



Federal Institute for
Materials Research
and Testing

'04

Annual Report



Materials Research and Testing operates under the guiding-line:

Safety and reliability in chemical and materials technologies

Objectives

The Federal Institute (Bundesanstalt für Materialforschung und -prüfung, BAM) has its responsibility in the interacting fields of Materials – Chemistry – Environment – Safety, in particular:

- ▶ statutory functions for technical safety in the public domain, especially relating to dangerous goods and substances
- ▶ collaboration in developing legal regulations like on safety standards and threshold values
- ▶ consulting on safety aspects of materials technology and chemistry for the Federal Government and industry
- ▶ development and supply of reference materials and reference methods, especially for chemical analysis and materials testing
- ▶ assistance in developing standards and technical rules for the evaluation of substances materials, structures and processes with reference to damage prevention, life time prediction, protection of the environment and conservation of economical values.

Activities

BAM is engaged in the interdependent and complementary activities:

- ▶ research and development
- ▶ testing, analysis, approvals
- ▶ consultation and information.

National and international cooperation

The tasks of BAM for technology, science, economy and society require interdisciplinary cooperation. BAM collaborates closely with technological institutions in Germany and abroad, especially with national institutes. It gives advice to Federal Ministries, economy associations, industrial enterprises and consumer organizations. It provides expertise to administrative authorities and law-courts. In the area of measurement, standardization, testing and quality assurance BAM is the competent national authority for testing techniques. BAM is cooperating with numerous technical, legislative and standardization bodies in order to develop technical rules and safety regulations and represents the Federal Republic of Germany both on the national and international level.

Status

BAM is a senior technical and scientific Federal Institute with responsibility to the Federal Ministry of Economics and Labour. It is the successor of the Public Materials Testing Office (Staatliches Materialprüfungsamt) founded in 1871 and of the Chemical-Technical State Institute (Chemisch-Technische Reichsanstalt) set up in 1920. BAM has a staff of about 1600, including over 700 scientists and engineers working at the main grounds of Berlin-Lichterfelde and at the extension Berlin-Adlershof.

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Contents

| | |
|---|----|
| The Federal Institute for Materials Research and Testing (BAM) in 2004 | 4 |
| Analytical Chemistry | 8 |
| Technical and Public Safety | 14 |
| Environmental Compatibility | 28 |
| Materials Technologies | 34 |
| Technical and Scientific Service Functions | 42 |
| Budget, Personnel, Statistics in 2004 | 47 |

The Federal Institute for Materials Research and Testing (BAM) in 2004

1



2



3



1 Berlin's governing mayor, Klaus Wowereit, signs BAM's Golden Book; on the right: President Prof. Dr. Manfred Hennecke.

2 100 years BAM main building

3 Visitors did not have to stay outside at the open day.

Tradition and future – BAM in 2004

An eventful year lies behind the Federal Institute for Materials Research and Testing (BAM) that presented not only outstanding results in the area of technical and scientific development which will be discussed in detail in the following key project areas, but also a year which was dominated by tradition and future. On 18 June 2004 BAM celebrated its 50th anniversary as a Federal Institute under the authority of the Federal Ministry of Economics and Labour (BMWA). Simultaneously, the fact that BAM's predecessor, the Royal Materials Testing Office moved to today's main building 100 years ago so founding the development of the Berlin-Dahlem scientific centre was commemorated. The governing mayor of Berlin, Klaus Wowereit, the then BMWA secretary of state, Dr. Alfred Tacke and the president of the Free University, Professor Dieter Lenzen were welcomed among the numerous guests of the event. They highlighted BAM's position and role for the Federal Republic of Germany, the city of Berlin and the Dahlem scientific centre in their speeches of greeting. In his commemorative address "Buildings of the Royal Materials Testing Office – evidence of the turn of an era" Professor Helmut Engel of the Berlin Monument Protection Endowment drew parallels between the emergence of BAM's current main building and the industrial architecture in the upwardly moving Berlin.

19 June 2004 was fully committed to BAM's open day. About 5000 visitors, among them many children and young people, learnt in more than 50 experiments and demonstrations how diverse, exciting and important materials research and testing is. The spectrum of mechanical investigations reached from non-destructive testing to the electron linear accelerator. The visitors learnt how the hardest materials can be cut by light or water and how soil samples can be analysed. A brick-work was made transparent by means of radar and one could steal the secrets of firework experts.

Another phase of construction of the Horstwalde open-air test facility was completed in September. This versatile test facility continuously increases its importance for BAM for undertaking tasks of technical and public safety. This second phase covers the plant



4 Hands-on shape memory alloys were a special attraction on 19 June 2004, in particular, for young visitors.

for investigating large tanks, e.g. spent fuel transport containers. On 19 August 2004 the drop tower was inaugurated, which enables drop tests of containers weighing up to 200 tons from a height of 9 metres onto an unyielding base. On 21 and 24 September the first tests were successfully performed on original containers within an international conference.

Evaluation

An institute like BAM, apart from the continuous internal evaluation of its work, undergoes regular external evaluation as well.

The evaluation commission headed by Professor Hubertus Christ started its work in June of the year. The committee members

5 Spent fuel transport container after a drop test from a height of 9 metres on an unyielding base



6 The visitors had a chance to get extensive information on the "Strain gauge as an irreplaceable measurement device for a reliable technology" and its history from a lecture of the same name and an accompanying exhibition.

gained a thorough overview of BAM's activities by visiting the individual departments and talking to numerous members of staff. Professor Christ kept BAM's staff fully informed of progress after each meeting of the evaluation commission, answering questions from members of staff so ensuring the greatest possible transparency of the assessment. The evaluation report will be handed over to the Federal Ministry of Economics and Labour on 18 May 2005.

In addition, the German Bundestag commissioned the Federal Government to have all governmental research institutes evaluated. This task has been undertaken by the Science Board that has already begun work with a written survey in which all departmental research establishments and relevant ministries have been included. Apart from this, individual investigations will also be carried out. One of the 13 institutions selected by the Science Board is BAM. The investigation by the Science Board will be focused on the necessity and quality of the research carried out by the institutions. In this respect BAM considers its position advantageous due to its research results and its integration into the international research community.



Year of Technology

After BAM had successfully participated in the "Year of Chemistry" with a series of lectures in 2003, recognized experts addressed a number of safety-relevant topics on technology in 20 evening lectures in 2004 and discussed with the participants the question: "How safe is technology?" Themes such as "Non-destructive testing of components – a contribution to safety in railroad traffic" or "Wind energy – a challenge to security and reliability" encountered wide public interest.

Reorganisation of the divisions

The reorganisation of the divisions was promoted and completed in the reported year. The complete abolishment of the organizational level of the laboratories and sections was suggested early in the organisational consultation by Prognos. In their meeting at the end of 2003, heads of department decided on the divisions reorganisation with the aim of creating a homogeneous working group



structure and simultaneously abolishing laboratories, sections and project groups. The working group "Implementation of the Reorganisation in the Divisions" headed by the Member of the Presidential Board, *Dr. Norbert Pfeil* was established to develop an implementation concept for the introduction of the working group structure. Their task was to prepare content and organizational aspects of the new structure in such a way that the introduction was guaranteed by 1 January 2005.

The objective of the new structure is to achieve a markedly more efficient use of personnel capacities by the abolition of the existing rigid organisational structures since members of staff can be more easily appointed to different tasks. An interdisciplinary approach to specialist topics is also facilitated by this structure.

7



Common appointment with the Humboldt University Berlin

Professor Dr. rer. nat. Ulrich Panne was appointed head of BAM's Department I Analytical Chemistry, Reference Materials on 1 September 2004 in combination with a C4(S) Professorship for Instrumental Analytical Chemistry at Humboldt University. An associated intensive co-operation supports a competence centre for analytical chemistry, one of the largest in Germany, for which Berlin-Adlershof provides excellent conditions.

External Advice

BAM receives external advice for its strategic operations by the advisory board, chaired by Dr. Eckhard Franz, head of BMW's Directorate General VIII responsible for BAM, and two advisory committees for BAM's department I Analytical Chemistry; Reference Materials (chaired by Prof. Dr. rer. nat. Dr. h.c. Heinz-Georg Wagner) and the BAM-Certification Body (chaired by Prof. Dr. Georg-Alexander Hoyer).

BAM's main project areas

7 Prof. Dr. rer. nat. Ulrich Panne

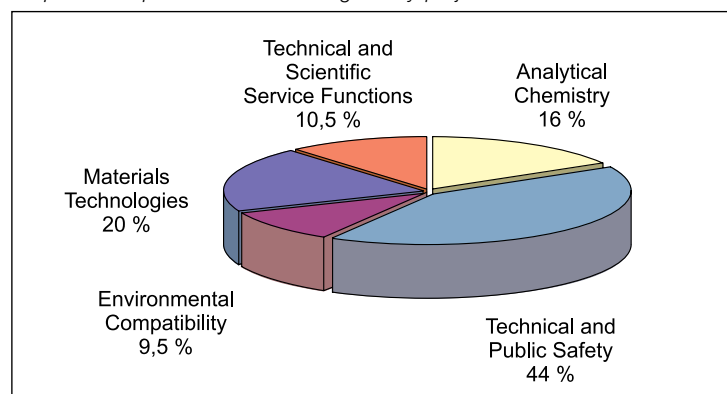
In BAM there are currently about 200 projects under the framework "Safety and reliability in chemical and materials technologies" which are grouped into the following key project areas:

- › Analytical Chemistry
- › Technical and Public Safety
- › Environmental Compatibility
- › Materials Technologies
- › Technical and Scientific Service Functions.

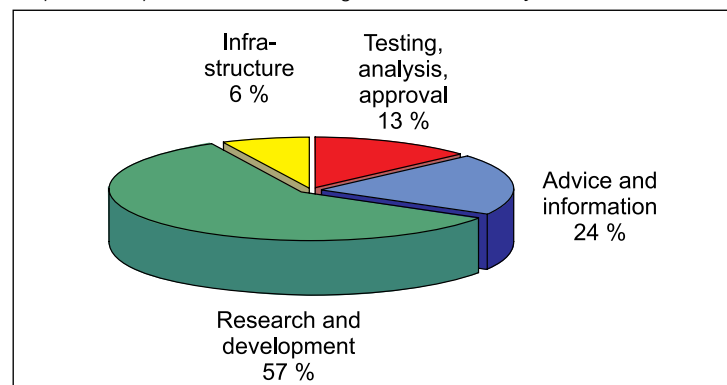
The percentage split of these key project areas as parts of BAM's specialist activity and the breakdown of research and development, testing, analysis and approval, advice, information and infrastructural tasks are shown in the two diagrams.

The main results in the key project areas will be discussed in detail in the following chapters.

Proportion of personnel contributing to key project areas



Proportion of personnel contributing to areas of activity



Analytical Chemistry

1



2



1 National standards for element determination
(Cu, Fe, Si)

2 Gas pumps for dynamic gas mixture production

With all its instrumental and methodical facets, modern analytical chemistry makes a substantial contribution to the safety of our daily products, our environment and our health. Safety of substances, materials and engineering products begins with quality assurance, which is indispensable for the added value of many products and processes. "Good" analysis in terms of quantity and quality determines the quality and value of final and intermediate products of industrial processes. Competence appears all the more important since the economic "site" Germany intends to distinguish itself mainly through the product characteristic "quality" on the global market in the future.

BAM contributes to this by providing a variety of research, consultation and information about analytical reference procedures and reference materials. Special emphasis is placed on the metrological ability to trace back chemical-analytical measurement results to the international system of units (SI), the manufacture, certification and provision of reference materials, the development of reliable methods and procedures, competence assessment of analytical laboratories as well as the completion of complex tests.

Metrology in chemistry

Calibration solutions produced from dissolving pure substances, form the basis of many measurements in chemical analysis. The mass fraction of the main components is rarely known with small uncertainty. Manufacturer's data for some impurities are usually taken equal for the sum of all impurities. To provide metrological traceability, mass fractions of the main components in pure substances are certified at BAM by determining all potential impurities and calculating the difference to the ideal purity of 100%. BAM succeeded for the first time to certify the mass fractions of the main components of 10 materials (Cu, Fe, Sn, Pb, Si, W, Bi, Si, NaCl, KCl) with an uncertainty less than 0.01% and they now form the national standards for element analysis (Figure 1). For this purpose, however, 91 potential elements had to be considered as impurities. Based on this experience, the P-62 CCQM interlaboratory comparison was organised as a pilot study aimed at determining a limited number of metallic analytes in high-purity nickel. The metrological link of commer-

cially marketed calibration solutions with the solutions produced gravimetrically from the primary standards was established by high-precision comparative measurements at Physikalisch-Technische Bundesanstalt (PTB)

In April 2004, with BAM acting as founding member, the Surface Analysis Working Group (SAWG) of the Comité Consultatif pour la Quantité de Matière (CCQM) was established. Previously there had been no interlaboratory comparisons of national metrological institutes of determining the amount of SiO₂ on silicon (expressed as thickness in nanometres). Therefore the P-38 pilot study was performed within CCQM, in which 31 participants from abroad determined the SiO₂ layer thickness on silicon using medium energy ion scattering spectrometry (MEIS), nuclear reaction analysis (NRA), rutherford and elastic backscattering spectrometry (RBS and EBS), X-ray photoelectron spectroscopy (XPS), secondary ion mass spectrometry (SIMS), ellipsometry, grazing-incidence X-ray reflectometry (GIXRR), neutron reflectometry and transmission electron microscopy (TEM). BAM's XPS results showed an excellent agreement with the reference values determined by the British National Physical Laboratory (NPL). A medium squared deviation of only 0.052 nm was obtained for layer thicknesses of 1.5 to 8 nm. The work in connection with P-38 is of great importance for a re-determination of Avogadro's constant, since a layer only few nanometres thick of SiO₂ must be measured precisely on a silicon ball.

Dynamic procedures for gas mixture production were investigated and metrologically qualified in co-operation with an industrial partner. To assess the uncertainty of the dynamic procedure as a primary method, a mathematical model was developed for the transport behaviour of a single pump and the interaction of several pumps of the same type in the mixing process (Figure 2). Thus the absolute throughput of a single pump and the mixture composition can be calculated as a function of operational parameters (geometrical set-up, number of strokes, ambient conditions) and gas properties. The model was verified by a comparison with the absolute transport behaviour (PTB) and with regulations of mixture composition (BAM). Using the model, the uncertainties of all

3 $^1\text{H-NMR}$ spectrum (400 MHz, DMSO-d_6) for purity determination of ginkgolid A, a natural healing substance

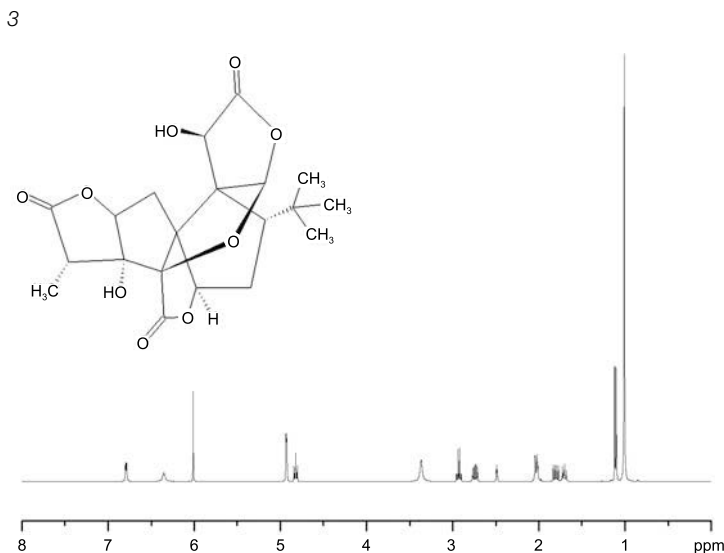
4 Reference material for the determination of PCBs in recycled electrical cables

parameters were transferred to the mixture composition, a complete uncertainty budget was calculated and suggestions were made to improve the manufacture of the gas mixture pumps.

Development of methods and procedures

To determine the purity of pharmaceuticals, herbal reference substances were investigated using high-resolution quantitative nuclear magnetic resonance spectroscopy (qNMR). qNMR as a potential primary method enabled the determination of purity from the main components for the first time. High precision and linearity were achieved for the studied pharmaceutical standards meeting the acceptance criteria established in the pharmaceutical industry and in comparable methods such as high performance liquid chromatography (HPLC), and gas chromatography (GS). *Figure 3* shows the NMR spectrum of ginkgolid A, a phytopharmaceutical formulation which has recently become known in particular for its function to protect cells and promote blood circulation.

