A.: Mineralogical problems in advanced power systems: The contribution of slag and gaseous chemical species to the fly ash, Eur. J. Mineral. **12**, 639-650 (2000).

Fili, T.; Rohwerder, M.; Stratmann, M.: Influence of surface plasma pretreatment on the interface properties of a-SiC:H-covered steel substrates. Adv. Eng. Mater. **2**, 378-380 (2000).

Frommeyer, G.; Drewes, E.J.; Engl, B.: Physical and mechanical properties of iron-aluminium-(Mn, Si) lightweight steels. J. Phys. (Les Ulis, Fr.) **10**, 1245-1253 (2000).

Fürbeth, W.; Stratmann, M.: Scanning Kelvin Probe investigations on the delamination of polymeric coatings from metallic surfaces. Prog. Org. Coatings **39**, 23-29 (2000).

Girgensohn, A.; Büchner, A.R.; Tacke, K.-H.: Twin roll strip casting of low carbon steels. Ironmaking Steelmaking **27**, 317-323 (2000).

Grabke, H.J.: Corrosion by carbonaceous gases, carburization and metal dusting, and methods of prevention. Mater. High Temp. **17** No. 4, 1-5 (2000).

Grabke, H.J.: Nickel-based alloys in carbonaceous gases. Corros. **56**, 801-808 (2000).

Grabke, H.J.: Surface and interface segregation in the oxidation of metals. Surf. Interface Anal. **30**, 112-119 (2000).

Grabke, H.J.; Müller-Lorenz, E.M.; Eltester, B.; Lucas, M.: Formation of chromium rich oxide scales for protection against metal dusting. Mater. High Temp. **17**, 339-346 (2000).

Grabke, H.J.; Riecke, E.: Absorption and diffusion of hydrogen in steels. Materiali in Tehnologije **34** No. 6, 331-342 (2000).

Grässel, O.; Krüger, L.; Frommeyer, G.; Meyer, L.W.: High strength Fe-Mn-(Al, Si) TRIP/TWIP steels development-properties-application. Int. J. Plasticity **16**, 1391-1409 (2000).

Grundmeier, G.; Schmidt, W.; Stratmann, M.: Corrosion protection by organic coatings: electrochemical mechanism and novel methods of investigation. Electrochim. Acta **45**, 2512-2533 (2000).

Hassel, A.W.; Aihara, M.; Seo, M.: Formation and corrosion of InP/In contacts in hydrochloric acid. Electrochim. Acta **45**, 4673-4682 (2000).

Jiménez, J.A.; Carsi, M.; Frommeyer, G.; Ruano, O.A.: Characterization and mechanical properties of a α/γ duplex stainless steel. Key Eng. Mater. **171-174**, 389-394 (2000).

Kaspar, R.; Tosal-Martinez, L.; Richter, J.; Köthe, A.: Eigenschaftspotential von mikrolegierten Stählen für Gesenkschmieden. Stahl Eisen **120** No. 10, 95-102 (2000).

Kowalik, T.; Adler, H.-J.; Plagge, A.; Stratmann, M.: Ultrathin layers of phosphorylated cellulose derivatives on aluminium surfaces. Macromol. Chem. Phys. **201**, 2064-2069 (2000).

Kowalik, T.; Adler, H.J. ; Plagge, A.; Stratmann, M.: Neue Wege der Haftungsvermittlung mit wasserbasierten Celluloselackfilmen für Keramik-, Stahl- und Titanoberflächen. Farbe und Lack **11**, 48-55 (2000).

Miyake, K.; Hanzawa, N.; Takahara, H.; Kobayashi, S.; Raabe, D.: Application of in situ-formed metallic-fiber-reinforced copper matrix composites to cables used for robots. Jpn. J. Appl. Phys. **39**, L119-L121 (2000).

Müller, R.; Heckmann, K.; Habermann, M.; Paul, T.; Stratmann, M.: New adhesion promoters for copper leadframes and epoxy resin. J. Adhesion **72**, 65-83 (2000).

