The German Materials Research Program 1985-1994

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1. Introduction

Germany has a tradition of long standing both in classical sciences like physics and chemistry, the basis for materials science, as well as in materials engineering. In the late sixties the far-reaching importance of materials development and processing was emphasized in an industrial memorandum on materials technology. In 1971 the developmental focal points of this study stimulated the Bundesministerium fiir Forschung und Technologic (BMFT) (Federal Ministry for Research and Technology) to launch a Research and Development (R&D) Program on New Technologies. For the first time the significance of materials technology as basic technology was underlined in this program, referring to its close connection to other governmental R&D programs in the field of e.g. (nuclear) physics, transportation, marine technology, energy and medicine. Two objectives of this materials technology program are worthy of mention.

Firstly materials such as metals, ceramics, polymers and composites each have their unique properties and thus have their share in different applications. However, due to continuous improvement they are also competing with each other. Sometimes substitution may be the consequence or even the goal of R&D activities, dependent on the application profile.

Secondly the tight relationship between materials development and materials processing: materials R&D has to go along with materials processing and engineering in order to demonstrate the potential of reproducible and reliable high performance materials/components. This presupposes interdisciplinary thinking and working between the natural and engineering sciences.

These ideas have been fundamental to all the programs of the BMFT, past present and future. The most important one is called **Materials Research Program** (1985-1994). It dealt with five focal points: ceramics, powder metallurgy, metallic high temperature materials and special materials, polymers and composites, and aimed at application oriented improvements of their properties. The general aspects of the Materials Research Program are reviewed in this report. The organizational structure and financial aspects are described, as well as the objectives and research elements of the five focal points.

This review is completed by some comments on materials research and materials processing sponsored in other R&D programs, by other Federal or State Departments in Germany. Also, there are some remarks on international cooperation. The review starts with an overview of German research institutes. In a forthcoming paper the planned German program Materials for Key Technologies will be presented.

2. Research institutes

A large number of research institutes in Universities, in the Max Planck Gesellschaft (MPG), the Fraunhofer Gesellschaft ffir Angewandte Forschung (FhG) (Fraunhofer Society for Applied Research) and the government laboratories as well as numerous industrial laboratories are involved in materials science and engineering. A comparison with the situation in other advanced industrial countries shows, however, that in Germany there are presently still only few interdisciplinary "materials science and engineering centers." The main research organizations involved in materials science and technology are presented in this paragraph. No attempt has been made to present a complete review.

University research is mostly funded through grants from the Deutsche Forschungsgemeinschaft (DFG), (German Science Foundation), an independent organization, which receives its funds from the government. BMFT and State Governments pay 50% each of the budget of the DFG; in addition, the BMFT pays for special programs in selected areas of special scientific importance, including some in the area of materials. Projects are selected by the DFG, using a peer review process. The DFG spends approximately 60 Mio. DM per year in (basic) materials science and engineering.

Also the Volkswagen-Stiftung (Volkswagen-Foundation) is to be mentioned with respect to basic research especially in University institutes. The financial support covers scientific equipment such as electron microscopes.

The Max Planck Society MPG is also an independent scientific organization, with some 59 institutes of basic research, whose budget is also shared 50/50 by BMFT and State Governments. Research funds for the MPG are provided as a lump sum; the allocation of funds for specific projects within the institutes is essentially decided by the directors and senior scientists of the institutes, with little interference by the MPG-management and practically no interference by the governments. The MP-Institute for Iron Research in Düsseldorf is jointly operated by MPG and Verein Deutscher Eisen- und Hüttenleute E.V. (VDEh), a speciality within the MPG. They contribute 50% each. The total amount spent in the MP Institutes for materials science including solid state physics amounts to approximately 140 Mio. DM per year (including contracts, e.g., from industry).

The Institutes of the Fraunhofer Society (FhG) are primarily concerned with applied research and engineering work in a number of disciplines.There are some 45 institutes. They are oriented towards the research needs of specific industrial sectors or areas of technology and more than one third are involved in materials research. Approximately 20% of their budget is provided by the BMFT and State Governments, and the balance is earned from contracts which come primarily from industry. The FhG spends a total of some 250 Mio. DM per year for materials research, processing and testing.

A large part of the materials science and engineering in Germany is performed in government operated laboratories. There are first of all the Großforschungseinrichtungen (GFE) (National Research Centers, like DLR in Braunschweig, Cologne and Stuttgart, GKSS in Geesthacht, KFA in Jülich and KFK in Karlsruhe). Their budget is shared 90/10 by BMFT and State Governments, respectively. The general R&D topics are wide spread, e.g., high energy and solid state physics, biotechnology, information technology, medical science and aerospace science and engineering. The predominant part of the materials research performed in the GFE's contributes to the solution of the engineering problems in the area of non nuclear energy, aerospace and information technology. There is also some minor fundamental theoretical and experimental research dedicated to materials science. Furthermore there are some Institute der Blauen Liste (historically listed up on a blue paper). BMFT and State Governments each contribute 50% of the budget. A few of them are dedicated to materials science and engineering, especially in the Eastern part of Germany. Their R&D deals with long term topics of governmental and industrial relevance. While it is difficult to determine precisely how much of the total research effort of these government operated laboratories is devoted to materials, the BMFT part may be estimated roughly to amount to 110 Mio. DM per year.