Two linked research projects aim to develop and validate a test method to determine polychlorinated biphenyls (PCB) in organic solid wastes as well as to provide relevant reference materials. The background for this is the EU guideline 96/59 EG for the removal of polychlorinated biphenyls and polychlorinated terphenyls (PCT), which was implemented in German regulations by putting the

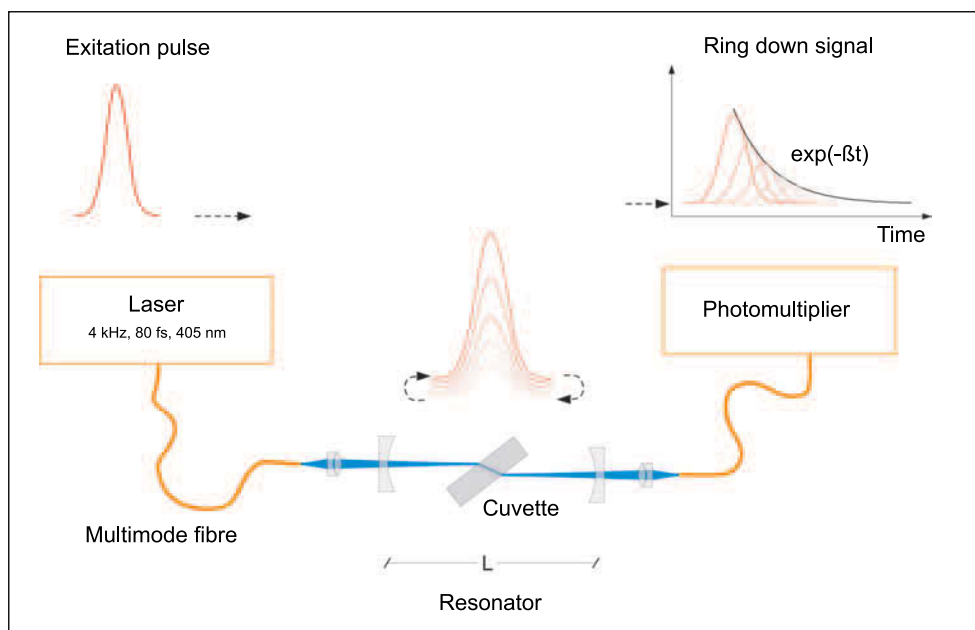


4

PCB/PCT Waste Ordinance into force in June 2000. A limiting value of 50 mg/kg PCB overall content is specified here for the controlled waste disposal. In order to ensure legal clarity, BAM has been commissioned by the Federal Ministry for Environment, Nature Protection and Reactor Safety (BMU) and the Federal Ministry for Economics and Labour (BMWA) in co-operation with the Verein Deutscher Metallhändler (VDM) to validate and confirm a relevant test method by interlaboratory comparison. Work was focussed on plastic granulates coming from the recycling of waste cables and waste wood (*Figure 4*) because of the use of PCB as a plasticiser and flame-retardant impregnating agent. A test method was developed which enables the use of various alternative extraction procedures and extraction agents. After a uniform cleaning by conventional ready made cartridges for solid phase extraction, selected PCB congeners can be analysed using gas-chromatographic procedures with various detectors (gas chromatography and electron capture detection, GC/ECD, or gas chromatography and mass spectrometry, GC/MS). The method was extensively tested for correctness, precision and robustness and finally validated in an interlaboratory comparison with the participation of 23 laboratories on three different plastic granulates. Reproducibility standard deviations between 15 % and 35 % were reached for the PCB 28, 52, 101, 138, 153 and 180 congeners within the content range of 0.1 to 5 mg/kg. In addition, the procedure describes the processing of technical granulates for laboratory analysis by gentle grinding.

Cavity ring down spectroscopy (CRDS) is a new method for high-sensitivity absorption measurements and can serve as a sensitive addition of the classical UV/VIS spectroscopy

5



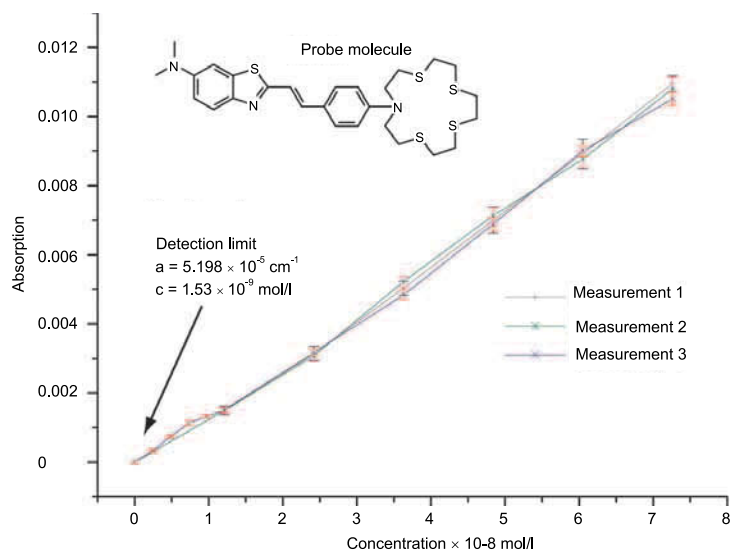
5 Experimental set-up and principle of cavity ring down spectroscopy (CRDS)

6 Absorption of the probe molecule (*N,N*-dimethyl-2-[2-[4-(1,4,7,10-tetrathia-13-azacyclopentadec-13yl)phenyl]-ethenyl]) as a function of concentration with the relevant measurement uncertainties. The detection limit in the absorption of $5.2 \times 10^{-5} \text{ cm}^{-1}$ here corresponds to a concentration of $1.5 \times 10^{-9} \text{ mol L}^{-1}$.

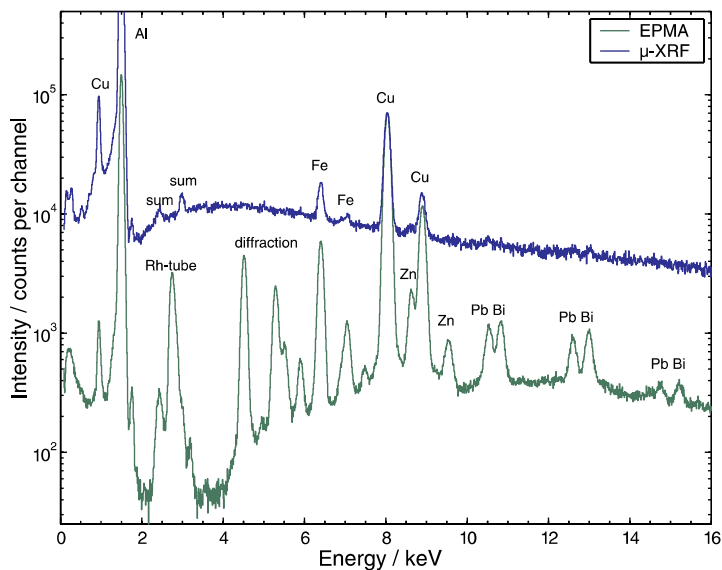
within the range of absorptions between $10^{-5} - 10^{-8} \text{ cm}^{-1}$. The objective of the current work is to extend the method for absorption measurements on solid bodies or in liquid phase. Arranging liquid cuvettes in Brewster angle and keeping the solvent at a constant temperature which is adapted to the refractive index of the cuvette, losses in the resonator can be reduced. Alignment of the resonator has been simplified using regenerative amplified femtosecond laser pulses and optical multimode fibres (Figure 5). An absorption of $5 \times 10^{-5} \text{ cm}^{-1}$ and a concentration of $1.5 \times 10^{-9} \text{ mol L}^{-1}$ have been proved for a probe molecule as an example (Figure 6). Conventional absorption spectrometers yield a detection limit two orders of magnitude poorer.

In the age of nano-technologies, scanning electron microscopes (SEM) are extended by different attachments into efficient analysis stations. A new attachment built as an experimental model, which for the first time enables local-resolution X-ray fluorescence spectroscopy (μ -XRF) using SEM, was investigated in the past three years together with small and medium enterprises (kleine und mittlere Unternehmen, KMU) of the Berlin-Brandenburg Optoelectronic Group (OptecBB). BAM's task was mainly to test the experimental model with the most diverse test tasks within the institute. The μ -XRF attachment was presented to the interested professionals in two collo-

quia in March 2004. Since most SEMs are equipped with an X-ray spectrometer for electron beam microanalysis (EBMA), this spectrometer can also be used for μ -XRF. Thus the detection limit for element contents can be lowered by two orders of magnitude at moderate expense. Figure 7 shows an example of the X-ray spectra of an aluminium wrought alloy using excitation with electrons (EBMA) or X-ray (μ -XRF). The content of lead, bismuth and zinc (about 0.2% by weight in each case) can only be proved in the fluorescence spectrum. Lateral element distributions can also be investigated using a computer-controlled sample table movement. Figure 8



6



7

shows the distribution of iron in a 6-pica letter from a laser printer, the edge length corresponding to 3.5 mm.

For the reduction of the emission of sulphur compounds, particularly SO_2 , into the atmosphere a further reduction of sulphur in fuels is necessary. In Germany, the use of fuel with a sulphur content less than $10 \text{ mg} \times \text{kg}^{-1}$ has already been encouraged by favourable taxes since early 2004, so industry has announced it will sell only such fuels. The oil processing industry mainly uses X-ray fluorescence analysis or high-temperature incineration combined with infrared or fluorescence detection in production control. These procedures however always require standards or reference materials for the calibration of the sulphur content within the lower mg/kg range. A relevant reference procedure was further developed on the basis of isotope dilution analysis with the application of thermal ionization mass spectrometry (Figure 9) and optimised for the range less than 10 mg sulphur per kg fuel. Trueness and reliability of this procedure have been confirmed meanwhile in different comparative studies – including CCQM. Since this procedure was available for reference analyses and other routine-analytical procedures for homogeneity and stability studies, the “Sulphur in petrol” project was initiated together with the European Institute for Reference Materials and Measurements (IRMM) and the British Laboratory of the Government Chemist (LGC). The objective of this project is the production and certification

7 X-ray spectra of an aluminium wrought alloy excited by electrons (EPMA) and/or X-ray (μ -XRF)

8 Distribution of iron in a 6-pica letter from a laser printer. Edge length corresponds to 3.5 mm.

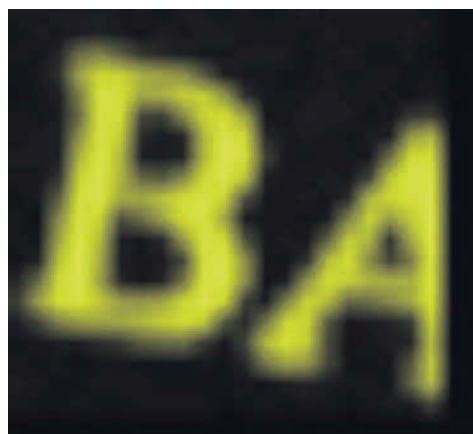
of a relevant set of European Reference Materials (ERM[®]) both for the support of the industry and as a check for limit values.

New certified reference materials

Due to their production technology, siliceous glasses contain water in form of silanol groups. Water content has a substantial influence on mechanical, optical, thermal and electrical properties of glasses and melts. It is checked during the production process using the fast and easy-to-apply infrared (IR) spectroscopy. For the calibration of IR spectroscopy the International Commission on Glass recommends the application of nuclear reaction analysis (NRA) using the $^1\text{H}(^{15}\text{N}, \alpha\gamma)^{12}\text{C}$ reaction. High-energy nitrogen ions, produced by BAM's 2-MV tandem ion accelerator, are shot at the sample in this method. The reaction product, i.e. gamma radiation with an energy of 4.43 MeV, is a direct measure of hydrogen content. Figure 10 illustrates a typical hydrogen depth profile. A hydrogen content of 540 ppm with an uncertainty of ± 40 ppm was determined for the basic glass of the German Glass Technology Society (BAM S-006) to be certified.

Interlaboratory comparisons – proficiency tests of analytical laboratories

Together with the accreditation bodies DAP and DACH and the Hanover High Finance Directorate (Oberfinanzdirektion Hannover, OFD), BAM organized the 10th BAM “Contaminated sites” proficiency test in the area of



8

9



a



b



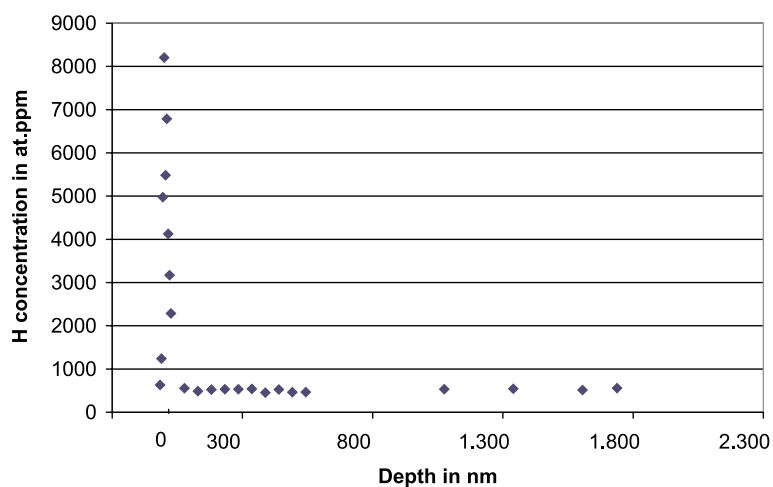
c

9 Procedure for the determination of sulphur in fuels: the sulphur-containing fuel sample is decomposed using a high-pressure asher (a), the sulphate produced is converted into arsenic(III)sulphide (b), separated and determined by a thermal ionization mass spectrometer (c).

chemical soil analysis in August 2004 with 159 participants. Altogether 7 different soil sample batches with up to 256 samples each were produced and characterized with regard to homogeneity of selected pollutants. The participants had to determine the contents of mineral oil hydrocarbons, polycyclic aromatic hydrocarbons (16 PAH according to EPA) and eight environmentally relevant elements. The evaluation of the results was

based on reference values for trueness and tolerance limits specified by BAM. With this proficiency test, BAM participates in an EU project (CoEPT) which deals with the comparison of the evaluation of qualification tests in different European countries. Here another reference material was provided by LGC to all proficiency test organizers. This material was also tested for PAH content in BAM's contaminated sites proficiency test this year.

10 Determination of a hydrogen depth profile in a glass sample using NRA



10

Technical and Public Safety

1



1 Drop test facility
on BAM's test site
in Horstwalde

2



2 Ignition risk investi-
gation

BAM fulfils its statutory responsibility by covering issues within the key project area "Technical and Public Safety". BAM considers its fields of activity to be within five main areas:

- Maintaining a high safety level and, if necessary, its enhancement
- Consultation on safety requirements versus economical and competition aspects
- Provision of technical expertise in social and regulatory decision making processes
- Assumption of direct government responsibility taking account of social, organisational and technical requirements
- Supporting European and worldwide product harmonisation, quality and safety standards.

Plant, incident and transport safety; product and material safety including structural safety and technical issues of traffic safety are considered within the major field of technical and public safety. Certain aspects of consumer, labour and environmental protection are also included. BAM provides information to all those interested by the specialist internet portal "Public Technical Safety - Dangerous Goods/Substances". The portal refers to offers of information by national and international authorities, institutions and associations concerning related issues. BAM has established "information platforms" within this portal about the state of the art.

To investigate and ensure technical and public safety, BAM maintains an outdoor test facility in Horstwalde, about 60 kilometres south of Berlin where in particular, fire, explosion and drop tests can be carried out. The test facility is in the middle of a major reconstruction project which runs from 2003 to 2005. In 2004 a new drop test facility was inaugurated and the drop tower and other equipment on the test facility, such as explosion and fire test stands, are unique within Europe.

Prevention of explosion from mechanically generated spark ignition

The European Directive 94/9/EG (Explosion Prevention Directive also known as ATEX for Atmosphères Explosibles) requires a conformity assessment and marking concerning explosion prevention for non-electric devices,

e.g. pumps, chain hoists, gears, fans. A prerequisite is an ignition risk analysis and assessment. Apart from hot surfaces, mechanically generated sparks, e.g. by friction, are the most frequent ignition sources of non-electric devices. Although well known as an ignition source, the mechanism is not sufficiently clarified and the ignition probabilities are difficult to estimate. BAM has carried out extensive investigations in 2004 into the effect of mechanically generated sparks in various combustible atmospheres with varying impact energy and material combinations. The results are being directly included in the EN 13463 standard series currently in draft, and most specifically in the EN 13463-1 base standard for explosion-proof non-electric devices and in the prEN 14986 draft standard for explosion-proof fans.

Code of Practice for explosion prevention in the production and use of acetylene

In co-operation with BAM, the European Industrial Gases Association (EIGA) has compiled a "Code of Practice – Acetylene" (IGC Doc. 123/04) which can be downloaded from the EIGA website (www.eiga.org). The development of such a Code of Practice was first prompted by the rescinding of the Acetylene Directive in Germany and the Explosives Act in England. In addition, the extensive experience in the gas industry should be recorded. As opposed to European and national regulations, such a Code of Practice has the advantage of not having to distinguish between requirements in properties and operational regulations. It will enable the gas industry to establish uniformly high European safety standards. The "Code of Practice – Acetylene" records specific measures on all parts of acetylene production and filling facilities in addition to general information on the special characteristics of acetylene and the basic measures for used materials, buildings, explosion prevention and maintenance of acetylene plants and information on behaviour in emergency. For the user of acetylene there are specific requirements and information on acetylene supply facilities and pipelines. Where relevant, references to European legislation and relevant standards have been included into the Code of Practice.

The specialist portal "Public Technical Safety – Dangerous Goods/Substances":
www.tes.bam.de/e_index.htm

Safety assessment of the storage and handling of highly-concentrated peroxyacetic acid

The Stendal cellulose factory, one of Germany's largest cellulose factories, started operation in October 2004. The cellulose is bleached by oxygen and specially distilled and stabilized approx. 40-% peroxyacetic acid is used as an oxygen provider. Due to a high production capacity and to ensure continuous operation, an adequate amount of peroxyacetic acid must be kept in stock. Within the licensing procedure, BAM as a recognized test authority, had to assess the safety of the proposed storage of 90 m³ of peroxyacetic acid in an insulated 120 m³ storage tank, based on the German accident prevention regulation for organic peroxides (BGV B4). Essential criteria were capability of pressure relief, thermal stability in connection with the cooling capacity available and behaviour in the case of an external fire. Additionally a safety-relevant evaluation of the organisational and equipment-related preventive measures took place. Both the entire facility and the organisational and equipment-related preventive measures had been conceived in such a way that BAM had no safety-related objection.