Panin, S.V.; Neumann, P.; Baibulatov, Sh.A.: Investigation on mesoscale deformation of intermetallic $Ni_{63}Al_{37}$ alloy under compression. Phys. Mesomech. **3** No. 1, 75-82 (2000).

Pérez, P.; Jiménez, J.A.; Frommeyer, G.; Adeva, P.: The influence of the alloy microstructure on the oxidation behavior of Ti-46Al-1Cr0.2Si alloy. Oxid. Met **53**, 99-124 (2000).

Piehl, C.; Tökei, Zs.; Grabke, H.J.: Influence of chromium diffusion and different surfaces finishes on the oxidation behaviour of chromium steels. Mater. High Temp. **17**, 243-246 (2000).

Plagge, A.; Stratmann, M.; Kowalik, T.; Adler, H.J.: Selected aspects of surface engineering with cellulose polymers. Adv. Eng. Mater. **2**, 376-378 (2000).

Raabe, D.: Metallurgical reasons and mechanical consequences of incomplete recrystallization. Stahl Eisen **120** No. 6, 73-78 (2000).

Raabe, D.: Scaling Monte Carlo kinetics of the Potts model using rate theory. Acta Mater. **48**, 1617-1628 (2000).

Raabe, D.: Yield surface simulation for partially recrystallized aluminium polycrystals on the basis of spatially discrete data. Comput. Mater. Sci. **19**, 13-26 (2000).

Raabe, D.; Becker, R.: Coupling of a crystal plasticity finite element model with a probabilistic cellular automaton for simulating primary static recrystallization in aluminium. Model. Simul. Mater. Sci. Eng. **8** No. 4, 445-462 (2000).

Raabe, D.; Miyake, K.; Takahara, H.: Processing, microstructure, and properties of ternary high-strength Cu-Cr-Ag in situ composites. Mater. Sci. Eng. **A 291**, 186-197 (2000).

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Tökei, Zs.; Viefhaus, H.; Hennesen, K.; Grabke, H.J.: Role of fast diffusion paths in the initial stages of oxidation of high temperature chromium steels. Solid State Phenomena **72**, 3-8 (2000).

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***Zaefferer**, S.; Glatzel, U.:* Orientation relationship of phases in an oxidation protection coating on a Ni-based superalloy single crystal, 13th Int. Conf. on Textures of Materials (ICOTOM 13). Seoul, Korea, 29.08.2002.

***Zaefferer**, S.; Glatzel, U.:* EBSD investigation of orientation relationships of phases in an oxidation protection coating on a Ni-based superalloy single crystal, Materials Week 2002, DGM. Munich, 30.09.2002.

The Institute in Public

The inauguration of the renovated main building of the institute on 6 February 2002 was used to present the work of the institute in public. There were about 150 invited guests from politics, industry and science with addresses by Mrs. Gabriele Behler, minister for school, science and research of the state of Nordrhein-Westfalen, Prof. Dr. Ekkehard D. Schulz, chairman of the ThyssenKrupp AG, Prof. Dr. Gert Kaiser, principal of the Heinrich-Heine-Universität in Düsseldorf, Prof. Dr. Dieter Ameling chairman of the VDEh, and Prof. Dr. Hubert Markl, president of the MPG and presentations by Profs. Neumann and Stratmann. A press conference resulted in 10 reports in various newspapers and journals.

Dr. Springorum (ex-chairman of the VDEh), Prof. Dr. D. Raabe, Prof. Dr. E.D. Schulz, Prof. Dr. D. Ameling, Mrs. G. Behler, Prof. Dr. H. Markl, and Prof. Dr. P. Neumann (from the left to the right) during the inauguration ceremony for the renovated main building of the institute.

On the occasion of Dr. O. Grässel having received the “Wissenschaftspreis des Wissenschaftszentrums Nordrhein-Westfalen 2000” numerous articles have been published in the local and national press.