The Material-Prtifungs-Amter (Institutes for Materials Testing) are dedicated to the short and medium term needs of industry. They are operated and mainly financed by the Bundesministerium für Wirtschaft (BMWi) (Federal Ministry for Economy), e.g., the Bundesanstalt für Materialforschung und -prüfung (BAM), and by the Wirtschaftsministerien der Lander (State Ministries for Economy). The range of their activities varies from preparations, implementations and surveyance of legal regulations mainly in the area of technical safety and environmental problems, prenormative and normative work, to all sorts of testing mainly of components under service conditions. Of course there are also contracts from industry. There is a small amount of R&D in these institutes in order to advance the state-of-the-art in the area of materials testing.

Approximately 60-70% of the research mentioned so far and performed in Universities, MPG, FhG and the government laboratories is funded by the Federal Government, BMFT being the central funding body. Out of the 9, 2 Billion DM budget of that Ministry in 1992, about 250 Mio. DM are channeled directly into materials science and engineering via the Materials Research Program to be presented next and the government laboratories operated by BMFT. There is furthermore much applied materials R&D sponsored by BMFT in its programs on energy, aerospace, information and environmental technology. Estimating the materials part within these R&D programs to be in the order of 20%, this amounts to another 700 Mio. DM.

3. Materials Research Program

General aspects

In October 1985 the BMFT inaugurated a Materials Research Program [1]. It ranked with the national key R&D programs of the BMFT together with, for example, those on information technology and biotechnology. The Materials Research Program underlined the technical and economic significance of advanced materials and therefore supported R&D in order to strengthen and secure long term technological competitiveness. The central objectives of this R&D program were the identification of a limited number of key areas in materials science and engineering with high scientific and economical potential and where major technological progress was to be expected by cooperative, interdisciplinary research efforts in joint projects between institutes and industrial laboratories. The objectives of the Materials Research Program were enhanced by specific organizational measures:

- Projects were favoured for funding where industrial companies and research institutions co-operate on a work-sharing basis in order to reach a common R&D goal [2]. Industry was asked to share 50% of the total expenses including those of the research institutions.
- Additionally some projects were included which aim at the reinforcement of the information and technology transfer, especially to the benefit of small and medium size companies.

The progress of materials science and technology on a broad scale from basic research to precompetitive applied research lay in the centre of interest of the R&D support. The Materials Research Program focussed on five major R&D fields and ran for ten years (1985-1994) with a total budget of 1.1 Billion DM:

- Ceramics (high-performance structural and functional ceramics),
- Powder metallurgy,
- Metallic high-temperature materials and special materials,
- Polymers (new high-performance structural and functional polymers),
- Composites.

Interim assessment of the Materials Research Program was summarized in a report [3] and in two Symposia on Materials Research 1988 and 1991, where results from funded projects were presented and discussed and supplemented by review lectures on materials science and technology [4]. There is a third one planned November 1994. The Materials Research Program has been evaluated by Arthur D. Little Int. Inc., Wiesbaden, Germany, 1992 on behalf of the BMFT [5]. This evaluation comprised an analysis of the effectiveness and efficiency of the program, the identification of future priority goals and recommendations and guide-lines for future activities. A summary is given in [61.

Table 1 gives a financial overview of the five major R&D fields. Institutes and industry get one third and two thirds of the funds, respectively. Metals rank in front of ceramics and polymers.

The Materials Research Program is managed by the Project Agency Materials and Raw Materials Research (PLR) in the Forschungszentrum Jiilich GmbH (KFA). PLR operates on a direct contract basis on behalf of BMFT, i.e. independent from KFA R&D activities. PLR is assisted by high ranking committees of honory scientific experts whose task is to review the proposals at a certain state of maturity. Since 1986, PLR has published annual reports (in German) covering the funded projects.

Table 1. Budget Distribution **(1985-1992)** with Respect to the Five R&D Areas of the German Materials Research Program [5].

Specific aspects

High performance ceramics

Main objective:

Improvement of physical and chemical properties of high performance ceramics and their demonstration in components under service conditions.

Specific objectives and selected examples of topics dealt with in the ceramics area:

- Fundamental experimental and theoretical findings on mechanically and thermally highly stressed ceramic materials with high reliability as basis for efficient components for engines and turbines,
- Plasma, gas and polymer pyrolysis synthesis as well as powder free, wet chemical synthesis, e.g., sol gel methods,
- Conditioning of materials, e.g., $Si₃N₄$, SiC , $Al₂O₃$, $ZrO₂$, $Al₂TiO₅$, cemented carbides and cermets, along with moulding/shaping techniques, e.g., uniaxial, (quasi)-isostatic pressing; injection moulding, (pressure) slip casting, electrophoretic deposition, as well as sintering and post sintering techniques, taking special care for reducing scatter of physical and chemical properties and costs; furthermore materials testing of components under service conditions for automotive, turbine, furnace and pump engineering) [7, 8],
- Active soldering for the joining of ceramics with ceramics and with metals and use at high temperatures,
- Development of special coating techniques for improved physical properties (e.g., diamond, cBN and SiC) as well as chemical properties. Included are thermal barrier coatings, corrosion and oxidation protective layers, hard materials coatings,
- Progress in machining, characterization and testing of oxide and nonoxide ceramics,
- Design of future materials tailored to meet the specific requirements of the applications in the field of mechanical, thermal and chemical engineering as well as electronic, magnetic, optical and biomedical engineering. Some examples with dielectrical, electronic and optical applications are given below:
- Processing of Size-Induced Metal-Insulator-Transition (SIMIT) active powders and structures,
- Piezo ceramics in multi-layer technique based on highly reactive powders,
- Growth and characterization of SiC-single crystals and SiC-epitaxial layers for applications in high temperature electronics and sensors,
- Development of wave guide materials with adjustable optical properties.