Explosion pressure inhibition of windows, doors and façades

BAM has developed a standardised method which can be used to test the resistance of glass windows, glass doors and glass façades against the effect of shock waves in air, such as open-air detonations of explosives. This arose from queries by companies that had to provide this safety-relevant proof for bids for constructing public buildings. BAM has tested over 60 samples in 9 tests since 2003. The tests and their evaluation are based on normal European standards and US-American regulations. The test specimens are arranged at different distances and exposed to specified pressure and impulse loadings by the detonation of 100 kg TNT. BAM's researchers record the incident and reflected shock air pressures at several measuring points, the duration of the positive pressure phase and the arrival time of the shock wave. They evaluate the resistance based on the destruction scenario and the pattern, distance of throw, and fragment size.

Risk assessment of chemical plants

In accordance with the European Seveso II Directive (2003/105/EG) the member states of the European Union must supervise the establishment of new factories, changes in existing plant and new developments adjacent to existing facilities, such as roads, locations with public access, residential areas etc. Methods for the evaluation of land-use planning and existing land development situations are currently discussed in a joint working group of the Major Hazard Commission and the Technical Committee for Plant Safety. Simultaneously, a working group composed of BAM staff members, the Chemical Professional Association (BG Chemie), chemical companies and the Federation of the Chemical Industry (VCI) has developed a concept for risk assessment of industrial sites. This method enables the determination of the risk to be affected by an event (future accident). The quality of the protection concept for the location and the potential impacts of the accident on the surrounding residential area are also considered. The newly developed method, as with other international procedures, is based on empirical quantities and mathematical relationships, but considers the safety philosophy successfully practiced in Germany to establish and operate industrial areas in accordance with the state of the art in safety engineering. Calibration and adjustment of the necessary conventions has been carried out by some practical tests, so that a concept conclusive in itself resulted. This approach has been presented in various committees and is included in the current discussions.

A new runway is about to be added to the Frankfurt/Main airport. The most favourable alternative has the disadvantage that the runway threshold would be approx. 700 m from the boundary of a chemical plant. The plant would directly lie in the approach – a situation unique in Germany and rather exceptional even internationally. In addition, it is important to note that the chemical plant is subject to the extended terms of the Accident Directive. If it were a new chemical plant, suitable actions should be planned that would consider the "air traffic" as an external source of danger. Depending on the situation, extensive protective buildings would be necessary, without which the building and operational

permission might not be granted at all. In Frankfurt the case is just the opposite: the airport extension would convert the plant, approved for a long time, into a sensitive, "risk-ridden" area. Since the problem could not be clarified on a local-political level, the operator of the chemical plant turned to the Federal Ministry for Environment, Nature Conservation and Reactor Safety (BMU) in 2004 and asked that the risk situation be considered by the Major Hazard Commission (SFK). BAM was invited as a member onto a working group of SFK which assessed the risk due to an aircraft crash. Two BAM research reports (No. 231 and No. 260) which investigated the risks of near-airport industrial sites were also included by the working group in the consideration. After extensive consultation the working group of SFK recommended a 'no' to a potential coexistence of runway and chemical plant, which was accepted by SFK.

Safe hydrogen technology – Network of Excellence

Safety issues emerge in connection with a foreseeable increasing use of hydrogen as a source of energy. BAM contributes to the development of new technologies and carries out relevant explosion prevention tests.

BAM is involved in the "HySafe" Network of Excellence initiated by the European Commission in the 6. Framework Program. 24 European companies and institutes and a Canadian university want to exchange their experience in this network, bundle their competence and make them available to other companies and institutions for research or development purposes. BAM's activity is focused on tasks which refer to regulations and standards. BAM initially contributed its extensive experience acquired by its researchers who have participated in the work of German and international specialist committees for many years.

Safe hydrogen technology – high pressure electrolysis

In a research project safety-relevant tests have been performed on a high-pressure electrolyser. This technology developed by

the Jülich Research Centre enables hydrogen production by water electrolysis under high pressures of up to 200 bar. The energetically expensive compression process can be omitted in this procedure. Problems were caused by impurities getting into the product gases by diffusion processes, which led to malfunctions and "near-misses". Investigations in the Jülich Research Centre and BAM have specified the precise operating conditions for the electrolyser. This was based on experimentally determined knowledge of the explosion limits of hydrogen-oxygen mixtures at process conditions.

Safe hydrogen technology – investigation of combustion engines

In co-operation with the Research and Engineering Centre (FIZ) of BMW plc, safety-relevant investigations are currently carried out on combustion engines using hydrogen fuel. A high-performance prototype of a 6-liter twelve-cylinder engine was tested in BAM in a special remote control operated test stand for explosion tests. The safety tests in BAM have the purpose of removing the dangers from hydrogen fuel which result from the high fluidity and rate of diffusion of hydrogen. For this purpose, small leakages are intentionally caused and hydrogen concentrations measured under near actual conditions in the engine components, suction apparatus etc. In a second step the effects of explosions are determined by deliberate ignition tests. The safety concept primarily plans the avoidance of combustible mixtures and only secondarily designs amendments to the engine components, so that they can resist hydrogen-air explosions.

Safe hydrogen technology – propulsion gas tank from composite materials

The technology of the hydrogen car and the necessary storage technologies must be made attractive and safe. Attractiveness is derived from manufacturing costs, the expenditure for refuelling and maintenance and recycling issues. The safety-relevant considerations are focused on the issues of sustainable safety assessment in the context of approvals and the behaviour in accident

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3 Pressure vessels of lightweight construction – with and without fibre reinforcement

4 International group of visitors at the high-pressure test equipment

5 CONSTOR in the drop test facility

situations. Experience from natural gas technology, gathered at BAM's Round Table with respect to CNG technology (Compressed Natural Gas) by experts of the most diverse branches of industry and research, provide, in particular the analysis of accident situations, a basis for the advancement and accident avoidance for hydrogen. One of many research projects dealing with hydrogen in vehicles is the EU project StorHy (Hydrogen Storage Systems for Automotive Application), whose subproject on safety issues, Safety Assessment and Requirement (SAR), is coordinated by BAM. SAR was launched in Berlin in June 2004 at a special symposium, where many interesting papers were presented by partners from the whole European Union, and BAM's new high-pressure test equipment was also presented. It is capable of loading a hydrogen container by hydraulic pressure to 1200 bar several times a minute even at $-60\text{ }^{\circ}\text{C}$.

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New drop test facility for storage and transport containers

BAM's new drop test facility in the open-air test facility at Horstwalde was officially commissioned at the end of September 2004. It was established and approved within a record time of only a few months. The facility consists of a 36-metre-high drop tower, a closed test hall with an 80-ton gantry crane and a 2600-ton unyielding target of reinforced concrete. The test stand enables drop heights of up to 30 metres with a stroke load of a maximum 200 tons making the plant unique world-wide. The hydraulic detachment device was developed by BAM and permits a momentum-free release for nearly every type of tank and container. In the first weeks of operation, two transport casks for radioactive substances were tested in the test stand. First the 181-ton-heavy CONSTOR V/TC of the German manufacturer GNS Company for Nuclear Service Ltd. was tested for its tightness in a horizontal drop from a height of nine metres. Such heavy containers could not be tested by material drop tests anywhere else. According to the manufacturer's plans the CONSTOR V/TC should hold either 32 spent fuel elements from pressurized water reactors or 69 spent fuel elements from boiling water reactors. BAM has also used the measurements to compare real loadings with computer simulations. The collected data help to perfect calculations based on the finite element method (FEM). Only a few days after the CONSTOR drop test the 141-ton-heavy MSF-69BG of the Japanese manufacturer Mitsubishi Heavy Industries (MHI) was diagonally dropped from a height of nine metres. This container will also hold spent fuel rods from nuclear power stations in the future.

PATRAM 2004 International specialist conference in Berlin

Together with the International Atomic Energy Agency (IAEA) and the Institute of Nuclear Materials Management (INMM) BAM organized the PATRAM 2004 specialist conference, the 14th International Symposium of Packaging and Transport of Radioactive Materials. This conference held every three years dedicates itself to issues of transport and storage of radioactive substances. It took place from 20 to 24 September in Berlin – for the second time since BAM had already organized the conference in 1980. This year over 700 scientists and specialists from 25 countries attended PATRAM. Over 300 of them visited BAM's Horstwalde test facility on the occasion of the CONSTOR and MSF-69BG drop tests.

Optical deformation measurement for drop tests of radioactive substances

Accidents are simulated in BAM by drop tests in connection with safety-relevant expert opinions on casks for the transport of radioactive materials. The drop position of the container is chosen in such a way that maximum damage occurs against the safety criteria to be tested. One of the safety criteria to be tested is the tightness of the cask lid system after drop test, which is checked by a number of measurements using various methods. A newly developed measurement method in this field is photogrammetry. The close-range photogrammetry used here is based on the triangulation of digital images of the lid to be assessed. It allows the determination of a potential shift in the lid due to the load caused by the drop test. This horizontal lid shifting can lead to a reduction in

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tightness due to an associated repositioning of the contact areas between metallic seals and flanges. Photogrammetry helps to better understand the results of tightness measurements, in particular the causes of loss of integrity. Our improved understanding of lid shifting gained by photogrammetry provides an important contribution to the computer simulation of drop tests.

Evaluation of non-destructive methods to detect damage on pre-stressed concrete structures

BAM carried out numerous investigations on buildings in the 1990s as damage frequently occurred in old pre-stressed concrete building components with post-tensioning members. Breaks due to material embrittlement in pre-stress steels were identified as the cause of damage in steel which is no longer manu-

6 MSF-69BG cask with optical marks

7 PATRAM participants visiting BAM's test site in Horstwalde

8 Hohenzollerndamm bridge

8



9



9 Fractures in pre-stressed steel

10 Crack initiation area on cracked surfaces

fractured, but is still present in many buildings, especially in bridges constructed prior to the 1960s.

A non-destructive test method (NDT) is of greatest importance for the reliable evaluation of the risk potential of such buildings, which can be used to reliably evaluate the state of the pre-stress steels. Several methods are currently being developed, which have already been tested in some cases in real buildings. Investigations on the longitudinal pre-stress tendons in the Hohenzollerndamm bridge, carried out for the Berlin Senate in the autumn of 1998 in connection with an expert opinion, found incipient cracks in several places. As a consequence, the bridge was pulled down in

10



2001 and 2002 and replaced with a new structure. Two girder segments of the old bridge came into BAM for further investigation. These girder segments were used by BAM's researchers together with those of Berlin Technical University, Braunschweig Technical University and Stuttgart University to analyse three non-destructive test methods for localization of cracks and ruptures. This offered a unique possibility to directly correlate the signals measured using these methods with potential damage on the girder segments over an overall length of approx. 5000 m of pre-stress steel. The girder segments were carefully destroyed in the summer of 2004 and the tendons previously examined were re-tested by BAM. On removal of the tendon sections, cracks and ruptures were found in the pre-stress steels which confirmed that the decision to pull down the bridge had been correct. The investigations and evaluations of the assessment of non-destructive testing methods were completed by the end of 2004 and will be published shortly.

Safety tests of bridge structures – development of a methods catalogue

Stability, road safety and durability of bridge structures in the German motorway and federal highway net is ensured by regular tests carried out by bridge test engineers in accordance with DIN 1076. If damage of unknown cause or extent is discovered by these tests, non-destructive or less invasive tests are performed. In order to guarantee the efficiency of non-destructive and less invasive test methods in the building industry (NDT building methods), the test personnel must be sufficiently well informed and updated to keep up with technical innovations. In co-operation with the Federal Highway Research Institute (BAST), BAM has developed a methods catalogue in the form of a computer program which contains over 1,300 examples of damage on individual components and links them with the technically feasible and economically justifiable NDT building methods. The NDT Building Compendium established by BAM in 1991 and continuously developed ever since is used as a database and currently contains 115 methods. Starting from the damage diagnosed, the building test engineer arrives at the rec-

11

NDT-CE Compendium

Short description
Characterisation
Applications
Assessment

Bundesanstalt für Straßenwesen
BAM Bundesanstalt für Materialforschung und -prüfung

Damage selection: BRÜCKEN, ÜBERBAU, BETON-RISSE-Standsicherheit

Method selection: Ultraschall-Echo für Betonbauteile

11 Application of the NDT Building Compendium

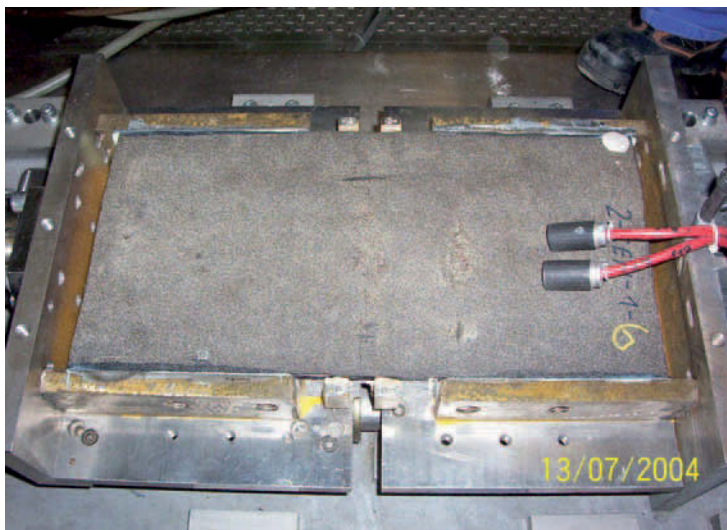
12 Testing a bridge liner

ommended method of the NDT Building Compendium via a link. Here the methods are characterized, the application described and the suitability evaluated. In addition, principle sketches and photos assist the understanding, and a list of regulations, equipment manufacturers and service providers give further information. In this way the analyst is put into a position where he is able to select the most suitable test methods and to supervise their proper execution. The methods catalogue is conceived in such a way that new methods, new regulations or examples of damage can be added without much additional programming work. It is intended to distribute this program to regulators concerned with testing buildings in 2005.

Safety of the traffic infrastructure – sealing of engineering structures, bridges, buildings and roads

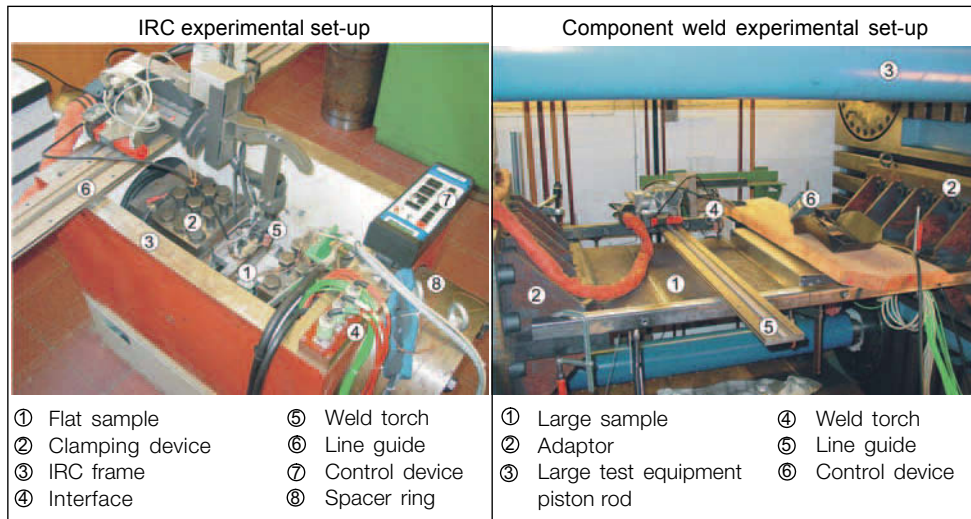
Currently, new harmonized test and requirement standards are being introduced in Europe, which regulate the sealing of bridges, tunnels and other roads against moisture and corrosion. BAM not only manages the ongoing standardisation activity, but has also been commissioned by the Federal Ministry for Traffic, Building and Housing (BMVBW) to carry out accompanying research. The research should create a basis on which new and modified approval tests can be assessed in comparison to the previous national evaluation standard. Based on this, the German

12



technical regulation (ZTV ING) will then be adjusted to the authoritative European law. In 2004 and 2005 the European standard prEN 14224 is the prime focus. This is the test criterion for ensuring the sealing function of liners under dynamic load (crack bridging capability). BAM scrutinises which bridge liner systems proven in Germany

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13 Tests on stress-strain distributions

14 Numerically calculated clamping degrees for orbital welded fittings

15 FE model to calculate the clamping degree on an overlapping butt joint

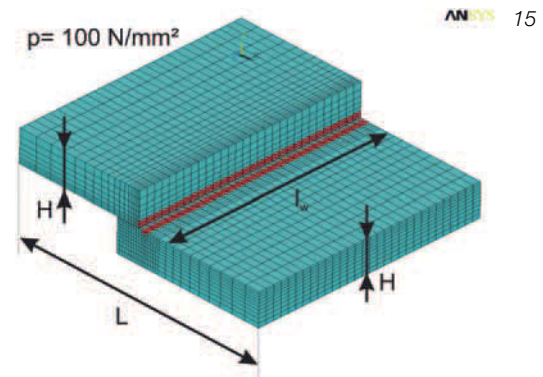
satisfy the new standard and the evaluation of the new results compared to previous experience. Furthermore, actions to be taken by building authorities and the building industry will be specified which will guarantee the high national safety level under the conditions of harmonized European standards. In 2005 further comparative investigations are intended to be performed to transfer test methods into national law.

content. But specifically concerning very stiff and complex welded structures, research studies and damage analyses have in recent years revealed that constructional aspects such as stiffness (restraint intensity) in the weld surrounding as well as joint geometry and joint preparation need to be included in cold cracking assessments.