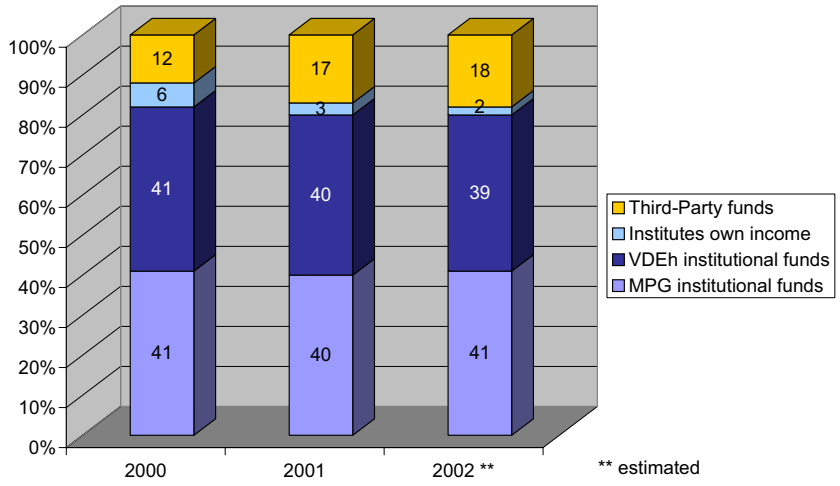
Several articles were published in newspapers and popular-science journals:

- ”Metalle mit dem Computer verformt“, D. Raabe, F. Roters in ”Frankfurter Allgemeine Zeitung“, 07 November 2001,
- ”Metalle gut in Form“, ”Metals in Good Shape“, D. Raabe, F. Roters in “Max Planck Forschung” 3/2001 and “Max Planck Research” 4/2001, respectively,
- ”Karosserie aus dem Computer”, D. Raabe in “Spektrum der Wissenschaft” 2 (2002) 88.

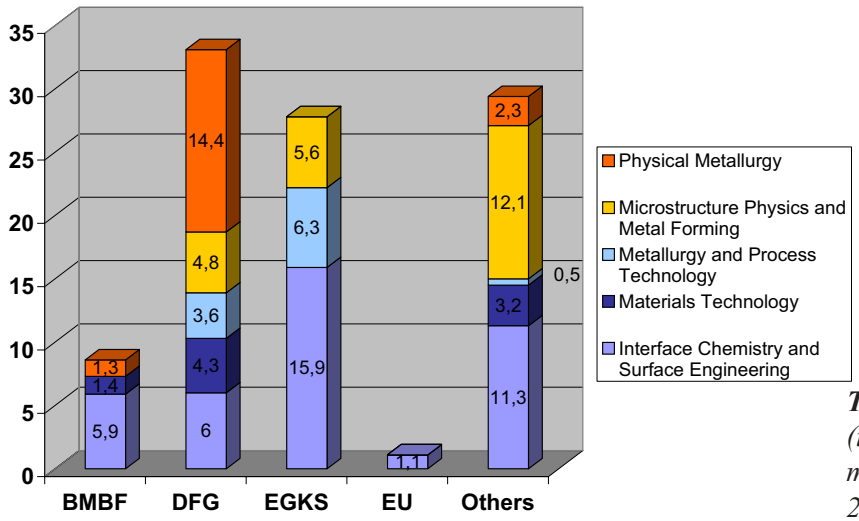
A television report entitled „Steels“ on 04 August 2002 by PRO7 in its program „Welt der Wunder“ focussed on thermomechanical processing of steels (D. Ponge) and scanning electron microscopy (S. Zaefferer). An interview with Prof. Raabe was broadcasted in television on the occasion of the EGKS anniversary in autumn 2002.

The institute was presented to students of the materials science of the RWTH Aachen on 10 July 2002 by Prof. Dr. D. Raabe and Drs. F. Roters and S. Zaefferer. On 01 October 2002, Dr. W. Rasp presented the institute to students of the Technische Universität Bergakademie Freiberg. For a group of students with a VDEh scholarship, a presentation of the institute was given on 26 November 2002 by Prof. Dr. D. Raabe and Drs. G. Grundmeier, M. Rohwerder, J. Gnauk, F. Roters, S. Zaefferer, and D. Ponge.