There is a parallel activity of the German Science Foundation (DFG) on High Performance Ceramics comprising chemical and physical fundamentals of the manufacturing processes; constitution, microstructure and properties; microstructure-resolved trace analysis.

As an example a rather fundamental joint project

"Development of thermally and mechanically highly stressed ceramic materials with high reliability as basis for efficient components for engines and turbines"

supported to 50% by BMFT and by industry, Bayer AG, Daimler Benz AG, Hoechst AG and MTU GmbH, and performed by the Max-Planck-Institute of Metals Research, Powder Metallurgy Laboratory, Stuttgart, is summarized [9].

A. Objectives

The subject of the now finished project was the development and characterization of advanced ceramics with:

- High strength and thermal shock resistance even at temperatures as high as 1500°C and
- Reliable long-time behaviour, low tendency towards subcritical crack growth, high resistance against creep, fatigue, oxidation and corrosion.

The work was concentrated on $Si₃N₄$ and SiC ceramics.

B. Working program

The research was performed by three working groups (including industrial partners):

- Processing
- Microstructural design
- High temperature properties/life time prediction

C. Illustrations of some results of the project

One of the major aims of the project was to give a basic insight into the optimal microstructural tailoring of $Si₃N₄$ -materials and to understand the conditions of reliability for the application of silicon nitride components.

A prerequisite for a reproducible microstructural design is control of the powder processing parameters. Moreover, optimized processing is essential to enable effective reproducible microstructural design. In the processing investigations special attention has been directed towards the reduction of powder contamination by the application of clean room conditions and the development of processing routes that would exploit the special conditions of a clean room to full advantage.

Microstructural development was focused on investigations into mechanisms of size and shape control for silicon nitride grains. For example parameters such as sintering temperature and time as well as gas pressure variations are basic tools used for a wide range of microstructural modifications. Figure 1 gives an impression of the microstrnctural differences that may be created by only two of the many parameters in the sintering procedure. Furthermore, detailed model experiments have emphasised the role of the α/β -ratio in the

Figure 1. Illustration of grain growth effects by variations of sintering time and sintering temperature. Quantitative studies indicate not only the shift of size but also shape parameters of the silicon nitride grain populations (SEM, plasma etched).

 $Si₃N₄$ starting powder concerning the different stages of phase transformation and grain growth. The control of size and shape distributions of the silicon nitride grains directly affects the path of the intergranular cracks (Fig. 2) in the material and hence the microstructural reinforcement. Therefore, precise characterization methods for the quantitative description of grain parameters have also been developed on the basis of modern stereology. As a consequence of these investigations, strength parameters far in excess of 1200 MPa at room temperature could be achieved combined with good reliability of the materials. This includes a Weibull-modulus $m > 45$ and four point flexure strength $\sigma > 500$ MPa at 1350° C.

For applications at elevated temperatures, not only the grain morphology but also the behaviour of the intergranular phases have been found to be important factors. Depending upon the chemistry (thermodynamics) of the secondary phase, strong influences on the degree of its crystallization have been found. Figure 3 shows a transmission electron micrograph of an almost completely crystallized triple junction between two silicon nitride grains. The thin layer of amorphous structure is clearly visible and remains so after extensive annealing. The thickness of these remaining grain boundary films is controlled by their chemistry (especially the cation concentration). Investigations with dedicated STEM clearly demonstrate the chemical variations in these films (Fig. 4). These grain boundary films should be one of the key factors for the high temperature behaviour of silicon nitride materials. In particular, properties such as creep exhibit drastic response to chemical variations in the grain boundary films. Figure 5 indicates the strong increase in creep deformation caused by the introduction of impurities into the grain boundary phase. The understanding of the primary creep behaviour based on viscous flow has been significantly improved by

Figure 2. Silicon nitride microstructure (SEM, plasma etched) with an intergranular crack. Grains with an overcritical width and higher aspect ratios amplify the energy dissipation during crack propagation by different mechanisms (microstructural reinforcement).

Figure 3. TEM image of an almost completely crystallized triple junction between two silicon nitride grains. At the subgrain boundary and amorphous film always remains which plays a dominating role in the high temperature properties of the ceramic material.

3D-modelling using the finite element method. A better understanding ofthe important thermal shock loading could be achieved. For the investigation, a new thermal shock method has been developed which enables the unstable, stable as well as subcritical crack growth to be followed in-situ. The developed materials exhibit superior mechanical properties including high creep and thermal shock resistance which are sufficient for the practical applications considered. Scaling up the work to achieve similar results in specimens with dimensions of actual components represents a formidable challenge, and particularly if problems associated with structural and chemical gradients are to be avoided. Scientific understanding as well as significant technological improvements have been developed for this purpose.