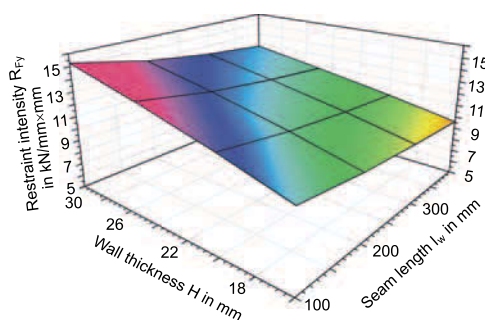
Especially well-directed numerical simulation has been a major point of focus in the year under review, taking precise account of the existing restraint intensity to allow assess-

Safety and reliability with the application of higher-strength base and filler materials for welding large steel structures

For weight reduction and cost saving in welding large steel structures, progressively higher-strength base and filler materials are being used. Component weldability assessment in terms of cold cracking has until now been focused predominantly on material issues. Cold cracks occur during welding as a result of a critical combination of microstructure, mechanical stress and hydrogen



14



ment of crack resistance in joined components. The restraint intensity partially represents the resilience of a structure surrounding a real component weld. Within the scope of a dissertation dealing with three-dimensional finite element calculations, single-run welds have particularly been considered, since an augmented cold cracking hazard must first be assumed for them with regard to real multirun welds and to the anticipated stress-strain distribution in root welds.

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Quantification relating to the dependence of the restraint intensity on various examined geometric parameters, e.g. plate and wall thickness, free clamping length L , joint configuration and also for the first time weld length l_w , was performed. For example, the restraint intensities calculated for plates and pipes showed a significant influence of the component geometry. For pipe cross-sections, in comparison to plates, the restraint intensity depending on the wall and weld thickness was up to 400 % higher, which means that finally there will also be a substantial increase in structural load. It was found for the first time that shrinkage restraint, specifically in thin root runs of pipes, may be very high causing high global and local stress levels after welding and, therefore, a generally increased risk of cold cracking compared to linear welds. Exact knowledge of the orbital stress-strain distribution over the entire pipe circumference is essential, since crack-critical overstress may particularly occur in pipe closure areas. The results already indicate how important it is to clarify the relationship between the weld surrounding and the resulting stress-strain behaviour in welded structures.

Increased safety for excavations

For earth works, in particular within the city, it is necessary to know the location of supply lines such as gas, electricity and water as precisely as possible. This should ensure that the work can be carried out by machines in a time-saving manner without causing damage to the lines which might endanger safety. In the "SmartRAD" EU project successfully completed in 2004, BAM and six other project partners developed a cost-efficient ground penetrating radar system especially for the detection of service lines. BAM's task



17

was the development and provision of a radar antenna for the prototype. A very compact antenna with good spatial resolution was developed using a laminated absorber material as a screen. The prototype has successfully demonstrated the fast detection of lines in several measurement jobs. This antenna technology is to be used and further developed in a follow-up project for the investigation of ballasted railway tracks.

16 Testing the ground antenna

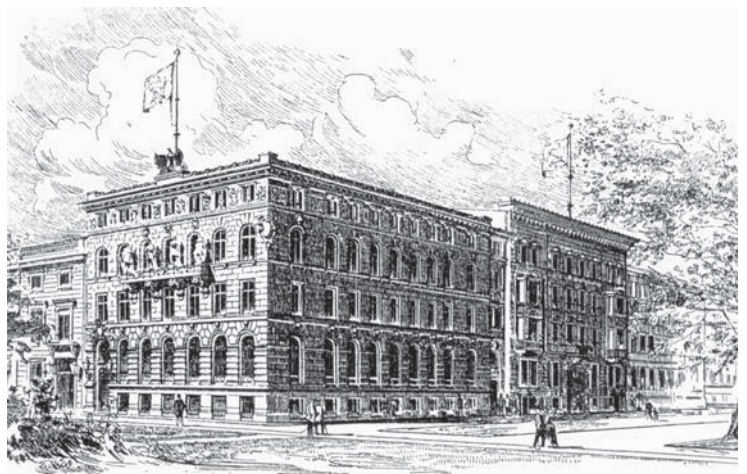
Safety assessment of old buildings

Ageing, changes of use and rising requirements in terms of the standard of existing buildings increase the necessity to maintain

17 The Ritterschaftsbank building

18 The Ritterschaftsbank building, drawing of 1896

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19 Land-based windmill

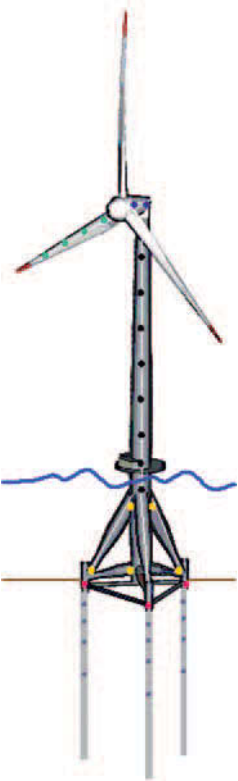
20 Offshore windmill

21 Main burner in BAM's SBI test apparatus

Safety of windmills

Sufficiently safe and economic foundations for windmills under offshore conditions with combined wind and wave force effects have so far yet to be developed according to the current state of the art. Because of the subsidence problem of cyclically horizontally and vertically loaded piles in the bottom of the North Sea with depths of 25 to 30 metres, the danger of insufficient stability of the entire structure must be considered. In addition, it must be guaranteed that the natural frequencies of the entire plant stay within a small range during its entire service life, so that vibration responses do not cause elevated resonance. Otherwise the danger of premature fatigue of the material used under operational loads would occur. Furthermore it must be considered that the dynamic behaviour of wind mills is also affected by changes in the range of the foundations. BAM has therefore been developing concepts and methods for the continuous monitoring and structural assessment of windmills, which include all components from the rotor blades to the piles in the bottom of the sea.

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and improve them. The task is to look after the substance and expand its use for as long a period as possible. From this results the question of safety and reliability of old buildings.

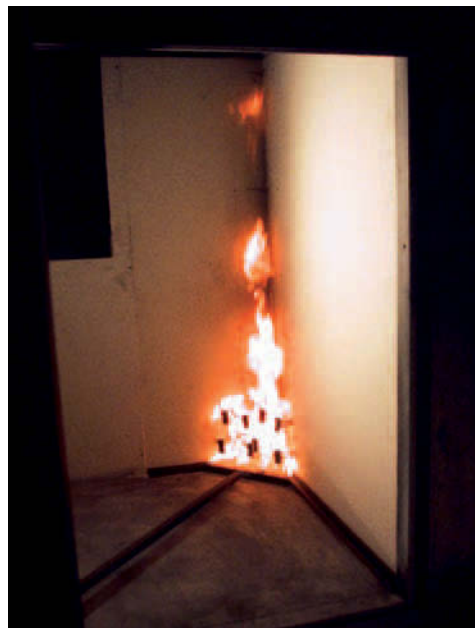
In the building of the previous Ritterschaftsbank (Knighthoodbank) in Berlin-Centre, whose restoration began in 2004, there are over 400 old wrought iron steel girders, which were evaluated by BAM to see they can be reused. Wrought iron girders possess a production-specific banded structure and a sheet-paste-like structure and are therefore strongly anisotropic. Unwanted additional elements such as phosphorus and nitrogen, which could not be sufficiently separated previously, promote embrittlement and ageing of the material. In view of the reuse of the old steel girders, the planned high live loads and very dangerous impact loadings from the reconstruction work were assessed based on random component samples. To protect the building substance, minimally invasive methods have been used in the investigations.

The results showed that profile elements can remain and be reused in the Ritterschaftsbank building.

Fire safety for high and complex buildings – modelling of fire scenarios

The world-wide trend towards ever higher and more complex buildings increases the need for new calculation methods for fire

21



safety. Computer programs are increasingly used which enable the calculation of complex fire scenarios. Fire safety is specially applied to the investigated building and the building can be considered in parts or in its totality. The new fire safety standards which have been standardised in Europe explicitly allow the use of computer programs for devising fire safety concepts. In the Federal Republic this part of the fire safety standards has not yet been implemented in national regulations. Criteria for the reliability of calculations and computer programs are currently being developed, therefore there is an increased need for research in this field. In the field of fire safety engineering, the specialist key area "Modelling of fire scenarios" is now being developed. It is based on the thesis "Modelling of an incipient fire and its application to the single burning item (SBI) test" which was awarded the Adolf Martens graduate student prize at the beginning of this year. Different computer programs are used for the calculation of different fire scenarios. These investigations enable the compilation of a catalogue of criteria which can be used to check the reliability of computer programs and calculations.

Fire safety – nanotechnology is implemented in fire retardancy

Polymer layer silicate nanocomposites show a fire retardancy effect while exhibiting an interesting property combination, acceptable costs and ecological safety. They are therefore the subject of industrial and academic research. The results are however often fragmentary or ambiguous. Several mechanisms occur and the strength of their appearance is different. The flame retardancy effect in real fire scenarios is determined by complex relationships. Therefore, systematic studies were carried out on model systems concerning fire behaviour, morphology, rheology, thermal properties and permeability and consistent descriptions were compiled. Various mechanisms and parameters were differentiated and evaluated concerning different fire tests. Potentials, restrictions and development chances of the concept were elaborated.



Corrosion protection by application of higher alloyed materials for corrosive dangerous goods transport tanks

22 Tanker made of special high-grade steel

Standard materials for tanks for the transport of dangerous goods are not corrosion resistant to many halogen containing or oxidizing and reducing media. In order to solve the various corrosion problems BAM extended an existing research program performed together with Thyssen Krupp VDM and IKS Dresden in 2004. Additionally regarding their suitability as tank materials for transport of dangerous goods, two high alloyed austenitic stainless steels (1.4529 and 1.4562) and one nickel-base alloy (2.4605) were tested to temperatures of up to 55 °C, and the results were evaluated according to the criteria of the BAM-list. The extended research program showed that these materials are a good alternative compared to inliners, because the costs of necessary repairs for inline-systems do not apply. Based on the first positive results the first tank vehicles made of 1.4562 were built, and were delivered to a waste treatment company.

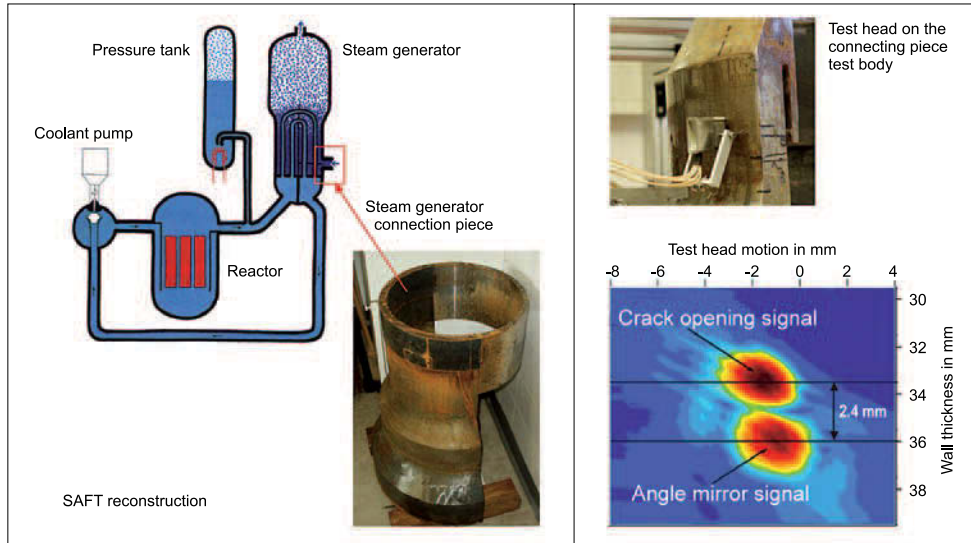
The resistance evaluations of the higher alloyed materials against dangerous goods will be incorporated within the soon to be published 8th issue of the BAM-list.

Defect sizing at feed water nozzles

During the outage of a nuclear power plant in-service ultrasonic inspection was carried out on steam generator feed-water nozzles. The

23 The SAFT analysis method

24 Wall thickness measurement on a test pipe



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evaluation of the measured data demonstrated unacceptable defects. Due to this situation the defective nozzles were cut out for detailed examination. Examination with ultrasound in the lab aimed to determine whether SAFT (Synthetic Aperture Focussing Technique) or TOFD (Time of Flight Diffraction) methods can help in the estimation of the defect size under practical conditions. Phased array probes for the generation of ultrasound have the advantage of a better adaptation of the sound propagation on the geometry of the nozzles. The experimental defect depth estimation is in good correlation to the machined defect size. This guarantees a much better defect size estimation in the future.

Corrosion inspection of pipelines to ensure safety in the chemical industry

The on-stream non-destructive inspection of wall thicknesses in insulated pipe lines is used to reduce the safety risk of on-going corrosion in industrial plants. Major improvements have been achieved by new software for computer based measurement and documentation. This software developed and validated at BAM, will be distributed worldwide by GE Inspection Technologies. The program is used together with new digital imaging-plate systems (Computed Radiography, CR) as film replacement. BAM leads the development of standards for ASTM and CEN (prEN 14784) for the application of CR in non-destructive testing. Together with IAEA and 12 international partners a coordinated research programme was started to investigate the application limits of projection radiography for steel pipes up to 500 mm diameter (see Figure 3).

24



Rail track examination using eddy current techniques

Currently the eddy current technique is used for rail testing under practical conditions in two examination trains. The measured data are essential input for the development of the evolution data software. Although the eddy current system was developed for the detection and evaluation of so called "head checks" (cracks in the rim of the track) other surface defects such as "Squats" and "Belgrospis" (special kind of track damage) can also be

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25 Testing the eddy current technology on a rail test train

26 Sweeper in Mozambique

detected with this system. Therefore the eddy current system was mounted on a grinding train. These trains are used to remove the rail defect using mechanical processes such as grinding. The evaluation of the defect depth after each movement of the grinding train gives the value for the optimisation of the grinding process and therefore guarantees that the damage zones on the track are completely removed so that the accident risk goes to zero.

Reliability and efficacy in mine sweeping

For the first time the design of reliability tests was applied to the investigation of safety and efficacy of the overall sweeping process of

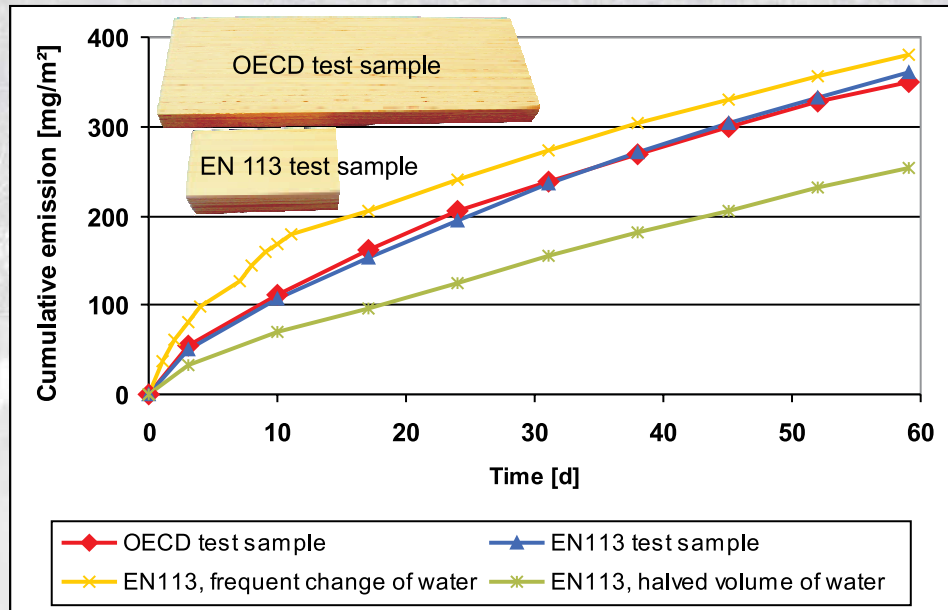
manual mine sweeping. Within a study of the Geneva Centre for Humanitarian Demining (GICHD) a reliability formula extended to a “modular model” was used in Mozambique which enabled the differentiation between physical, environmental and human factors in detail. *Figure 25* shows a sweeper “searching” (poking and digging) for the cause of a metal detector signal. The result can be a “false signal” due to metal fragments (nail, insert, top left) or the sought after mine (insert, bottom left). In particular the increase of efficacy by using magnetic shovels or rakes for the fast removal of these “false signals” was tested.

26



Environmental Compatibility

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1 Emission measurement on treated wood, test samples submerged in water over a long time; OECD test sample: 150 x 50 x 10 mm, saw-rough surface; EN 113 test sample: 50 x 25 x 15 mm, planed surface

2 Elution of wood preservative from wood piles

The term 'environmental compatibility' of materials describes the interface between a manufactured product and the environment in a qualitative manner. Manufactured goods can simultaneously pose a threat to the environment due to emission of toxic ingredients and may themselves be at risk from environmental impact such as surface damage or embrittlement due to weathering, radiation, chemical or biogenic impact.