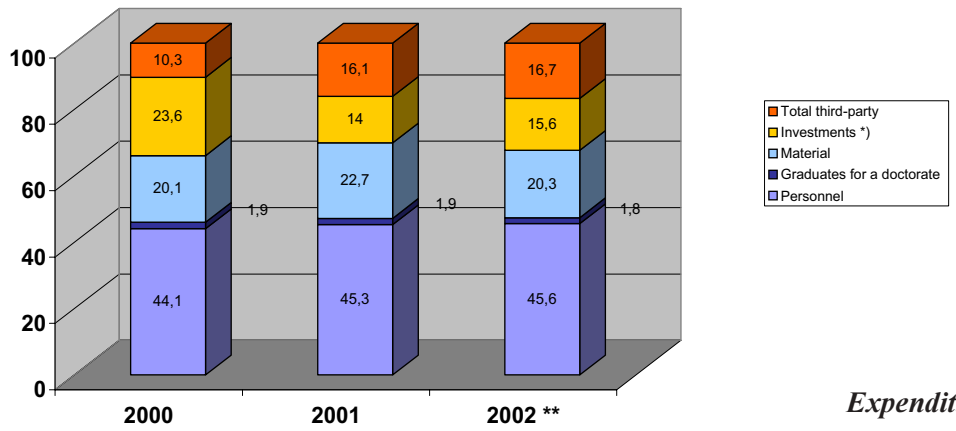
Budget of the Institute



Revenue (in%)
(without appointment-related investment funds and general reconstruction of the buildings)



Third-party funds
(including personnel, material and investments 2000 - 2002 (in %))



Expenditure (in %)

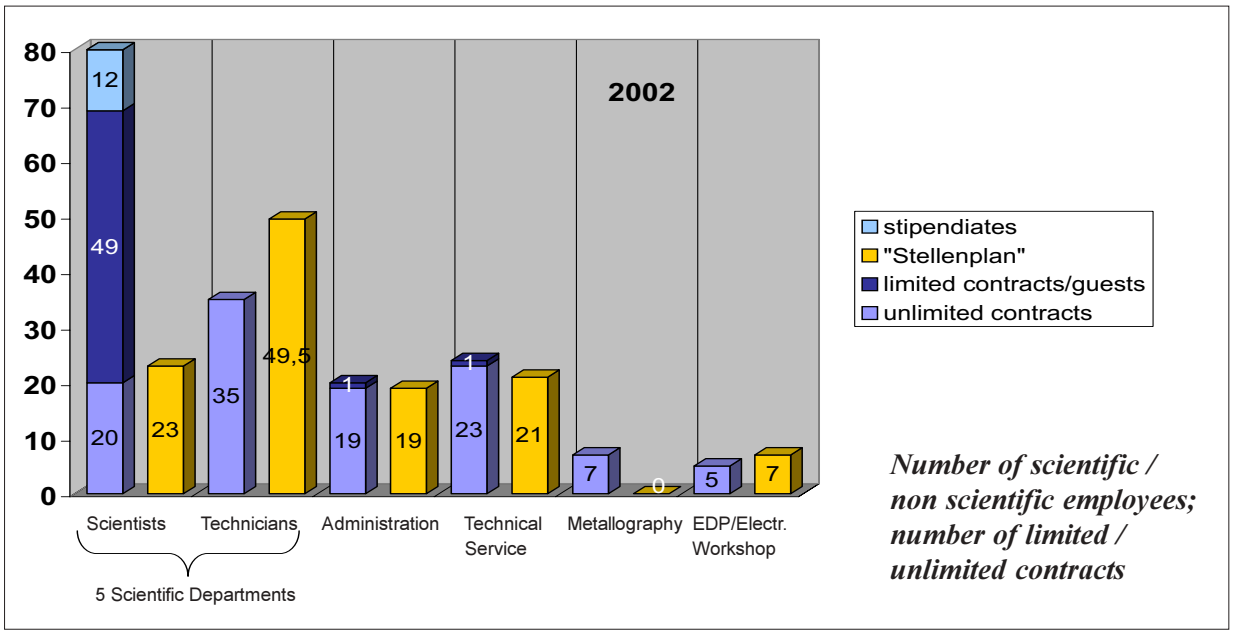
*) including: Large scale apparatus/ EDP (BAR)/ Appointment-related investments/ Separate investments for basic equipment
** estimated

