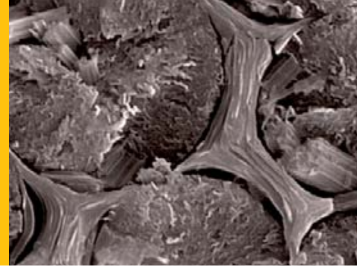
ExtreMat

New Materials for Extreme Environments



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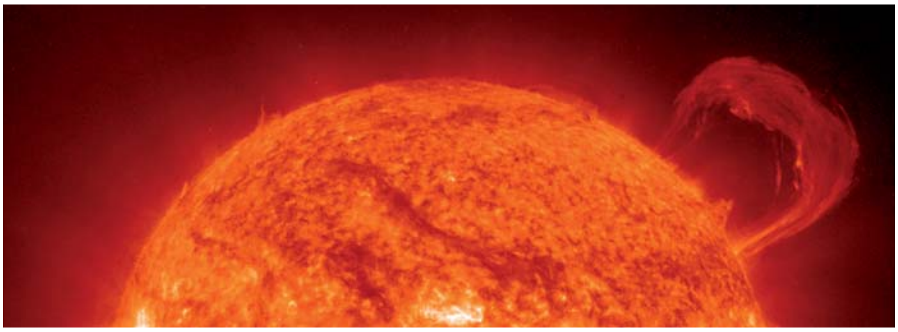
Five Years at the Front of European Materials Research

In 2004 the European Commission decided to fund one of the most ambitious materials research projects ever: The FP6 Integrated Project "ExtreMat – New Materials for Extreme Environments". Five years later the project is approaching its end - time to have a look back at the research work done and the results achieved so far.



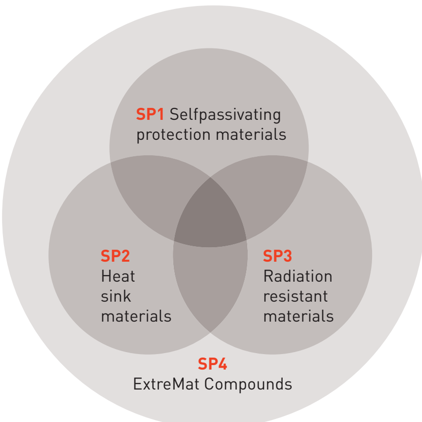
Taming the power of the sun in fusion reactors requires materials that withstand extreme conditions (picture: SOHO, ESA&NASA)

Hotter, faster, smaller – many industrial products and systems expand towards regimes where temperatures and heat loads on components become destructive. Especially the combination with additional load factors like rapid fluctuations, thermo-mechanical stresses, aggressive chemical substances or irradiation, brings conventional materials to their limits. Some have excellent functionalities but cannot withstand the extreme environments, others that withstand the extreme conditions cannot fulfil the required functions.

Extremely challenging requirements for materials occur in areas as different as e.g. power generation, aerospace, opto- or power-electronics. Widening the operational window of existing technical systems, e.g. towards higher efficiency or higher packaging density, as well as creating completely new top-end systems like fusion reactors, requires real breakthroughs in materials technology. These are beyond reach with incremental materials development. To make a leap forward on this way was the goal of the ExtreMat project.

Starting point of ExtreMat was the fact that underlying basic questions of materials behaviour under extreme loading conditions were similar in different sectors and industries, while the materials research was still rather fractured. With the ExtreMat project, the industrial and scientific expertise, methods, tools, laboratories and test facilities of different sectors were combined in a single 'Integrated Project'. This large 'critical mass' was the key factor for the research work of ExtreMat. However, at the same time it was also a big challenge from an organisational point of view. In this respect, ExtreMat was an experiment in itself.

Research work in ExtreMat focused on four main topics, organised as interacting sub-projects:



Each sub-project had its own coordinator, project meetings etc, but overlapped with respect to project partners and interacted by the exchange of materials and results. The total project was organised in four phases.