Harmonization of test methods to determine biocide emissions from preservative-treated wood in water

The authorisation procedure according to the European Biocides Directive includes an environmental risk assessment. For wood preservatives (WP) a CEN working group has suggested laboratory procedures, which can be used to estimate WP emissions in water under different conditions of use. However in order to be able to recommend these procedures for the production of biocide dossiers, experience was missing both for the execution of these methods and the use of the resulting data for risk assessment. A research project for testing the method proposals was supported by the German Federal Environmental Agency (UBA). *Figure 1* shows the results of laboratory tests on treated wood in which the test samples were continuously submerged in water. The water was changed and analysed according to a certain time pattern. Laboratory experiments on propiconazol emission on different test samples (EN 113 and OECD) have provided comparable results while reduced water volume and frequent water changes have lead to different emission rates. This observation is typical for organic substances with limited water solubility. The test samples from pitch pine differ in their dimensions and surface texture.

Limiting emission of volatile organic compounds from building and other products

When used indoors, building products may impair air quality by emitting volatile organic compounds. BAM supports the use of low-emission products by the development and testing of measurement methods and the provision of measurement data within the

implementation of the EU Construction Products Guideline (Essential requirement No. 3: Hygiene, Health, Environment). This ensures that, in addition to identifying health requirements, technical and economic feasibility is also considered. An evaluation scheme developed by the Committee for Health Evaluation of Building Products (AgBB) under BAM's participation has been tested by the example of the product groups of insulation and sealing materials, plasters, varnishes, paints, wood based materials, adhesives and floor coverings. The majority of the tested products met the requirements specified in the AgBB evaluation scheme, whereby there are also particularly low-emission products in each product group. In most cases it will be possible even for the more strongly emitting products to meet the requirements by optimising the manufacture process. Health aspects should be more strongly considered in the European harmonized standardization work in the future (e.g. for insulating materials, floor coverings etc.).

Suitable testing institutes have been recognized nationally and internationally by BAM (EU, USA, Asia) for the use of test methods for particularly low-emission products in connection with the "Blue Angel" Environmental Label, for example CANON Inc., RICOH COMPANY Ltd. (Japan); Air Quality Science Inc. – AQS (USA).

Internet-based data base for fibres and fibre products

The use of natural fibres requires reliable knowledge about usage, life span and environmental properties. The knowledge pool 'Material: natural fibres and natural fibre products' is an internet-based data base developed jointly with the Oberhavel Regional Innovation Alliance (RIO e. V.). It offers information about parameters, material testing and quality assurance, production processes, as well as engineering and design in linked modules from the same source and has been tested online since October 2004.

Since October 2004
online:
www.my-ensemble.net
www.rio-ev.de

3



Environmental influences on and damage mechanisms in historical church windows

The basis for the restoration of historical stained glass windows, using the example of glass windows from the baroque church in Lindow/Brandenburg, is a careful damage analysis. Both the chemical composition of glass and paint layers has been analysed and the crystalline compounds of the surface coatings have been determined using scientific investigation methods such as electron probe microanalysis, micro Raman and IR spectroscopy as well as X-ray diffraction. The development of restoration concepts takes place in close co-operation with monument conservators, restorers and scientists. In this way the gypsum coating identified on the glass surfaces could be removed by ammonium carbonate pastes. Based on the analysis results, a suitable glass paint was developed in the laboratory within the project for repair of defects and made available to the restoration workshop.

Recycling of crushed concrete fines – contribution to reducing construction waste

The Waste Avoidance and Management Act has established the basis for a sustainable waste and resource management. Apart from reducing the amount of waste the legislation aims at achieving the highest quality re-use of residues as possible.

Recycling of crushed concrete fines, which is a particularly problematic material, was investigated in the RECDEMO EU LIFE demonstration project carried out by three industrial partners and coordinated by BAM. The objective of the work was the wet processing of the material to produce an aggregate suitable for concrete and mortar production as well as the full re-use of the residues.

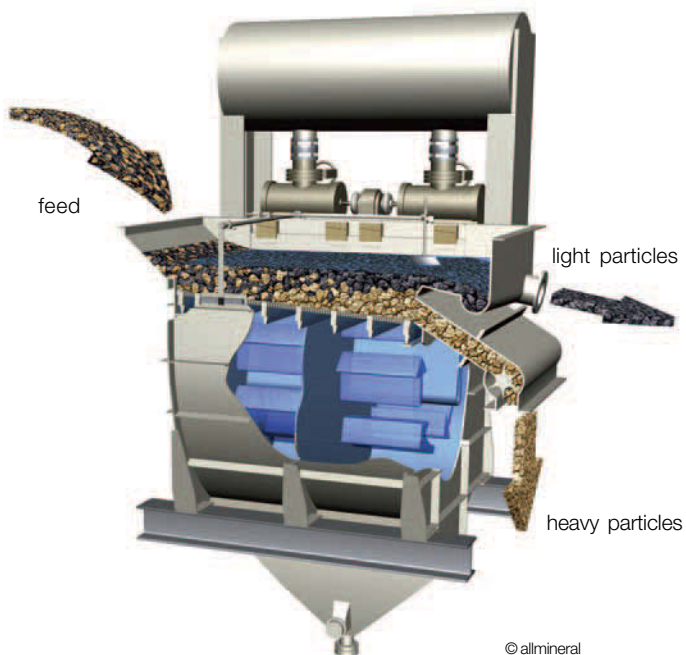
Crushed concrete fines were treated in BAM's pilot plant (throughput up to 1 t/h) for wet processing in several process steps and separated into a natural stone-like material and lighter residues using density separation in a jig. Investigation of building materials manufactured from this substance confirmed that the characteristics of crushed concrete fines can be improved by wet processing.

Tests to utilize residual substances in a composting plant of an industrial partner showed that the procedure can be performed

The project in the internet:
www.recdemo.bam.de

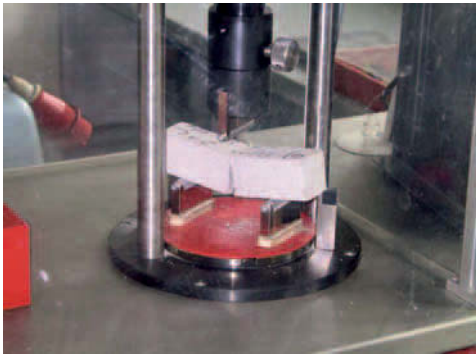
3 Stained glass window, the Lindow church from 1898

4 Rock particles with various densities are pumped through openings in the screen plate by pulsating water and sorted according to their density by their different settling velocity.



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almost free of residues. Processing can thus simultaneously contribute to reducing the amount of waste and saving resources by the replacement of natural substances.

Use of filter sludge containing silica as an additive for concrete and mortar

Filter sludge (FS) has a high specific surface and contains 70 % silica in addition to other inorganic components. Sulphates and chlorides damaging to concrete can be removed by wet processing. This enables the use of filter sludge as a mineral additive in concrete and mortar production and thus saving natural resources and reducing the amount of material to be landfilled. Strength and pore structure characteristics of cementitious mortars with FS-supplement were investigated for their utilisation potential. The results show that up to 10 % of cement can be replaced by filter sludge in a mortar without any loss of strength. However this requires the addition of superplasticizers in order to achieve sufficient workability. An increasing FS content causes a superproportional decrease in the $\text{Ca}(\text{OH})_2$ content in the mortar and a refinement of its pore structure. These are indications that the filter sludge has not only a

function as a filler, but is also involved in cement hydration as a reactive pozzolan.

Plastic drains

Collection and safe drainage of water is a central problem in many geotechnical structures. Currently, plastic drain elements, so-called Geocomposite Drains (GCD), are being increasingly used for this purpose.



7

Where these elements must be efficient over the long term, for example in final landfill caps (Figure 8), special requirements apply to their stability. The determination of the long-term water discharge capacity first requires the measurement of the change in thickness as a

www.bam.de/english/expertise/areas_of_expertise/department_4/division_43/div_43_wg2.htm

function of time under a representative compression-shear stress. The creep curve extrapolated to 10^4 hours provides the final thickness of the drain. In addition, the compression stress that is needed to adjust this thickness in the laboratory is determined. The water discharge capacity determined at this compression stress is then the design basis. It must be guaranteed, however, that aging processes do not cause relevant material changes such as creep behaviour over a

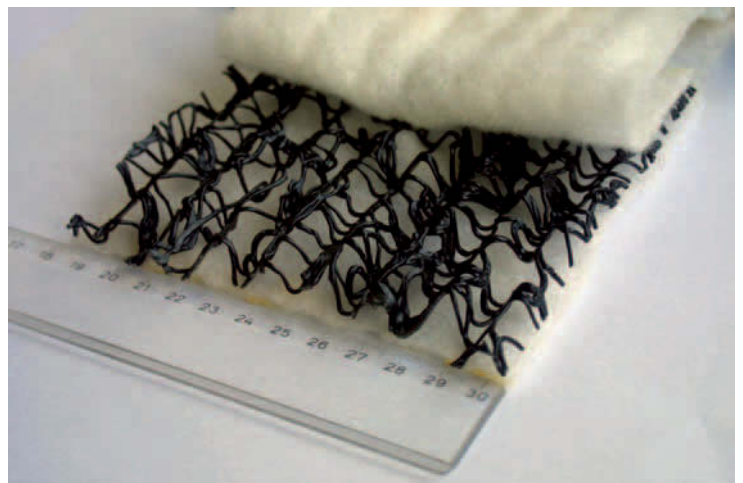
5 Testing building material

6 Composting

7 Silica-containing filter sludge

8 Plastic drain element consisting of a random layer of polypropylene ropes as a drain core and two punched nonwoven materials as a filter and protective layer (Naue Fasertechnik GmbH & Co. KG)

6



8



9

period of at least 100 years. Therefore the plastic drain is exposed to the combined effect of compression-shear stress and a high temperature (80 °C) in creep shear tests. If the samples reach service lives of at least 10³ hours without significant changes, the requirements are fulfilled for their long-term shear strength.

Damage assessment of environmental impacts on historical buildings

www.onsiteformasonry.bam.de

9 Placement of a plastic drain element on the slope of a land-fill cap

10 Moisture investigation after the 2002 flood catastrophe using radar at Veltrusy Castle near Prague, Czech Republic

11 Morphological comparison of *Incisitermes marginipennis* (left) and the M14 termite strain (right). Neither soldier cast (top) nor worker cast (bottom) exhibit any differences.

The ONSITEFORMASONRY project sponsored by the European Union was successfully completed after a 3-year period on 1 November 2004. The objective of the project was to develop procedure combinations and strategies for damage assessment of environmental impacts on historical buildings. The emphasis over the last year was on the execution of common case studies on selected historical buildings. Environmental impacts damaged the Veltrusy castle near Prague in the aftermath of the 2002 flood disaster. Moisture measurements were carried out using radar and microwave bore-hole methods a year after the flood. The results showed that even the inside of the brick-work was already dry at this time and expensive drying measures were not required before the repair.

Chromatographic determination of species for termites

Termites are feared as wood destroying insects in warm and moderate zones. Dam-



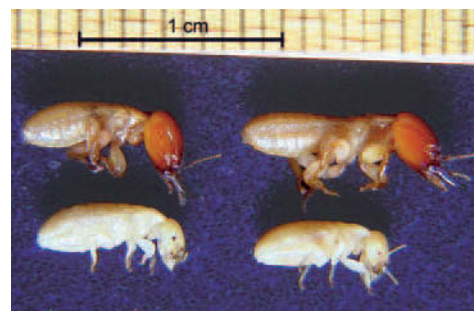
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age caused by them is estimated in the USA alone to be above an annual 10 billion US \$. A constant increase of local occurrences is remarkable even in colder regions. This propagation is particularly helped by rising earth temperatures within urban regions, particularly in the proximity of centrally heated cellars or near long-distance heating pipelines. Traditional curative treatment measures using the “chemical club” have usually only limited success and should no longer be performed in view of residents and the environment.

Understanding the behaviour of termites enables the development of gentle control measures. For this purpose the termite species in question must be known, since a species-specific behaviour within this group of insects requires a deliberate tuning of the methods.

The determination of species based on morphological characteristics often proves difficult, since the identification keys exhibit large gaps (Figure 11).

More reliable statements can be obtained by using data from genetic comparisons. Also simpler methods may be very helpful, such

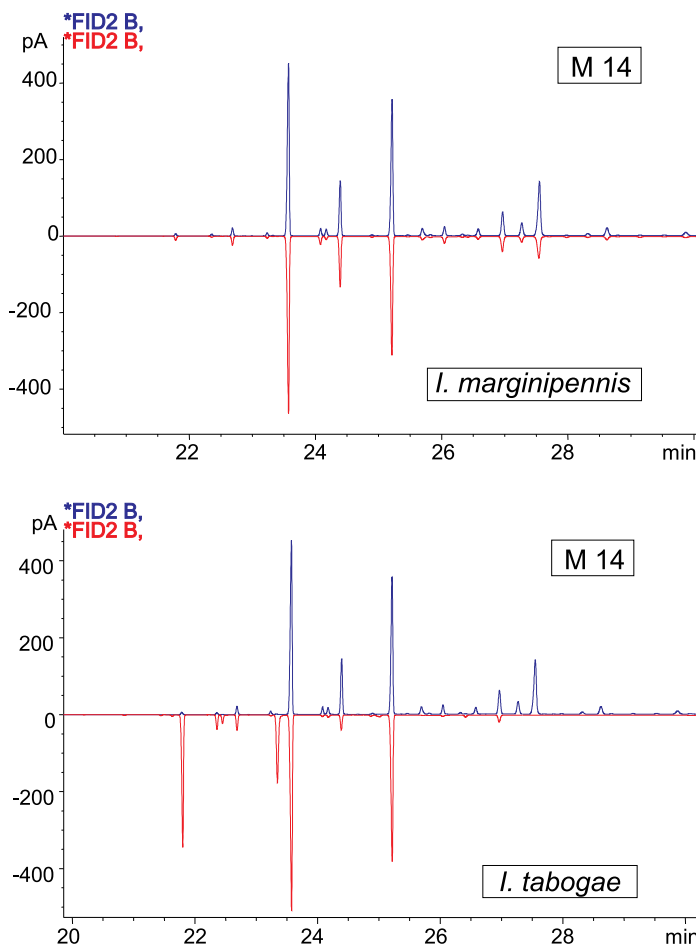


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as the analysis of the very species-specific hydrocarbons on the body surface of insects. A comparison with relevant data of well-known species (in co-operation with the Berlin Free University) can also provide information here.

An unknown termite species from Mexico, marked M 14, could be assigned to the group of dry wood termites due to its way of life and seemed to belong to the *Incisitermes* genus

according to morphological characteristics. GC analyses of cuticular hydrocarbons of *I. marginipennis* and *I. tabogae* were used as comparison (Figure 12). The chromatogram of M 14 and *I. marginipennis* show good agreement. Therefore it is probably the same species. This first finding was confirmed by other biochemical investigations using gel electrophoresis and finally by a genetic analysis.



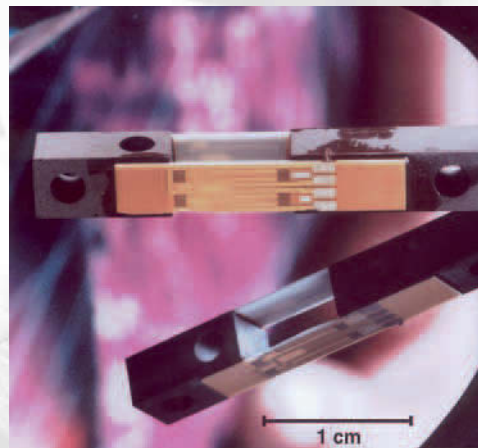
12 Comparison of chromatograms of cuticular hydrocarbons of various termite species. The agreement of the unknown M 14 species with *Incisitermes marginipennis* (top) is nearly perfect, while marked differences with the related species *I. tabogae* (bottom) are clearly visible.

Materials Technologies



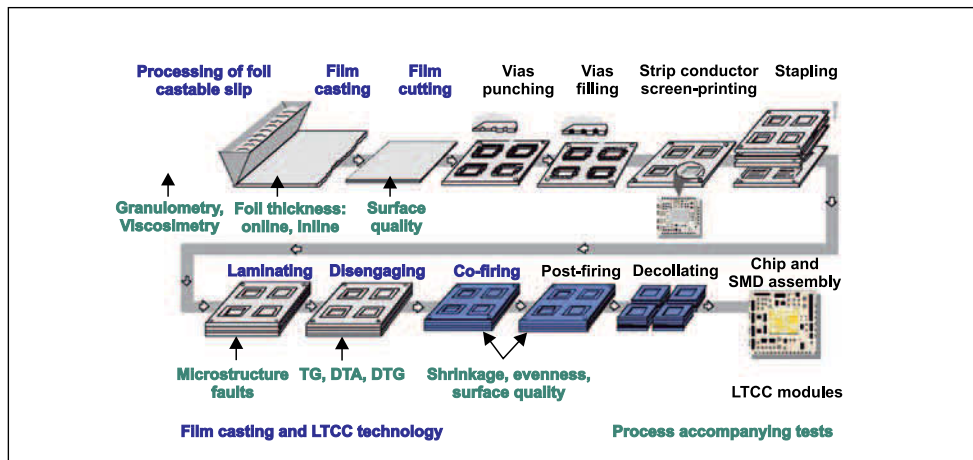
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1 ASK21 glider, made of glass-fibre reinforced plastic (GRP)

2 Bending sensors in LTCC technology



3

3 "Low temperature co-fired ceramic (LTCC) multilayer technology" flow chart

The central task of the key project area 'materials technologies' is the assessment of integrity, reliability and safety of parts and components. The work focuses on areas of technology of great importance for technical and public safety and on those with a key position for the economy of the Federal Republic of Germany.