As an example of a final product, Fig. 6 shows one set of inlet and outlet valves for an automotive combustion engine made from the developed $Si₃N₄$ material. Such valves have already been tested with great success in long term experiments by one of the industrial partners.

In this fundamental project all relevant steps for the development of this manifold class of materials based on silicon nitride have been investigated in a complex manner. Now the basis for tailoring of high strength and reliability parameters as well as for a variety of important properties under high temperature conditions are developed. Therefore, further breakthrough in a number of application fields can be expected.

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Figure 4. Examination with Dedicated STEM. The high lateral resolution of this analyzing technique detected differences in the chemical composition of the very thin grain boundary film in comparison to the surrounding matrix grains.

Powder metallurgy

Main objective:

- Improvement of mechanical, thermal and chemical properties of semifinished and finished components by optimization of powder metallurgy techniques.

Specific objectives and selected examples of topics dealt with in the PM area:

- Development of new methods of powder synthesis and powder processing on laboratory and pilot scale, e.g., ultrasonic wave and liquid gas melt atomization,
- Fundamental findings on mechanical alloying for the production of metastable alloys,
- High strength and highly corrosion resistant Al alloys e.g. for structural components in aerospace engineering,
- Increase of maximum service temperature of Ti alloys to 600°C by dispersion strengthening,

Figure 5. Creep behavior of an annealed silicon nitride material without impurities (SN-5YB) and with Al and Al 4- Ca impurities in the liquid phase. The strong decrease of the creep resistivity with impurities demonstrate the control of creep by the grain boundary film chemistry.

- Techniques for the production of high strength austenitic steels and corrosion and wear resistant hard alloys with highest nitrogen contents,
- Development of a corrosion resistant ferritic oxide dispersion strengthenend (ODS) superalloy for applications up to 1350° C and tests of components,
- Sinter components with high porosity and asymmetric and gradient structure by means of centrifugal powder metallurgy methods,
- Powder metallurgy of refractory alloys,
- Injection moulding of high strength steels, corrosion resistant steels and hard metals,
- Explosive compaction of metal powders supported by computer simulation of the compaction mechanism,
- Development of nanometer scale material,
- Development of materials with (self-) adjustable properties under service conditions (intelligent materials),
- Development of longer life and more efficient cutting and shaping tools,

Figure 6. One set of Si₃N₄ inlet and outlet valves for an automotive combustion engine of Daimler-Benz (Mercedes 300E). Such valves have already been tested with great success in long term experiments within the engines. Significant savings of fuel consumption and weight as well as noise reductions were measured.

Lowering of friction and wear of components under increasingly aggressive environmental conditions (temperature, pressure, media).

Dispersion strengthening of AI-, Fe-, Ni-, Co-, Nb- and Cu-based alloys by oxides, carbides and borides of 10-50 nm diameter improves high temperature strength and corrosion resistance. Mechanical alloying of appropriate powders has progressed beyond the laboratory scale and is now being demonstrated on an industrial scale. Together with well adjusted thermomechanical treatment mechanical alloying is the most promissing technique to realize dispersion strenghtening. Noise damping and wear resistance by means of reinforcing particles are furthermore improved in Al-alloys. Appropriate combinations of carbides, nitrides, borides, oxides and multicomponent powders, as well as appropriate particle size and particle distribution are also important for improving cutting and shaping properties of tooling materials.

Special intermetallic compounds are successfully processed by powder metallurgy techniques.

The powder metallurgy route is one of the most promising methods for producing functionally gradient materials with properties/functions and chemical composition/microstructure varying continuously over the cross section of a component. R&D is on going in order to "bridge" smoothly different surface and volume properties and to avoid failure of the interface, typical for conventional multicomponent systems/composites.

As an example for the application in engineering industries a typical PM-project

"New oxide dispersion strengthened (ODS) superalloys for components exposed to hot gases"

is shortly presented. This joint project is based on a more fundamental joint project in the first phase of the Materials Research Program [10] and aims now at:

- Higher efficiency for energy conversion in combustion engines and turbines in order to save resources and reduce pollution.
- Improvement of PM-processing steps starting with the mechanical alloying and concentrating on the thermo-mechanical treatment (recristallization) with respect to texture and mechanical anisotropy for components in realistic dimensions.
- Improvement of joining and coating techniques.
- Improvement of component reliability in order to reduce safety risks e.g. in energy industry or aerospace transport.

This project is financially supported to 45% by BMFT and 55% by industry. The partners are Max-Planck-Institut für Metallforschung, Institut für Werkstoffwissenschaft, Stuttgart, PM-Hochtemperaturmetall GmbH, Daimler Benz AG, ASEA-Brown-Boveri AG and Metallgesellschaft AG.

Improvements in creep rupture strength of austenitic and ferritic ODS alloys are shown in figure 7 and compared with common materials.

Nozzles for dry-low-NO_x burners made of the newly developed ferritic ODS alloy (PM 2000) reached 12 times the service life of the conventional ones. Examples of components like rods for high temperature tensile testing equipment (PM 3030), moulds for the production of a washing machine sight glass (PM 2000), agitator for molten glass (PM 2000) and a gasturbine blade blank piece (PM 3030) are shown in figures 8-11.

Metallic high temperature materials and special materials

Main objective:

Development of metallic materials for use at higher temperatures and improved strength to specific weight ratio.