Personnel Structure

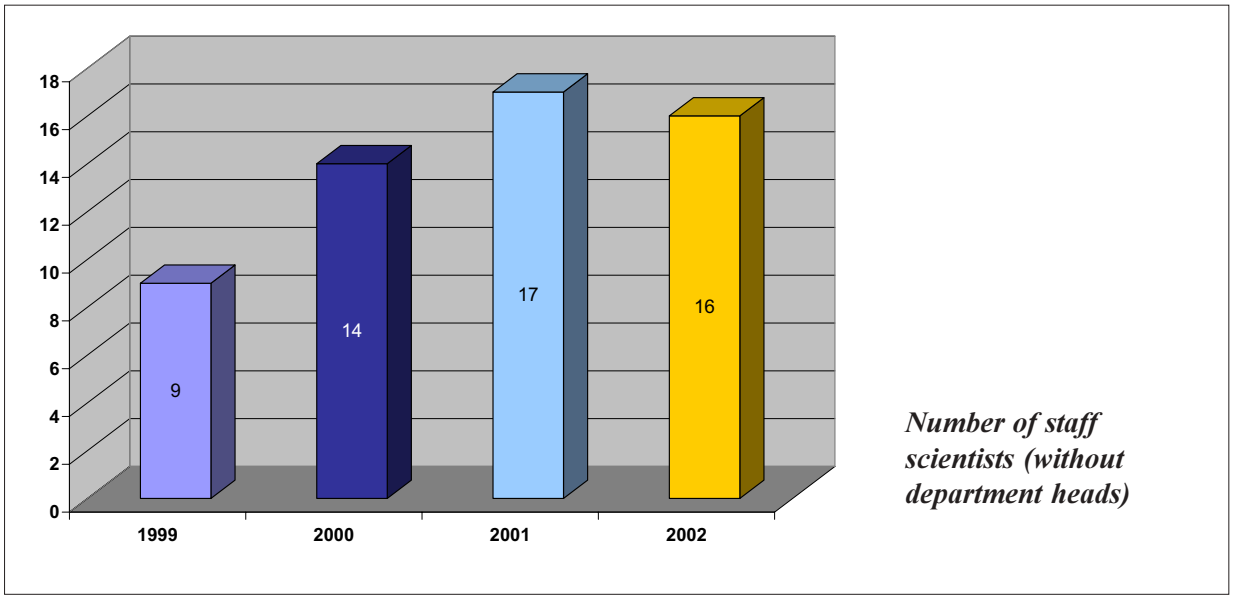
Directors and Division Heads at the Institute

Name	Date of appointment	Status as of 01.01.2003
Frommeyer, Georg	01.01.1983	division head
Grabke, Hans-Jürgen	01.01.1972	retired on 29.05.2000
Neumann, Peter	21.11.1980*	director
Raabe, Dierk	01.07.1999	director
Stratmann, Martin	01.01.2000**	director

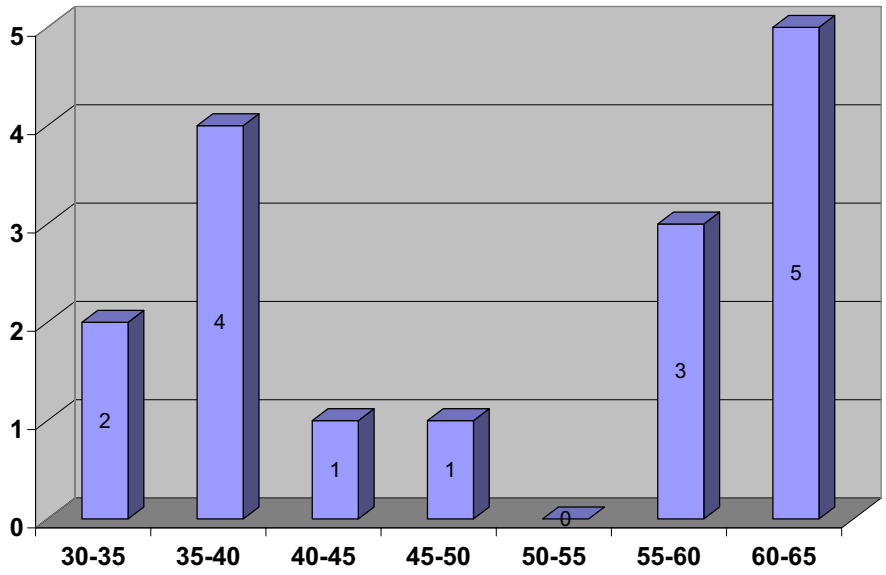
*chief executive from 01.11.1990 to 30.05.2002
**chief executive since 01.06.2002