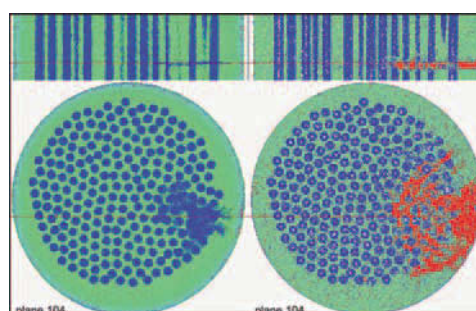
The materials engineering approach assumes that material properties undergo considerable changes during the manufacturing process of a component. Under operating conditions, a component is often exposed to complex environmental conditions giving rise to mechanical, thermal and corrosive loadings which are a decisive factor in its behaviour and lifetime characteristics. Therefore materials engineering is based on materials science, design and manufacture technology and endeavours to characterize and forecast the component behaviour based on this common platform of understanding.

Inspection technique and simulation of safety-relevant components

Fibre reinforced plastic materials (FRP) were used to build gliders for the first time in the 1960s. For older models the certified lifetime of 12.000 flight hours has already been reached. In many cases a decision has to be taken about the extension of the period of operation for which adequate test and simulation methods are required. BAM has developed inspection procedures and compiled results about the behaviour of fibre composites under real loading, which enable the determination of the safety condition of the

material. For this purpose, fatigue tests have been carried out on samples and a representative component. Damage evaluation forms the basis of the investigation of material conditions of the airplanes concerned. The activities are focused on gliders because their light structure is designed and certified considering material loadings which exceed those of commercial airliners or wind mill blades by 50%.

Blades of aeronautical and industrial gas turbines are exposed to very high temperatures of the combustion gases and complex mechanical loading by centrifugal forces, gas pressure and changes in temperature during operation. Technical single crystals of nickel-based superalloys are used for the most strongly stressed blade stages. Compared with conventionally manufactured (polycrystalline) or directionally solidified blades, single crystal blades achieve a much better thermo-mechanical performance due to the lack of grain boundaries and they can therefore be used at higher operational temperatures. This leads to a more efficient energy conversion and increased turbine efficiency. In co-operation with turbine manufacturers, material damage due to thermomechanical load and manufacture-induced microstructural inhomogeneity



4

5 Microstructure of a concrete sample:

- a) reflected-light microscopy,
- b) element distribution sulphur (green) calcium (red) silicon (blue)

6 Welding head for hybrid welding

geneities, such as pores, has been investigated in order to obtain parameters for an improved service life assessment of these safety-relevant, highly stressed components.

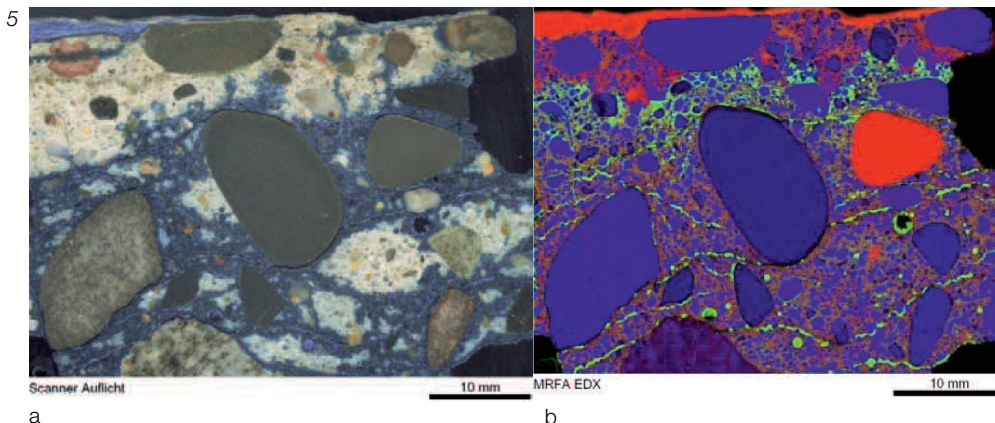
Lighter and more efficient materials at higher operational temperatures are needed for the further development of aeronautical gas turbines. Promising candidates are metal matrix composite materials (MMC) which combine the proven characteristics of titanium alloys with high strength properties of SiC fibres. A lifetime analysis of components from these materials however requires the understanding of the damage mechanism, which is essentially determined by processes at the metal-fibre interface. Spatial resolution and contrast of the interface-sensitive synchrotron refraction computer tomography at BESSY BAMline were considerably increased by the use of asymmetrical Bragg crystals. The damage evolution was characterized in thermally and mechanically loaded samples of a titanium matrix composite material reinforced with long SiC fibres.

Micro X-ray fluorescence analysis (μ XRF) offers new possibilities for the chemical analysis on large material surfaces, which has so far not yet been possible in this form. A fast chemical profile can be established with the help of large-area element distribution images, in particular, for damage diagnosis of heterogeneous samples, such as concrete. The method supplements scanning electron microscopy in an ideal way, if chemical investigations of large sample surfaces have to be performed.

Innovative joining processes, such as laser arc hybrid welding, have been developed for



advanced materials. In order to characterize these weld joints the influence of process-specific solidification morphologies on the mechanical-technological and corrosion properties must be investigated. For example, welding of stainless steel (Figure 6) must ensure hot crack resistance in view of potential metastable solidification modes, in particular when their composition is near the eutectic trough. Hybrid weldings have been carried out on the basic materials 1.4301, 1.4306, 1.4828 and 1.4404 using identical welding-rod materials and welding speeds up to 3 m/min. The batches tested so far have not exhibited any impairment in hot crack resistance for the pure arc method. For the new procedure mode 'laser plus plasma plus powder' a new plasma powder burner was developed and tested. The burner is suitable for hybrid welding using Nd:YAG laser and works with plasma currents up to 300 A at both electrode polarities and powder transport types up to approximately 100 g/min. The procedure excels in high process stability and good seam forming. For example, hybrid welds of 6 mm sheet metals made of 1.4565 material and using the identical powder have succeeded in minimising the formation of





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re-melt cracks at the I joints within the heat influence zone by proper seam forming in such a way that tensile strength of such a weld joint is not impaired as compared to the basic material.

Sensor technology, ageing mechanisms

Moisture measurement is of fundamental importance in many vital areas, for instance moisture determination of inhaled gases in compressed gas bottles of life saving systems, emergency oxygen supply in airplanes or devices for artificial respiration during operations and of unconscious patients. The development of innovative miniaturised laminated structures for coulometric trace moisture measurement with fast response times forms the basis of the development of small, reliable moisture measuring systems.

Fibre-optic sensors constantly gain importance for the monitoring of structures. Optical conductors are responsible for the safe and reliable data transfer and are a substantial component of the monitoring system. They must therefore be included in the safety concept. In this context the aging behaviour of new polymer optical fibres (POF) as well as the effect of ageing mechanisms on the optical data transfer was investigated. To prove an accelerated degradation of optical cladding as compared to the optical core of a POF and to standard PMMA, new test methods were developed using chemoluminescence measuring procedures and the ageing mechanisms were simulated by a new modelling software. These investigations



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were carried out in close co-operation with car manufacturers, suppliers and Potsdam University and their results have been included in current regulations both on national and international level (VDI/VDE, DKE, IEC).

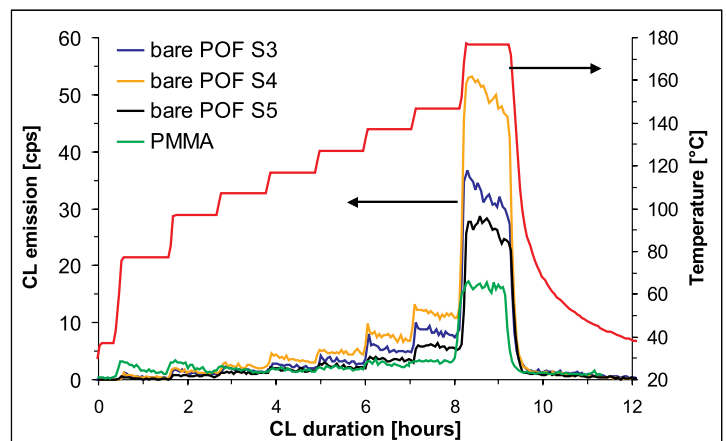
Thermal stability of components depends in many cases on the local concentration and high-temperature transport properties of gases solved in the material. By further developing a high-temperature vacuum hot extraction device with mass spectrometer gas detection the proof sensitivity of dissolved gases was markedly increased. It is possible to determine hydrogen content at ppm in soda-lime-silica and silica glasses and to reliably prove hydrogen impermeability of silica-, cover-, and seal-glasses – an important aspect in ageing of e.g. halogen and discharge lamps. Hydrogen emission behaviour of metallic materials can also be characterized. Thus reliability in the investigation of hydrogen embrittlement of construction materials increases.

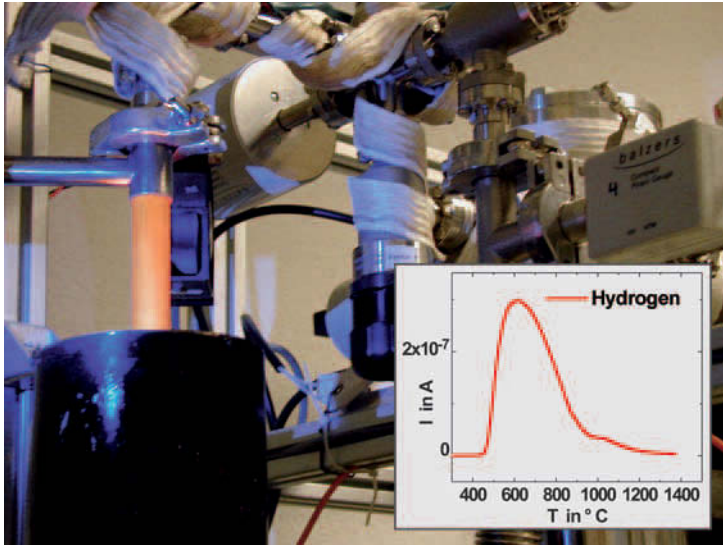
7 Planar coulometric trace moisture sensor and facility for sensor testing under increased pressure

8 Test facility for the investigation of the ageing behaviour of POF

9 Proof of accelerated degradation of the optical cladding as compared to the optical core of a POF and to standard PMMA using chemoluminescence measuring methods

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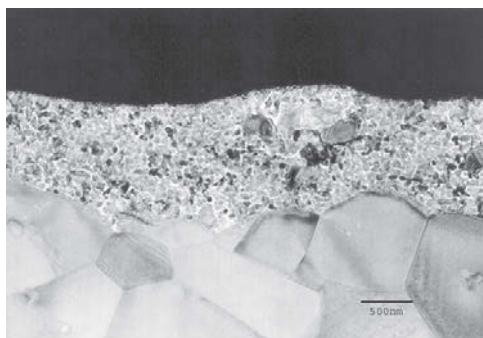
Development and characterisation of interfaces and surfaces

Diamond-like carbon coatings (e.g. a-C:H) have already been used with big success in various applications. The positive effects of these coatings are based on their role as wear and friction reducers with coefficient of friction values well under 0.1 for unlubricated sliding of steel. The fields of application reach from hard disks for computers to the injection system and valve-gear of combustion engines. However, they are not universally applicable "miracle coatings" as could be supposed from the large number of publications on the positive friction and wear properties. Experiments on the wear behaviour of a-C-H layers using Al_2O_3 balls as counter bodies with identical loads show a clear dependence on temperature: while the a-C-H layer easily copes with the loads at room temperature (24 °C), it wears through after the same test duration at 150 °C and completely fails at 250 °C.

10 Vacuum hot extraction device with mass spectrometer gas detection, hydrogen emission curve from a Ti alloy

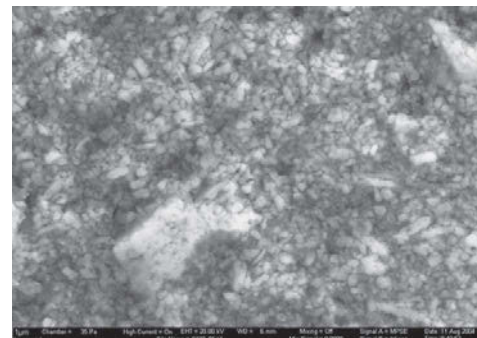
11 Al_2O_3 layer on corundum after heat treatment at 1200 °C; left: TEM image of a focused ion beam (FIB) lamella, right: SEM image of the layer (polished cross-section)

11

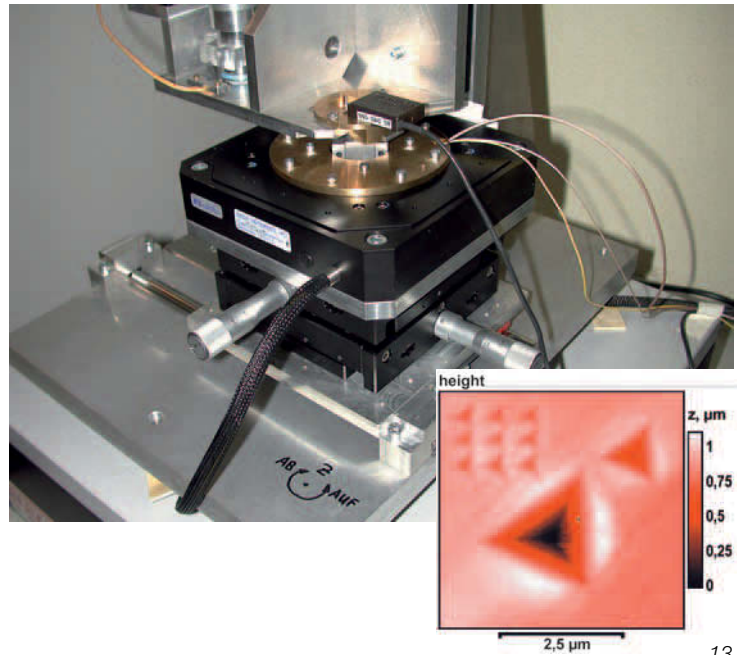


Higher gas inlet temperatures improve turbine efficiency. In order to achieve this goal, heat-insulating layer systems for the metallic blades and vanes are being developed. Conventional heat-insulating layer systems for turbine blades consist of an adhesion layer (Al_2O_3 forming alloy) and an external layer of zircon oxide ceramics stabilized by Y_2O_3 . Applying an alumina layer (corundum) to the adhesion layer may result in improvement. This should serve as a diffusion barrier for oxygen from the atmosphere and for aluminium from the adhesion layer and thus retard the oxidation of the latter and the formation of a thick thermally grown oxide coating. If the thickness of a thermally grown oxide coating reaches about 10 μm , delaminations occur and the heat-insulating layers flake off. The sol suspension method offers a cost-efficient alternative for the production of the additional alumina or corundum layers. Here the substrates will be coated with mixtures of alumo or boehmite sols with corundum suspensions and different additional heat treatments will be applied. This procedure offers the advantage of germ formation taking place within the volume (and not on the surface) of the substrates as compared to chemical or physical vapour deposition (CVD or PVD) procedures. The result is a fine-grained structure without any texture and thus there are no directional channels available for oxygen diffusion. Such a structure should, in addition, ensure higher strength of the layers than the directional grain growth in the other procedures mentioned.

In co-operation with the University of Kiel, coated implant materials were investigated and evaluated for the use in dental surgery. Preceding in-vitro studies showed that TiN and ZrN layers, which were deposited using



PVD procedures on titanium implants, may markedly reduce bacterial accumulation, in particular on critical spots of the implant. On the other hand, a maximum number of focal contacts with the surrounding tissue is necessary. Here it was not clear to what extent this was affected by the coating and the topography of the uncoated and coated surfaces. It was proved that extremely smooth surfaces (polished titanium and coated glass) provide the highest number of focal contacts, regardless of how the sometimes variable roughness is formed in the nanometer range. In the micrometer range, however, increasing roughness results in a clear reduction of the number of focal contacts. Despite extensive modifications in coating technology its influence on both layer systems was relatively small. However, adhesion capability of fibroblasts and wear resistance of the surface can considerably be increased by the coating.



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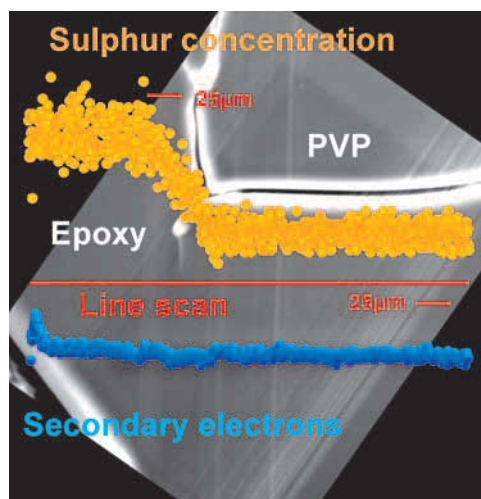
Modern techniques for mounting micro-chips frequently involve adhesives. Instead of being soldered, chips are glued. This produces direct interfaces between epoxides and, for example, copper. Using scanning force microscopy (SFM), in particular through the contrasts of mechanical properties and energy dispersive X-ray analysis (EDX) it could be proved that the closeness of copper to hardening epoxy adhesives strongly affects the local stiffness and chemical composition. For the assessment of the reliability of such systems, understanding of hardening processes on a small scale is of great importance because the adhesive layers must be sufficiently thin to achieve careful use of material and good heat transfer.