Specific objectives and selected examples of topics dealt with in the field of metallic high temperature materials and special materials:

- Development of powder metallurgy and ingot melting processing techniques for intermetallic compounds,
- Development of low pressure precision and sand casting methods for light metal alloys, as well as super plastic forming, diffusion, laser and electron beam welding,

Figure 7. Creep rupture strength of ODS-Alloys compared with common materials.

- Development of refractory nitrides, borides and carbon-nitrides for vacuum plasma spraying,
- Development of pressure electro-slag-refining (DESU) techniques for high temperature and corrosion resistant steels,
- Development of an uncoated superalloy for use up to 1100° C,

Figure 8. Rods for high temperatue tensile testing equipment. Material: PM 3030.

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Figure 9. Mould for the production of a washing machine sight glass. Material: PM 2000.

- Development of special coatings in order to improve resistance to corrosion, wear and crack initiation especially for components to be used at higher temperatures,
- Development of intermetallic compounds for use up to and above 1200°C,
- Manufacturing of single crystalline superalloy turbine blades,
- Development of new materials testing methods,
- Development of materials with special functional properties, such as permanent magnetic properties and shape memory properties.

In order to improve the efficiency of equipment in energy conversion processes, the operating temperature and pressure are constantly increasing. Thus creep and fatigue properties have to be further improved for conventional metals already operated at their highest service temperature, e.g., by means of direct and single crystalline solidification, dispersion strengthening and thermomechanical treatment.

Intermetallic compounds based on comprehensive fundamental R&D performed in the first phase of the Materials Research Program are now further developed with respect to specific requirement profiles especially from automotive engineering. Light metal alloys are further developed with respect to physical metallurgy as well as to joining techniques and casting techniques. Still higher temperature and lower weight are technical goals for Fe- and

Figure 10. Agitator for molten glass. Material: PM 2000.

Figure 11. Gas turbine blade (blank piece). Material: PM 3030.

Ni-base alloys. Life time prediction models are also improved. Special materials comprise among others hard magnets, e.g., rare earth/transition metal combinations, sometimes also intermetallics. R&D is generally directed to higher service temperatures.

Examples of the application of intermetallics in engineering industries and the aims of joints projects are summarized:

- Engine components for higher temperatures (\geq 1100 $^{\circ}$ C), based on alloys from the NiAl-(FeA1-) systems, without coating,
- Plates and foils for the use in aviation and space flight, $(*)$
- Wrought and precision cast turbine blades and vanes, $(*)$
- Precision cast, machined and wrought engine components (*) (*) Alloys based on the TiAl-system
- Light weight components for combustion engines by ingot melting, based on the MgSisystem.

There is a parallel activity on intermetallics by the DFG.

As an example of a development in the field of intermetallics, "TiA1 for jet engine applications" is shortly presented [11]. This project is also based on a more fundamental one and is now financially supported to 50% by BMFT and industry. The partners are GKSS-Forschungszentrum Geesthacht GmbH, Forschungszentrum Jtilich GmbH KFA, MTU Motoren-und Turbinenunion Mtincben GmbH, ASEA-Brown-Boveri AG, B6hler AG und Titan-Aluminium-FeinguB GmbH.

Advanced jet engines with high operating efficiency demand new classes of low density and high temperature resistant materials. To meet these demands titanium aluminides are being developed to replace high density Ni-base superalloys. TiA1 has higher oxidation and corrosion resistance than most titanium alloys. Ingot metallurgy routes are feasible for TiA1 turbine component processing. Blade and casing prototypes have been produced successfully by investment casting. Although considerable progress has been made, adequate quality at acceptable cost has still to be realized.

TiA1 has higher weight-specific stiffness than titanium and Ni-base superalloys. TiA1 is fire-resistant under the temperature and pressure conditions of high pressure compressors. The mechanical properties of TiA1 materials achievable today are summarized below (see also Fig. 12): weight specific yield strength, creep and fatigue resistance below 700° C are relatively high. Elongation is in the range of 1-2, 5% at room temperature. Fracture toughness and notched impact resistance are poor. Special design rules and lifing concepts as well as proper quality control need to be developed to overcome poor damage tolerance. MTU has recently conducted a spin test of a TiA1 turbine blade. The blade was made by investment casting and grinding. The blades were tested at 700° C and 16.000 rpm in a hot gas stream and exhibited no detectable damage. The application of TiA1 for high pressure compressors and low pressure turbine components will be made possible by advanced design techniques and future testing experiences.

Figure 12.

New polymers

Main objective:

- Development and demonstration of polymer high performance materials with superior or completely new physical, chemical and technical properties or combination of properties.

Specific objectives and selected examples of topics, dealt with in the polymer area:

- Development of new processes for thermoplastic aromatic polycarbonate with glass transition temperature up to 190° C.
- Toughening of duromeres by introducing liquid crystal moieties.
- Development of liquid crystal polymers suitable for advanced processing techniques.
- Development of heat-resistant polymers.
- Synthesis of stable polymers with high electrical conductivity.
- Development of low loss polymeric optical fibers $(< 100 \text{ dB/km})$.
- Optimization of production; characterization and search for applications of ultra thin layers.
- Development of new polymeric electroluminescent materials.
- Development of a new system with best nonlinear optical properties.
- Development of high resolution (down to $0.2 \mu m$) photo-resists.
- Development of new dye containing liquid crystal polymers for optical information storage and optical holography as well as polarization holography.