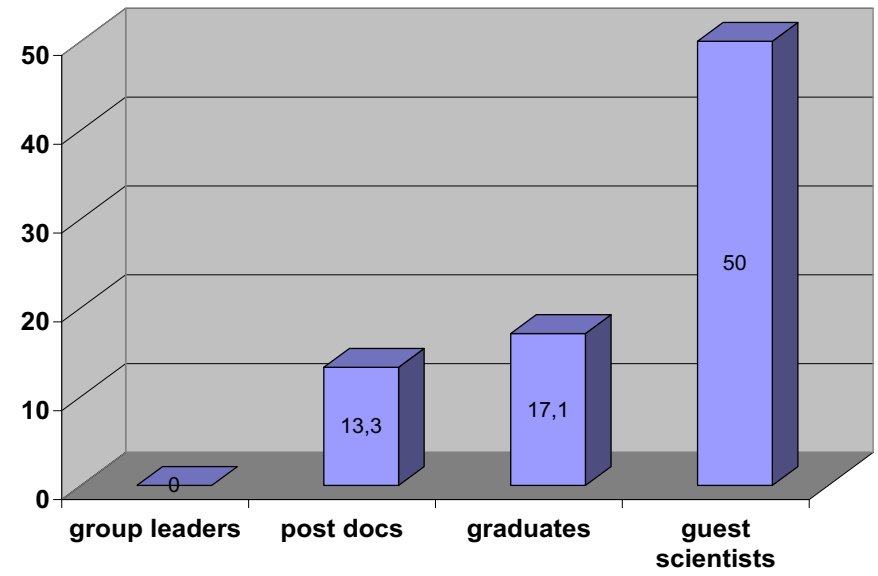
Number of scientific / non scientific employees; number of limited / unlimited contracts



Number of staff scientists (without department heads)