In small components, in particular with heterogeneous materials, microindentation (Figure 13) is often the only method to be used to determine local values for the mechanical properties such as hardness and stiffness. Also, the measuring point placement must be defined with high accuracy. Therefore a measuring station was built, which enables both imaging scanning of the surface (similar to scanning force microscope – SFM), and – without changing the measuring system – specified measurements of stiffness and hardness by nanoindentation.

Concrete repair

Regulations for concrete repair fail to adequately describe the requirements of the supporting substrate. Therefore BAM, commissioned by the Federal Highway Research Institute (BAST), carried out investigations to clarify the influence of roughness and moisture of the substrate. Different roughness values were characterized using the common sand surface procedure and a laser optics method modified by BAM. Moisture states ranging from air-dry to water-saturated were measured using gravimetric methods, electrical resistance measurement and microwave measuring methods. An evaluation of pull-off tests and an assessment based on the relevant regulations showed that neither roughness nor moisture state of the concrete

12



12 Copper distribution at an interface

13 Nanoindentation measuring station, measurements on PMMA



14

14 Shotcrete test stand with specified concrete test samples for the application of repair systems under real conditions

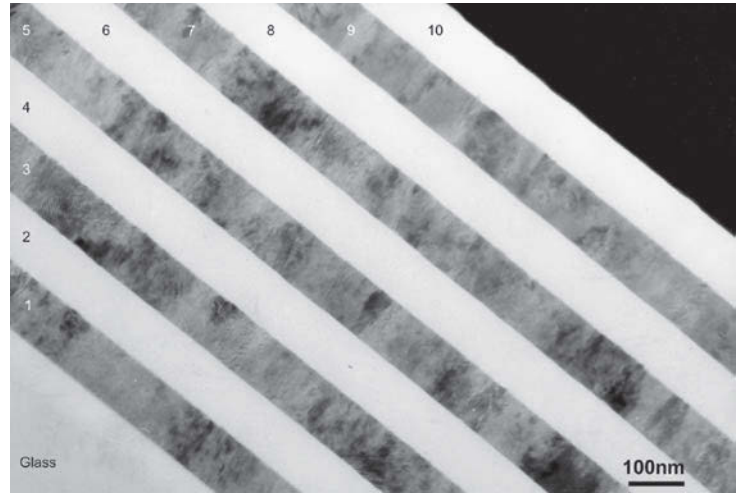
15 Lock chamber walls being inspected

16 TEM image of TiO_2/SiO_2 mixed-layer system on BK7 glass

plates influence the adhesion of shotcrete/ mortar as proved by the pull-off strength test.

A large part of German hydraulic structures from the first half of the 20th century were built using low-quality concrete. These structures often exhibit damage near the surface which requires repair but not replacement. Applying shotcrete layers is a suitable remedy for this purpose. Commercial shotcretes were initially developed for bridge and structural engineering and most of them can also be used in hydraulic engineering. Apart from positive characteristics, such as high lifetime and strength, they exhibit an excessive elastic modulus. Therefore their suitability is restricted to the durable repair of poor concrete. BAM, in co-operation with the Federal Waterways Engineering and Research Institute (BAW) and different shotcrete manufacturers, carried out fundamental investigations for the development of suitable repair systems.

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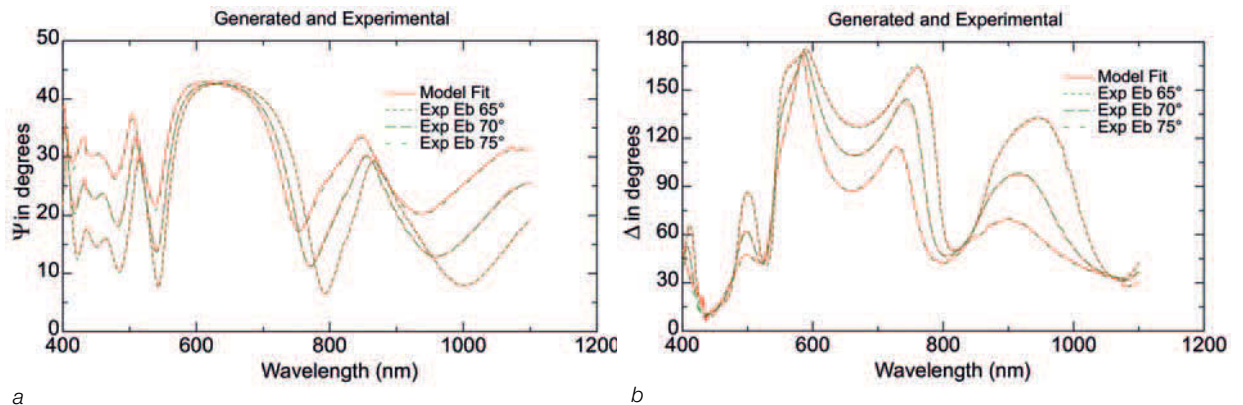


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Reference materials, reference methods

Reference materials are essential for tasks such as calibration of measuring instruments or the validation of measuring methods. Together with reference procedures, they form the basis for a quality-assured determination of material parameters. BAM develops reference materials within the area of materials engineering too. In particular for the validation of new test methods, information can be obtained from research projects, which may be necessary for the preparation, characterisation and potential certification of reference materials. Thus reference samples for nanoindentation and for the quantitative determination of elastic coefficients using scanning force microscopy were developed. Different polymers and different manufacturing processes were tested to ensure that the mechanical characteristics do not change over the entire depth of the actual indentation. Polymethyl methacrylate (PMMA) excels by the fact that it is completely non-crystalline and commercially available in very high purity. The selected preparation method avoided the formation of an undesirable layer on the surface and the achieved surface roughness is very small. A sample, whose elastic modulus and transversal contraction ratio were previously determined, is available now. Those users who want to characterize the last 100 nm of their indentation tip are particularly interested since this has not been possible at specified force resolution using hard materials.

17



a

b

Layer systems (TiN, TiC, VN, VC monolayers on 100Cr6 steel and Ti/Al and TiO₂/SiO₂ mixed layers on 100Cr6 steel and/or BK7 glass), which have already been used in various interlaboratory comparisons and EU projects in the field of depth profile analysis, were entered in the BAM catalogue of reference materials after being certified. For certification, the layer thickness was measured on batch reference samples using scanning electron microscopy (SEM) or, in the case of TiO₂/SiO₂ mixed layers, additionally using transmission electron microscopy (TEM) in transversal sections, i.e. destructively. Each sample was also tested non-destructively. The following non-destructive testing methods were used: grazing-incidence X-ray diffraction (GIXRD) for Ti/Al mixed layers, surface acoustic waves (SAW) for TiN, TiC, VN and VC monolayers and spectroscopic ellipsometry (SE) for TiO₂/SiO₂ mixed layers. Ellipsometry was assigned a key role here

since it was also used to validate the transversal section procedure. A SiO₂ monolayer was used which had been measured using both SEM and ellipsometry. To determine the optical material constants (Figures 16 and 17) and to assess result uncertainty the Publicly Available Specification (PAS) 1022 entitled "Reference method for the determination of optical and dielectric material properties and layer thicknesses of thin layers using ellipsometry", developed by BAM, was used. The efficiency of the method has been proved by a very good agreement between simulation and measurement of the amplitude and phase information for the TiO₂/SiO₂ mixed-layer system. For the 10-layer system (5 double layers with a nominal layer thickness of 100 nm) an agreement of approx. ± 0.5% (5 nm) of the average values of the overall coating thickness was obtained for TEM, which is a very good value for altogether 11 interfaces.

17 SE spectrum (simulation and measurement at different angles of incidence) of the TiO₂/SiO₂ mixed-layer system on BK7 glass (BAM-L101),
a) amplitude,
b) phase information

Technical and Scientific Service Functions



Special force transducer with a force transmitter unit for the monitoring of retaining hawsers of an oil platform (left)

Transport ship with oil platform on the way to the site (middle)

Two installed special force transducers on the oil platform (right)

Due to its multidisciplinary structure, BAM is in the position of being able to perform work not only in the areas of its original main project focuses, but also in fields where a multidisciplinary approach is required. In particular these are quality assurance in testing and approval, development and maintenance of information systems, world-wide applicable methods for conformity assessment and further development of measurement and test methods.

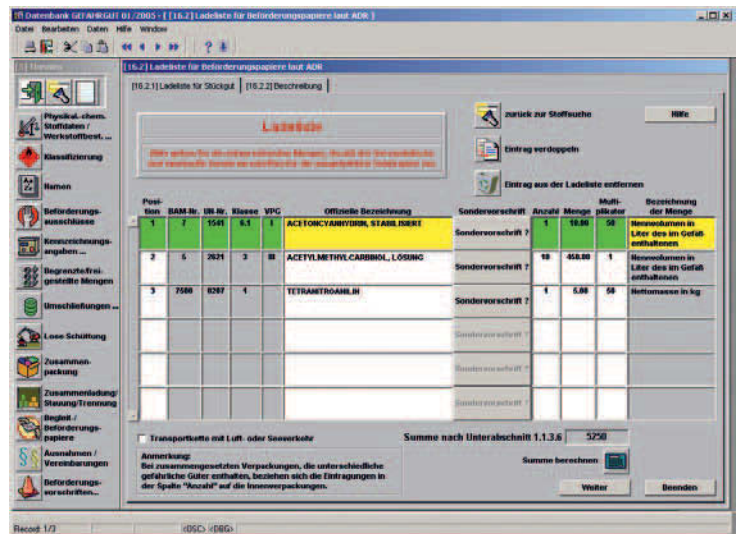
Information systems

The information system Database Dangerous Goods (Datenbank GEFAHRGUT) has been on the market for four years and the number of users is steadily increasing. The database contributes to a high level of safety in the transport of dangerous goods in two ways: Customers can gather information via its user interface and companies integrate the data in their software to optimise value creation chains by controlling enterprise procedures.

The database and the quality-tested data are continuously being enhanced. Version 5.0 was published at the turn of the year 2004/2005. It contains updated regulations for air, sea, inland waterway and land transport which come into effect on 1 January 2005.

The database contains a new module for the creation of transport documents for the road transport. The correct compilation of transport documents for dangerous goods can avoid accidents where dangerous goods are released and help limit the effects of accidents. In the module for transport documents the user is interactively guided by the program in such a way that errors, which could be made by a less competent user are technically excluded, as far as possible. Operating instructions supplement the guidance throughout the program. As a result the user obtains a transport document, which is orientated towards the UN model regulations and implements the requirements of the dangerous goods regulations for the road transport. The project is supported by the Federal Ministry for Transport, Building and Housing (BMVBW).

Technical exchange has been improved in other database and internet applications.



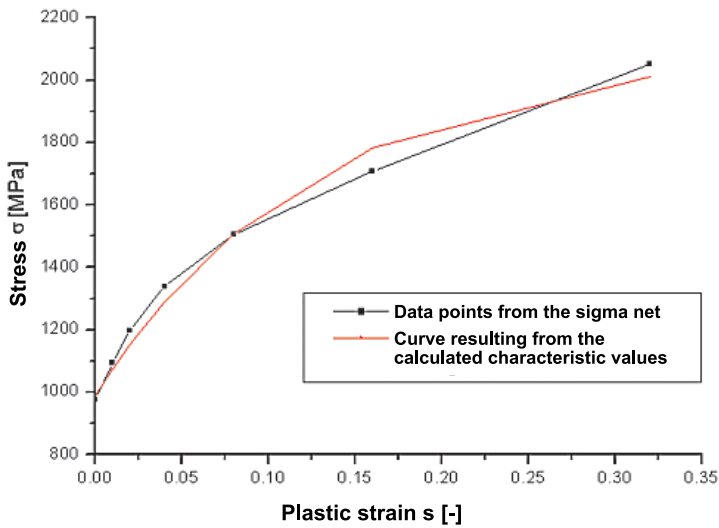
1 Transport loading list documents as per ADR

They are available for scientific information exchange to national and international user groups such as the Ellipsometry Working Group, the working groups of the International Laboratory Accreditation Cooperation (ILAC) and the members of the “COORETEC CO₂ Reduction Technology” national working group.

Advancement and reliability assessment of sensor technology, measurement and test technology

The instrumented indentation test represents a new, higher level of the classical hardness test and it ranks among the most modern test methods in the field of material characterisation. This procedure enables the investigation of elastic and plastic material behaviour as well as the influence of the load period. It is therefore particularly attractive to industrial users. Depending on the objective of the test, various geometries of the indenter can be used. The instrumented indentation test is a low-destruction test and permits the testing of all materials and material combinations as well as material behaviour e.g. hard metal creep. It is universally applicable regarding test force, indentation depth and area of application. A particular advantage is the possibility of local testing without sampling and sample preparation. This is of great importance for damage analysis and/or material characterisation for very expensive or embedded components. Unlike conventional hardness testing methods the computer-

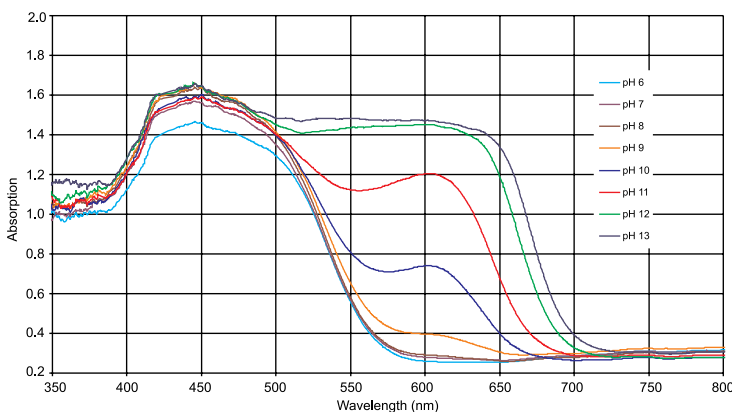
Data bases and internet applications:
www.dgg.bam.de
www.ake.bam.de/
www.eptis.bam.de/iris/



2 Stress-strain curves for 402 HBW steel determined using a spherical indenter and neuronal nets

aided evaluation of the measurement data leads to objective results. Ongoing research and development activities have the objective of using instrumented indentation tests for the determination of material properties which can, so far, only be determined by traction tests. In co-operation with the Institute for Materials Research II of the Karlsruhe Research Centre and the Wildau Technical High School for Applied Sciences the question of whether measurement data evaluation using neuronal nets would enable the determination of the traction characteristics of a material from the instrumented indentation test was investigated. The available investigation and simulation results look good (Figure 2). The material Young's modulus and yield point parameters are in close agreement.

3 Change of reflection spectrum of a fibre optical pH sensor in buffer solutions in the pH range of 13 to 6



direct danger to humans and the environment. The risks of basic components and structures can be reduced by sensors embedded into structures or components for continuous monitoring. Mechanical parameters have already monitored in this way for a long time. For buildings and structures exposed to severe chemical impacts by acids (e.g. cooling tower shells with flowing condensate, bored piles, rock anchors or foundations in carbonic acid containing groundwater, sewers) there is so far no possibility of monitoring the depth of chemical damage. This type of damage is particularly critical because there is a long incubation period and it usually only becomes visible just prior to the total destruction. The reduction of corrosion protection of the reinforcing steels and a subsequent degeneration can lead to a sudden failure of the component or the structure. To solve this problem a new fibre-optic sensor has been developed for monitoring the alkalinity of the concrete in co-operation with universities, small and medium enterprises (SME) and the Federal Institute for Geosciences and Raw Materials (BGR). The sensor is directly embedded into the concrete and works within a pH value range from 12 to 9 (Figure 3). The functioning of the new pH optode was successfully proven in the laboratory. Follow-up field tests should prove the practical suitability.

The reference procedure developed in BAM for the testing and calibration of large test machines and/or large force transducers up to 36 MN this year made a special contribution to the safety of high risk structures. It was used to test and calibrate special force transducers manufactured by a British company for Norwegian offshore oil platforms. Such force transducers are used for the monitoring of the retaining hawsers (illustration p. 46). Since high storm seas may cause extreme loads and high danger potential to humans and the environment, reliability is paramount. This BAM reference procedure has proved its efficiency in an international comparative measurement (Figure 4) and enabled the testing and calibration of these special force transducers up to 17 MN according to ISO 7500-1 Class 1.

Quality assurance in testing and approval

After in-depth consultation with the regulatory bodies concerned, BAM published the Dangerous Goods Regulation BAM-GGR 001 anew. It deals with the quality assurance and the official monitoring of the production of packagings, IBCs and large packagings for the transport of dangerous goods. Motives for the revision of the decentralized organised system of approved quality assurance measures by manufacturers, and the monitoring through supervisory bodies recognized by BAM, were a stronger adherence to the rules specified in the European regulations, the strengthening of the idea of quality management (ISO 9001) and the recognition of services provided by quality associations. The technical contents of the GGR 001 are compatible with the ISO DIS 16106 draft standard, which was initiated from the German side with the ambition of internationalisation of quality assurance in this area.