Successful R&D on polymers in recent years have been mainly based on fundamental results from chemistry and physics with respect to molecular structural, molecular interaction and molecular architecture of polymers, and have employed computer modelling methods for molecular design.

The Project

"Ultra thin polymer layers (UTPL) as basis for new optically, electronically and biologically effective systems"

is summarized referring to application in engineering industries [12]. This joint project is also based upon a more fundamental joint project completed in the first phase of the Materials Research Program. The new project is funded to 50 % by BMFT and industry. The partners are Max-Planck-Institut ftir Polymerforschung, Mainz, Max-Planck-Institut für Biophysikalische Chemie, Göttingen, Universität Mainz, BASF AG, Bayer AG, Hoechst AG und Siemens AG.

The more fundamental topics deal with supramolecular structure including synthesis, concepts and materials science and combines new polymers with new properties:

- *Molecules showing self organization*
- *Techniques to manipulate molecules and*
- *Unconventional properties like*
	- *9 electronic/ionic conductivity,*
	- *9 optical properties and*
	- *recognition phenomena/sensor.*

The more advanced and more applied topics deal with the following topics;

- Optimization of UTPL's and development of selective ion- and gas sensors as well as surfaces with improved tribological properties based on the new UTPL's.

- Optimization of Langmuir-Blodgett-Layers (LBL) and development of an optical biosensor based on the new LBL's.
- Development of physico-chemical characterization of molecular mono- and multilayers for applications primarily in the field of non-linear optics and
- Assessment of sensor properties and of non-linear optical behaviour of UTPL's.

Composites

Main objective:

Development and demonstration of composites with superior properties or combination of properties compared with those of monolithic materials. Special care is taken for processing techniques and manufacturing costs.

Specific objectives and selected examples of topics, dealt with in the area of composites are given below;

- Processing of Al_2O_3 filament yarns and fibres.
- Remarkable improvement of a carbon fibre reinforced thermoplast composite by appropriate fibre dressing.
- Improvement of high temperature strength of AI- and Mg-alloys by fibre reinforcement.
- Improvement of high temperature oxidation resistance of carbon fibre reinforced carbon.
- Development of fibre reinforced cordierite and SiC with high defect tolerance.
- Processing of high purity powders and improvement of product uniformity by clean room conditions.
- Development of suitable nondestructive testing methods, especially for interface studies.

Layer, fibre and particle reinforced composites are successfully used in various applications. Many materials may be combined in very different ways as matrix as well as reinforcing materials: glass, metal, ceramics, carbon and polymers. This focal point thus deals with their combinations and has strong connections with the four focal points mentioned before.

Physical bonding of conventional polymer composites could be improved by chemical bonding if the reinforcing materials could be introduced on a molecular basis before or during polymerisation. Higher temperature resistance could be achieved by using thermoplastics.

Metal matrix composites are to be improved with respect to high temperature strength and fatigue properties by means of (coated) reinforcing materials. Ceramic matrix composites are mainly designed to "bridge" the relatively low fracture toughness of monolithic ceramics.

Joining, machinability and recycling of (new) composites are requirements which pose special technical problems and have also to be taken into account with respect to economical aspects.

There is a shift in funding intensity of highly innovative R&D from polymer matrix composites to metal and ceramics matrix composites. In parallel technology transfer in the field of polymer matrix composites has been supported since 1988 by means of demonstration centers for fibre reinforced plastics.

As an example for applications in engineering industries the aims of a typical metal matrix composite (MMC) joint project

"Development and optimization of fibre reinforced Al-alloy-parts manufactured by squeeze casting"

is briefly described [13]. This project was performed in the frame work of the European Cooperation in the Field of Scientific and Technical Research (COST, see below): Casting and solidification technology (COST 504). The German part of this project was financially supported to 50% by BMFT and industry. The German partners were Fraunhofer-Institut, Labor für Betriebsfestigkeit Darmstadt, Kolbenschmidt AG and Didier-Werke AG. The Austrian partner BMW-Motoren GmbH, Steyr, was responsible for the piston design and the component testing as well as for their financing. The financing of COST R&D is up to the participating countries.

The state of the art of MMC-development based on this COST-project is summarized as follows:

Fibre preforms with high and reproducible quality were developed in order to meet the necessary requirements for further processing (squeeze casting) and composite component behaviour. Squeeze casting, machining and testing of locally fibre reinforced Al-alloys were developed and demonstrated in diesel engine pistons (s. Figure 13, 14). Characteristic materials data were determined for component design and life time prediction. Nondestructive testing methods were developed and engine tests were performed by the engine producer.

4. Materials development in other programs

High performance materials play a crucial role in improving existing technologies and in implementing new technologies. Thus it appears "natural" that there is a substantial amount of materials development going on in other technology programs. The distinction between materials research and materials development/processing is somewhat arbitrary and is only used here for the sake of simplicity.