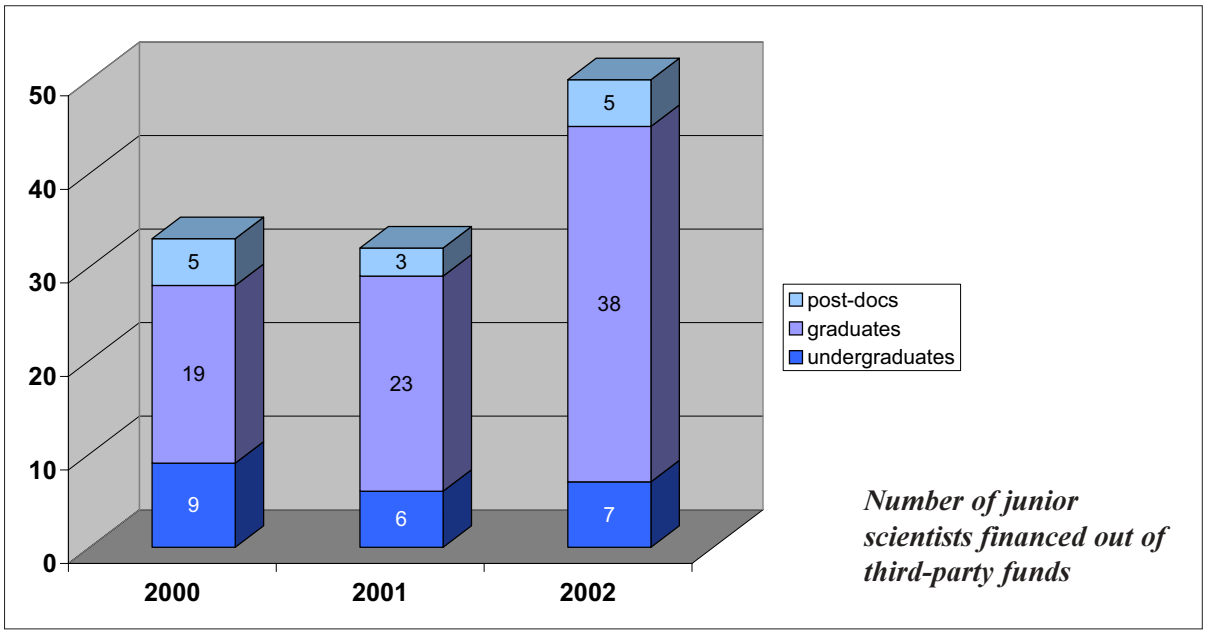


Age distribution of scientists with unlimited contracts (in numbers) (01.01.2003)

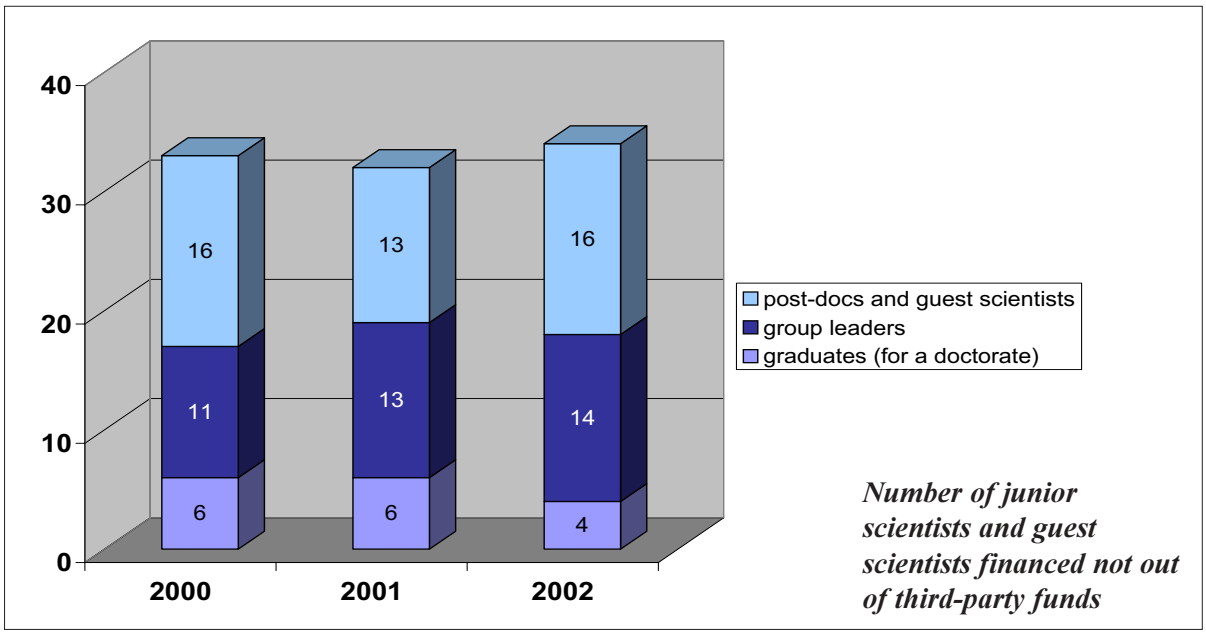


Percentage of female scientists (in %) (31.12.2002)

Junior Scientists



Number of junior scientists financed out of third-party funds



Number of junior scientists and guest scientists financed not out of third-party funds

Doctoral Theses

Dr.rer.nat. ***H. Brehm***, University Jena 2000.

Dr.-Ing. ***S. Drücker***, TU Berlin 2001.

Dr.rer.nat. ***M. Eumann***, RWTH Aachen 2002.

Dr.-Ing. ***J. Gnauk***, TU Berlin 2002.