Department IV was accredited under the framework of the development and maintenance of a quality system for BAM's research, testing and advisory activities. Thus all specialist departments of BAM now have an internationally recognized confirmation of their competence for the basic procedures in the areas of material testing and analytic chemistry. An overall internal audit system with regularly trained auditors was also installed.

Conformity assessment and accreditation

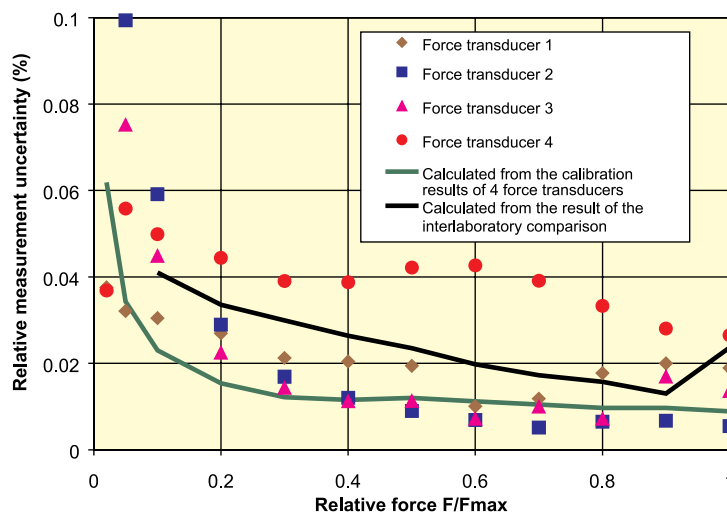
International comparability and acknowledgment of laboratories and consulting engineers test results is forwarded by BAM through its conformity assessment and accreditation activities. BAM continuously participates in standardization and has advised the Federal Ministry for Economy and Labour (BMWA) on the fundamentals and consequences when preparing a German accreditation law. Relevant standards, draft standards and normative documents are specified in *Table 1*.

In the secretariat work of the International Accreditation Forum (IAF) the following mutual acknowledgment agreements (MLA) were signed by the German side: for Environment Monitoring Systems (UMS) by the German Association for Accreditation GmbH (TGA) and for Product Certification by the German Accreditation System for Testing GmbH (DAP) and the German Accreditation Body for Technology e. V. (DATech).

The EUROLAB secretariat acting in BAM achieved an agreement with the European Cooperation for Accreditation (EA) that the fulfilment of the substantial requirements of ISO 9001 can be confirmed in an accompanying letter to an accreditation certificate.

Sponsored by the Federal Ministries for Economy and Labour as well as for Economic

The international IAF recognition agreement includes the certificates of BAM's product certification body accredited by DAP in a positive way. See www.iaf.nu



4 Measurement uncertainty of BAM's reference method for testing and calibration of large force transducers in the range up to 20 MN

Tabelle 1
Standards, draft standards and normative documents (with participation of BAM-S.4)

| | | |
|---------------------------------------|--|---|
| ISO/IEC 17000 | Conformity assessment- General vocabulary | Rev. Guide 2:1996, Section 12 bis 17 |
| ISO/IEC Guide 67 | Fundamentals of product certification | Published in 2004 |
| ISO/IEC Guide 60 | Conformity assessment – Code of good practice | Published in 2004 |
| ISO/IEC 17011 | General requirements for accreditation bodies accrediting CABs | Summary DIN EN 45003 DIN EN 45010 DIN V 55390 |
| ISO/IEC 17040 | General requirements for peer assessment of conformity assessment bodies | |
| ISO/IEC 17021 | General requirements for bodies operating assessment and certification of management systems | Summary of ISO/IEC Guides 62 and 66 |
| PAS 17001; PAS 17002; PAS 17003 | Impartiality and related bodies; Confidentiality; Complaints and appeals | Published in 2004 |
| ISO/IEC 17050 -1 ; -2 | Supplier's declaration of conformity; Supporting documents | Published in 2004 |
| ISO/IEC 17025/A1 | Amendment to ISO/IEC 17025 | Adaption of DIN EN ISO/IEC 17025 to ISO 9001:2000 |

Co-operation the International Technology Transfer Working Group (iCATT) supported altogether eleven projects from those countries that joined the European Union in 2004, potential ascension countries, developing countries and the GUS states in the development of conformity assessment systems.

BAM also acts as a notified body of the EU in the context of several guidelines and the BAM certification body (BZS) as one of the central bodies of the states for safety engineering (ZLS) and an accredited body by DAP (DAP

re-accreditation 2004). The certification authorities for explosives and dangerous good packaging acting within their own certification responsibility participate in the committees of BZS. BAM certificates are particularly sought after within the field of safety engineering. The extension of the scope of BZS since 2003 has been proved. Within the three new certification fields, three new certificates were issued for "Explosion prevention" in 2004, 26 for "Pressure devices" and eight for "General" (special certification within legally non regulated areas).

| Budget | 2004 | 2003 | 2002 |
|---|-------------|-------------|-------------|
| Federal budget | 98,3 | 95,1 | 93,7 |
| comprising: | | | |
| Staff expenses | 63,4 | 64,5 | 64,0 |
| Investments | 16,5 | 18,9 | 15,1 |
| Other expenses | 14,1 | 14,2 | 14,8 |
| Fees for testing, approvals, etc. | 7,6 (6,4*) | 6,7 (6,1*) | 6,6 (6,4*) |
| Research funds (external sources) | 13,2 | 11,2 | 13,1 |

Budget numbers for the fiscal years are in Million EURO

* Transferred to the Federal Ministry of Finance

| Personnel | 2004 | 2003 | 2002 |
|--|-------------|-------------|-------------|
| Permanent staff (approx. 60% scientists and engineers) | 1151 | 1173,5 | 1196 |
| Temporary staff (research projects) | 180 | 179 | 182 |
| Temporary staff (other) | 131 | 135 | 156 |
| Apprentices, trainees | 124 | 122 | 110 |
| Visiting scientists, guests | 43 | 34 | 30 |
| Total | 1629 | 1643,5 | 1674 |

Statistics **in 2004**

Research and development

| | |
|---------------------------------------|-----|
| Scientific and technical publications | 509 |
| Lectures, presentations, courses | 812 |

Testing, analysis, approvals

| | |
|---------------------------------|-------|
| Test reports, certificates, ca. | 6.000 |
|---------------------------------|-------|

Consultation and information

| | |
|--|-------|
| National and international commitments (boards, committees, organisations), thereof: | 1.264 |
| Statutory and regulation committees | 142 |
| Standardisation committees (ISO, CEN, DIN) | 553 |



Status: 1 May 2005

Chairman of the Advisory Council

Dr. Eckhard Franz (Federal Ministry of Economics and Labour)

President

Prof. Dr. M. Hennecke phone: 1000

Vice-President

Dr. Th. Böllinghaus phone: 1020

Member of the Presidential Board

Dr. N. Pfeil phone: 1030

Assistant to the Presidential Board

Ms Dr. A. Recknagel phone: 1012 Dr. B. Scharfel phone: 1021

Section 6.1 Public Relations and Marketing

Dr. J. Lexow phone: 1001

Section 6.2 Strategic Planning, Controlling

N. N.

DEPARTMENT Z Administration and Internal Services

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Section Z.1 Organization

J. Fischer phone: 2100

Section Z.2 Budget

M. Fischer phone: 2120

Section Z.3 Personnel

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Section Z.4 Legal Office, Purchasing Office

Ms Ch. von Vangerow phone: 2140

Section Z.5 Professional Training

Ms A. Rau phone: 2150

Section Z.6 Internal Facilities

Dr. G. Plauk phone: 2200

Section Z.7 Buildings and Technical Services

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Section Z.9 Information, Documentation

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DEPARTMENT V Materials Engineering

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DEPARTMENT VI Performance of Polymeric Materials

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DEPARTMENT VII Safety of Structures

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DEPARTMENT VIII Materials Protection, Non-Destructive Testing

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DEPARTMENT S Interdisciplinary Scientific and Technological Operations

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Plant Safety: Constructional Explosion Protection Dr. R. Grätz 3488 Plant Safety: Safety Engineering and Risk Assessment Dr. B. Schalau 4496 Plant Safety: Acetylene Ms Dr. C. Wilrich 3492 Safety Related Properties of Gases Dr. V. Schröder 3440 Safe Handling of Oxygen Dr. Ch. Binder 1211 Gas Detectors Dr. V. Lohse 3446

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Testing and Investigation, Load Securing Dr. Th. Goedecke 1311 Approval and Use Dr. P. Blümel 1312 Quality Assurance and Control W. Kraus 1313 Database Dangerous Goods – Datenbank GEFÄHRGUT Dr. M. Lieske 1314 Information Management - Technical and Public Safety Dr. M. Nitsche 1306

Division IV.1 Biology Materials Protection and Environmental Issues

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Materials Resistance against Microorganisms Ms Dr. I. Stephan 1412 Materials Protection against Fungi and Insects Dr. R. Plarre 3832 Microbiology, Reference Organisms Dr. H.-J. Kunte 3849

Division V.1 Microstructure of Engineering Materials

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Quantitative Materialography Dr. D. Bettge 1512 Electron Microscopy Dr. W. Österle 1511 Constitution, Thermodynamics Dr. A. Kranzmann 1021 Phase Analysis Dr. G. Nolze 3513

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Chemical Resistance Dr. H. Schröder 1611 Thermal Resistance Dr. H.-E. Maneck 1612 Combustion Behaviour and Fire Retardancy Dr. B. Scharfel 1021 Climatic Resistance Dr. V. Wachtendorf 1613 Elastomers and Reference Materials Dr. J. Keim 1614

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Composition, Microstructure and Transport Mechanisms Dr. D. Hoffmann 1712 Concrete Technology, Repair Systems Dr. H.-C. Kühne 3229 Mechanical Properties Dr. H. Winkler 1713 Condition Analysis and Damage Diagnosis Dr. F. Weise 3237 Bituminous Materials and Sealing Technology Ch. Recknagel 3296

Division VIII.1 Tribology and Wear Protection

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Optimisation of Friction and Wear Protection Dr. D. Klaffke 1812 Tribological Failure Analysis Dr. M. Woydt 1811 Hydrogen and Cryotribology Dr. Th. Gradt 3531 Micro-/Nanotribology and Modelling Dr. H. Klotz 1814 High Temperature Tribology Dr. M. Woydt 1811

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Reliability of Testing Systems Dr. C. Ullner 1914 Fibre Optic Sensors Dr. W. Habel 1916 Sensors and Measurement Systems Dr. J. Kurth 1912 Optical Measurement Methods and Reference Materials K.-P. Gründer 3633

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Organic Reference Materials, Proficiency Testing Dr. R. Becker 1121 Organic Trace Analysis Dr. T. Win 1122 Bio and Food Analysis Ms Dr. I. Nehls 1120 Analysis of Sum Parameters Dr. M. Koch 1124

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Assessment of Dangerous Goods/ Substances F. Krischok 3707 Flammable Bulk Materials und Dusts Dr. U. Krause 4442 Explosive Substances in Chemical Industries Dr. K.-D. Wehrstedt 4413 Explosion Dynamics Dr. H. Hieronymus 3426 Chemical Process Safety Ms Dr. U. Rockland 3409 Information Systems, CHEMSAFE Ms Dr. M. Molnarne 3452

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Tank Safety, Mechanical and Thermal Investigations A. Ulrich 1326 Tank Farms, Incident Assessment Dr. Ch. Balke 1322 Risk Management Dr. R. Konersmann 3936 Tanks for Transport, Transport Technology Dr. M. Pötsch 1323 Pressure Equipment Dr. G. Mair 1324 Pressure Equipment – Fittings Dr. S. Aris 1244 Compatibility Assessment of Materials for Tanks and Containers Ms M. Weltshev 3959

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Environmental Impact and Damage Mechanisms Dr. M. Bücker 4144 Emissions from Materials Dr. O. Jann 1422 Environmental Compatibility of Textiles Dr. W. Schiller 1423 Fibres and Fibre Products Dr. S. Seeger 3802

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Advanced Metallic Materials Dr. H. Klingelhöffer 1521 Advanced Ceramic Materials Ms Dr. B. Rehmer 1522 High Strain Rate Loading Dr. A. Hamann 1523 Modelling, Simulation Dr. B. Fedelich 3104

Division VI.2 Mechanics of Polymers and Composites

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Scanning Probe Microscopy and Nanotechnology Dr. H. Sturm 1624 Interfaces and Thin Films Dr. E. Schulz 1622 Micromechanics Dr. G. Kallinka 4312 Fatigue Testing, Failure Analysis, Damage Mechanics Dr. H.-V. Rudolph 1605 Materials Mechanics, Dangerous Goods Packaging, Acoustic Emission Testing Dr. J. Bohse 1302

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Thin Film Technology, Electrochemistry Surface Test Methods Dr. U. Beck 1821 Pulse Laser Technology, Laser Safety Dr. J. Krüger 1822 Surface and Thin Film Analysis, In-situ Preparation Dr. W. Unger 1823 Chemical Sensor Technology, Sol Gel Technology Dr. Th. Hübert 1824 Nanotechnology Dr. G. Reiners 1820

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Inspector for Data Protection Ms Ch. von Vangerow phone: 2140

Occupational Safety and Health, In-house Environmental Protection A. Jaenicke phone: 1006

Quality Management Representative J. Ehreke phone: 3174

Staff Council (Chairman) B. Sloeck phone: 2500

Equal Opportunities Representative Ms G. Walter phone: 2310

Representative for Severely Disabled Persons S. Proll phone: 2505



Ms Dr. M. Wioka phone: 1942

BAM-Certification Body Secretariat

Dr. R. Schmidt phone: 3715

* administered by

The main areas of expertise of the technical departments

Department
I

Analytical Chemistry; Reference Materials

Provision and assessment of reliable methods and reference materials in analytical chemistry.

Department
II

Chemical Safety Engineering

Guaranteeing and improving safety in the handling of explosive, flammable or otherwise dangerously reactive substances, systems of substances or articles as well as pressurised gases in the field of legislation on dangerous substances and goods, explosives and weapons, equipment safety and immission control.

Department
III

Containment Systems for Dangerous Materials

Ensuring and improving the level of safety of containment systems for the transport and storage of hazardous materials, including safe transport and storage practices. BAM in its role as an element of the Federal administration is the competent authority in this field.

Department
IV

Environmental Compatibility of Materials

Multidisciplinary research on long-term interactions between materials and the environment. The investigations cover a range of technical, ecological and economic considerations and the results are made available to the government, the public and to industry to help to promote the environmental compatibility of manufactured goods.

Department
V

Materials Engineering

Safety and reliability of components under mechanical and thermal loading – an interdisciplinary approach – comprising materials sciences, design and manufacturing technology.

Department
VI

Performance of Polymeric Materials

The interdisciplinary assessment of the functional safety of polymers, their component parts and functional interfaces under mechanical, chemical, thermal and climatic stresses; developing modified test procedures; the development and certification of reference materials.

Department
VII

Safety of Structures

Examination and evaluation of the safety, reliability and durability of building materials, structures and technical equipment exposed to various climatic, corrosive and other conditions and corrosion in equipment and plants.

Department
VIII

Materials Protection; Non-destructive Testing

Ensuring the proper functioning of materials during use in order to prevent impairment; the development, validation and provision of non-destructive test procedures for the early detection of flaws and enabling the safe functioning of technical components and plants.

Department
S

Interdisciplinary Scientific and Technological Operations

Finding solutions to interdisciplinary scientific and technological problems in testing and information management, provision of support in these areas; management of BAM's quality assurance system; guidance on accreditation in Germany and improvements in quality within the testing sector.

A Brief History of BAM

- 1870 The Prussian Ministry of Commerce, Trade and Public Works announces the establishment of a Mechanical and Technical Research Institute. Its task is to perform experiments of general scientific and public interest and to test the strength of components.
- 1904 The Royal Materials Testing Office is established in Berlin-Dahlem following the merger of the Royal Mechanical Testing Institute with the Royal Testing Station for Building Materials (founded in 1875) and the Royal Chemical Technical Testing Office (founded in 1877).
- 1919 Renamed the Public Materials Testing Office (MPA), the institute is responsible to the Prussian Ministry of Science, Fine Arts and Public Education; from 1936 on the Public X-ray Investigation Office is included.
- 1920 The State Chemical Technical Institute (CTR) is established under the State Ministry of the Interior from the Military Testing Office, established in 1889 as the Central Research Office for Explosives.
- 1945 MPA and CTR are united and operate under the jurisdiction of Berlin City Council.
- 1954 The Federal Republic of Germany takes over responsibility for MPA/CTA as Federal Institute for Mechanical and Chemical Testing (BAM), renamed the Federal Institute for Materials Testing in 1956. In addition BAM takes over responsibility for public materials testing for the state of Berlin.
- 1969 Under the Statute on Explosive Substances BAM is granted the status of senior federal authority; an amendment to the law in 1986 adds the word "research" to BAM's title.
- 1975 Under the Statute on the Transport of Hazardous Goods BAM is given greater responsibility in the field of public technical safety.
- 1990 German reunification and a recommendation from the German Scientific Council strengthen BAM's function as a federal chemical technical institute. Its personnel is increased by staff gained from the defunct Office for Standardisation, Measurements and Product Testing (ASMW) and Academy of Sciences in the former GDR. Responsibility for public testing for Berlin is gradually ended.
- 1995 Following an external evaluation and extensive reorganisation, BAM is given a new statute, revised management structures and methods and a future-oriented profile as an essential element of the technical and scientific infrastructure of the Federal Republic of Germany under a decree from the Federal Ministry of Economics.