Energy

Extensive work is performed to develop and apply high performance materials under the Program on Energy Research and Technology. A few examples will be given here. Much of the work is focussed on the economic conversion and storage of energy, and include for example the development of new burner systems, exhaust sensors, heat exchangers, the Sodium-Sulfur storage battery, fuel cells, the hydrogen technology, which involve mainly SiC, Al_2O_3 and ZrO_2 . Other work is dealing with combined cycle power engineering which involves research on new generation gas and steam turbines, coal gasification, pressurized

Figure 13. D1-Piston with fiber reinforced combustion bowl edge.

fluidized bed combustion, and involves mainly Fe- and Ni-base-superalloys for near term in the applications and $Si₃N₄$ and SiC for future applications in blading and for thermal barriers respectively, in gas turbines. Concerning new energy sources e.g. a SiC alternative to the metallic solar thermal central receiver system has been tested (gas cooled solar tower power plant, GAST).

Figure 14. Thermal fatigue resistance. Comparison reinforced/unreinforced.

Aerospace technologies

Materials development concentrates on low specific weight, high specific strength (at high temperature), improved reliability, oxidation and corrosion resistance etc. Thus carbon and polymer composites, light metal alloys and composites are favored materials for structural components.

Information technology

There is a focal point Micro/Peripherials within the concept of the Federal Government to support the development of microelectronic, information and communication technologies. Within this focal point special emphasis is laid on sensors and power electronics. Semiconductors as well as functional metals and polymers are involved.

Environmental technology

Within the Program on Environmental Research and Technology the BMFT supports the development and testing of new materials and processing methods in order to reduce exhaust emissions of traditional and advanced technologies.

Physical technologies

There is a focal point on surface technologies e.g. laser-, plasma-, ion-based methods, concerning thin films and thick film applications, including high temperature superconductors.

Health

Since 1973 the development and clinical testing of alumina-ceramics for artificial joint systems has been supported by the BMFT under a program entitled "R&D in the Service of Health." The good abrasive wear resistance and inertness of α Al₂O₃ has improved the life time of artificial limbs. R&D is still concentrating on a better understanding of the interaction between the ceramic surface and the living bone tissue. However, most efforts are now being directed towards improving long term stability, bioactivity and resorption of the implants (ceramics, glass ceramics, glasses, polymers, metals and composites).

Besides the Program on Materials Research of the BMFT there are materials R&D programs in some States (e.g. Bavaria and B aden-Wtirttemberg) which also include cooperation between science and industry. Furthermore there are application oriented development programs which involve materials science and engineering supported by the Federal Minister of Defense and Economics as well as by each State.

As an example, the "Arbeitsgemeinschaft Industrieller Forschungsvereinigungen 'Otto von Guericke' e. V." (AIF) supported by the Federal Minister of Economy (BMWi) is described. R&D of relevance for industrial branches are performed in institutes with special know how and managed by a scientific and industrial board of trustees. The respective industrial branch must spend at least the same amount of money (as the BMWi) for jointly shared R&D. 3-5 Mio. DM per year go into the area of materials.

5. International activities

There are four multinational activities in the field of high performance materials which are supported by the Program on Materials Research and worth mentioning in this context. These are COST, EUREKA, IEA and VAMAS (see also [6]).

COST

COST stands for European Cooperation in the Field of Scientific and Technical Research, and it includes the EC and EFTA member states together with Czech Republic, Hungary, Kroatia, Poland, Slovac Republic, Slovenia, Turkey. Technical research in the field of materials in the framework of COST dates back to 1971. As in the forementioned Program on Materials Research, engineers and scientists cooperate on a work sharing basis, but the cooperation is here however, on a European level. Participation in COST actions is up to the COST member states (fi la carte). Financial support is generally provided by the COST member state and no central funding is involved.

The current activities with German participation comprise:

- 501 Advanced materials for power engineering components,
- 503 Powder metallurgy,
- 504 Casting and solidification technology,
- 507 Thermodynamic data for new light metal alloys,
- 509 Corrosion and protection of metals in contact with concrete,
- 510 Advanced materials for temperatures above 1500° C-Development of testing methods,
- 511 Interaction of microbial systems with industrial materials,
- 512 Modelling in materials science and processing (MMSP),
- 513 Improvements in availability and quality of intermetallic materials,
- 514 Ferroelectric ceramic thin films,
- 515 Plasma and ion surface engineering (PISE) techniques for materials.

Two further projects are in preparation, and they are;

- 518 Molecular materials and functional polymers for advanced devices,
- 519 Modification of glass surfaces.

COST Materials Actions have a long tradition of high level European scientific and industrial cooperation. So far this holds true especially for metallic structural materials with specific strength at high temperatures and with low specific weight. Thus their main contribution was to energy, environment and transportation technologies (COST 501-509). The dialogue with industrial and scientific representatives on possible new COST Materials Actions revealed—besides further ongoing R&D needs in those technologies—increasing interest in European R&D efforts in information and communication techniques as well as bio- and medical techniques. At the same time R&D on non-metallic high performance materials like ceramics/glasses and polymers had to be included. Thus all classes of high performance materials are now dealt with within COST Materials Actions. They all serve to promote key technologies that are important for the competitiveness of European industry (COST 510-519) and sometimes are in competition with each other.

The advantages of opening COST to participation from non-COST-States, in particular from other European States on a case by case basis where there is a clear scientific justification and where the benefits are mutual, is worthwhile mentioning. For example, in 507 the Baikow Institute of Metallurgy in Moscow participates, and provides enormous know how on Al-multicomponent alloys.