Dr.-Ing. ***O. Grässel***, TU Clausthal 2000.

Dr.-Ing. ***R. Haardt***, RWTH Aachen 2001.

Dr.rer.nat. ***J. Herrmann***, RWTH Aachen 2000.

Dr.-Ing. ***F. Löffler***, RWTH Aachen 2002.

Dr.rer.nat. ***C. Piehl***, University Dortmund 2001.

Dr.-Ing. ***A. Pramono***, RWTH Aachen 2000.

Dr.rer.nat. ***N. Sämann***, University Dortmund 2001.

Dr.rer.nat. ***T. Schlüter***, University Dortmund 2001.

Dr.rer.nat. ***C. Schroer***, University Dortmund 2000.

Dr.rer.nat. ***I. Steinert***, RWTH Aachen 2000.

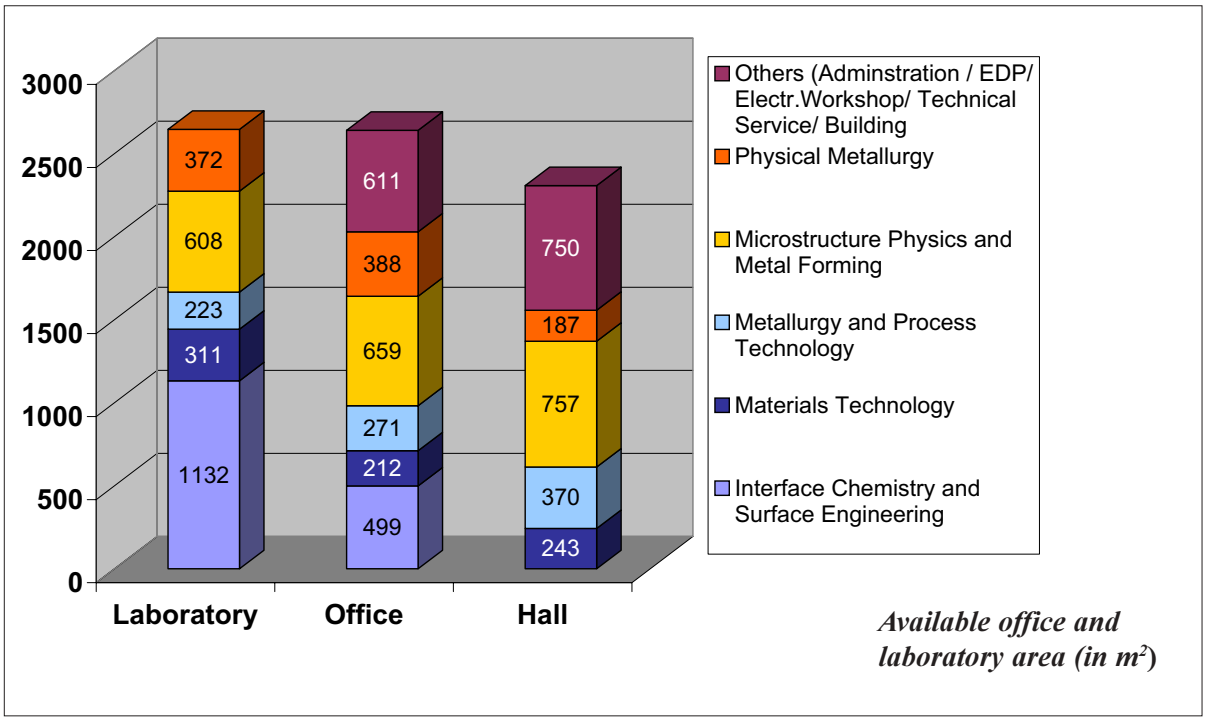
Dr.-Ing. ***H.-G. Vanik***, RWTH Aachen 2002.

Dr.-Ing. ***U. Weidig***, RWTH Aachen 2000.

Dr.-Ing. ***M. Wettlaufer***, RWTH Aachen 2000.

Dr.-Ing. ***C. Wolff***, RWTH Aachen 2001.

Office and Laboratory Area



Available office and laboratory area (in m²)