EUREKA

EUREKA is another important European initiative in the field of applied research and development and was started in 1985. It also includes EC and EFTA member states. The financial support comes from the member states with no central funding, and it covers also "new materials." Thus it is very similar to COST. In contrast to COST, there are separate joint international projects and no concerted actions devoted to specific materials technologies comprising several international joint projects under one technical leadership. Germany participates in 23 joint projects in the technological area "new materials". Including materials engineering this area displays the highest number of projects among all other technological areas in EUREKA.

IEA

In 1979 the United States of America and the Federal Republic of Germany signed an International Energy Agency (IEA) Implementing Agreement for a "Program of Research and Development on High Temperature Materials for Automotive Engines". This cooperation is dedicated to prenormative R&D in the field of high performance ceramics. It includes various annexes which are listed below;

Technical Annex I: "Ceramics for Automotive Gas Turbine Engines" FRG, USA 1979- 1984

Technical Annex II: "Cooperative Program on Ceramics for Advanced Engines and Other Conservation Applications"

FRG, Sweden, USA 1985-1989, Subtask 1 Information Exchange, Subtask 2 Powder Characterization, Subtask 3 Ceramic Characterization, Subtask 4 Mechanical Properties.

IEA Amendment to the Technical Annex II. FRG, Japan, Sweden, USA 1990-1993, Subtask 5 Mechanical Characterization of Structural Ceramics, Subtask 6 Characterization of Ceramic Powders, Subtask 7 Machining of Ceramics, Subtask 8 Characterization of Ceramic Powders, Secondary Properties.

VAMAS

High performance materials play also an important role in the Versailles Project on Advanced Materials and Standards (VAMAS). Seven countries (CAN, D, F, I, Japan, UK, USA) and the CEC participate in VAMAS.

VAMAS as well as the IEA cooperation comprises the further development and international comparison of characterization and testing methods and round robin testsprestandardization R&D, similar to the IEA topics, however, covering mainly non ceramic materials.

CEC program Brite/EuRAM

The funding by the European Communities (EC) plays an important role in the area of materials technology in Europe. The Commission of the European Communities (CEC), which is the EC executive body has initiated the COST activity, participates in COST also with its Joint Research Centers. It supports financially the administrative work and studies (not R & D) of the COST Management Committees, and participates also in EUREKA and VAMAS as a member. Key efforts of the EC in this field present the R&D programs running since 1985 within its framework programs for research and technology. The most important one with respect to materials is the current program "Industrial and Materials Technologies" (1991-1994), called also Brite/EuRam II (Basic Research in Industrial Technologies for Europe/European Research on Advanced Materials). Brite/EuRam II with a budget of about 1.2 Billion DM of which about 50% are foreseen for materials research including raw materials and recycling comprises

- industrial research (77% of the total budget)
- focussed fundamental research (10%)
- cooperation research for small and medium enterprises (9%)
- feasibility studies (1%)
- concerted actions (1%) and
- specific training (2%).

The technical research areas enclose

Area 1—Materials/Raw Materials

Sub-areas: Raw materials; recycling; structural materials; functional materials for magnetic, superconducting, optical, electrical and biomaterial applications; mass commodity materials;

Area 2—Design and Manufacturing

Sub-areas: Design of products and processes; manufacturing; engineering and management strategies for the whole product life cycle.

Area 3-Aeronautics Research

Like COST, Brite/EuRam also deals with precompetitive work sharing European R&D. Participants from EFTA can submit cooperative project proposals together with at least two other proposers from EC-countries. They cannot act as project coordinator and don't receive any funding from the Community. From 1994 on the EFTA-participants (except Switzerland) should be fully integrated with equal rights as EC-participants. Partners from Central and Eastern European Countries can participate, subject to the prior consent of the Commission, in the projects as subcontractors on a project by project basis.

"Concerted Actions" in these CEC programs differ slightly from concerted actions ~i la COST. The common feature is the coordination of several European R&D activities related to an embracing materials topic. In COST the single R&D projects are evaluated and monitored by the COST Management Committee (and paid by the project partners and their national government). In Brite/EuRam the single R&D projects/activities are selected, monitored and financed outside the "Concerted Actions." However, the CEC pays for the organization/performance of the "Concerted Action" i.e. meetings of all size, exchanges/visits to other participating establishments, preparation and distribution of materials used for round robin test and dissemination of information etc.

The program will be most probably continued in a similar manner also beyond 1994 within the 4th-FTE-Framework Program (1994-1998). The call for proposals for the next round will be announced sometime after October 1994. For further details the Commission of the European Communities, DG XII-C, Brite-EuRam Program, Rue Montoyer 75, B-1049 Brussels or the National Contact Points should be addressed.

The scientific community and industry of Germany are very active in Brite/EuRam. More than 20% of the CEC budget goes to Germany (corresponding to approximately ca. 80 Mio. DM/per year). Thus a considerable amount of materials R&D in Germany is supported by the CEC. On one hand this means an international acknowledgment of the high level R&D in Germany and on the other hand it opens chances for European cooperation on the basis of mutual benefit. The prerequisites for all such sorts of international cooperation are the national R&D programs. However, they also profit from the scientific and technical competition and results of international cooperation.

Conclusion

Materials research and development are prerequisites for improving existing technologies and implementing new technologies. Science and industry in Germany agree to this view and cooperate on a work sharing basis financially supported by industry, Federal and State Government Agencies. If there is mutual benefit there are also bilateral and multilateral cooperations.

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