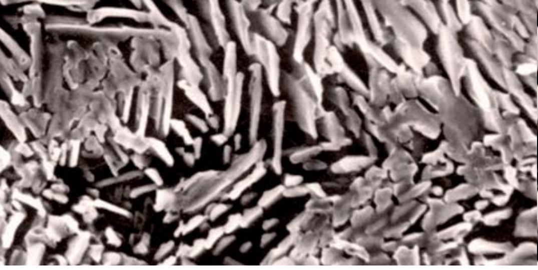
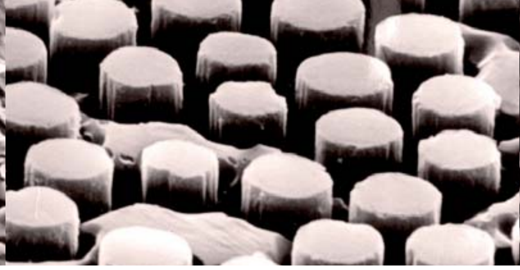
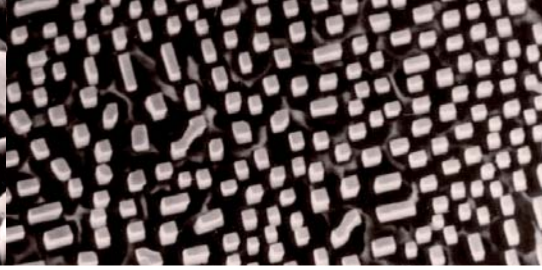
During an initial **Requirement Definition Phase**, 32 User Requirement Specifications for specific applications were defined and evaluated by a Scientific Industrial Committee (SIC). Those user requirements were then transformed into 15 Materials Requirements Specifications (MRS).

In the **Concept Definition Phase**, a comprehensive list of alternative research and development concepts were generated by a broad experimental and theoretical screening regarding possible solutions for the MRS. Evaluation by the SIC resulted in a selection of the most promising ones with lowest risk and highest chance of success, as a starting point for the research work.

During the **Research and Development Phase** the selected R&D concepts were implemented and investigated in parallel. With continuing research effort and accumulated new results, the concepts were gradually channelled down into finally 20 Materials Industrialisation Concepts (MIC). In addition, a long-term irradiation campaign was started to prove the radiation resistance of those new materials intended for use in nuclear environments.

The final **Industrialisation Phase** was dedicated to the investigation of the up-scaling potential of the new materials and compound technologies with respect to industry-like manufacturing. The major results and findings of the whole process are described on the following pages.

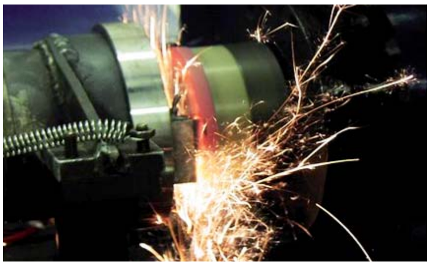
Uwe Schüssler,
Bayern Innovativ,
Nuremberg, Germany,
Disseminaton Management

Sub-project 1: Self-passivating Protection Materials

The motivation of Sub-project 1 (Self-passivating Protection Materials) was to merge the expertise dispersed in different fields of applications in which protection materials are required to withstand extreme environments (aerospace, fusion, fission, turbine engines and brakes). The idea was to identify common scenarios under extreme environment such as high thermal and mechanical loads, operation under oxidizing/corrosive atmosphere at high temperature, high thermal shocks etc to pursue a breakthrough materials development, and to share application oriented test facilities, which can only be available in the frame of such an integrated project.

Carmen García-Rosales
CEIT, San Sebastián, Spain
Coordinator of sub-project 1



Friction and wear tests of short fibre C_f/SiC for high power brake applications (picture: MERL)

C-based plasma-facing materials

New Ti-doped isotropic graphites were developed for fusion application with thermal conductivity ≥ 220 W m^{-1} K^{-1}, flexural strength ~100 MPa and chemical erosion by hydrogen bombardment reduced by a factor 4 to 10.

Actively cooled plasma-facing mock-ups prepared with these materials were tested under high heat fluxes, surviving the targeted load of 100 cycles at 20 MW/m^2. Furthermore, Ti-doped C/C composites with through thickness thermal conductivity ~ 290 W m^{-1} K^{-1} and reduced chemical erosion were produced; preliminary high heat flux tests up to 20 MW/m^2 on mock-ups with these doped C/C showed significant improvement compared to undoped C/C. In addition, a method to increase the overall Ti content up to ~ 2 at. % was developed.

Assembled C/SiC bearing with SiC-based CVD lubricant coating for space vehicles (picture: MT Aerospace)

> Looking back on the last 4 years, I consider a great enrichment to have been involved in the ExtreMat project as SP1 Coordinator. It has been a challenge to coordinate such a large sub-project (21 institutions are involved in SP1), but the enthusiastic commitment of all partners made this job easy, and their intensive work and the exchange of a large number of materials samples and tests results led to the excellent results obtained within SP1 and the whole project.
>
> Carmen García-Rosales

Oxidation-resistant materials

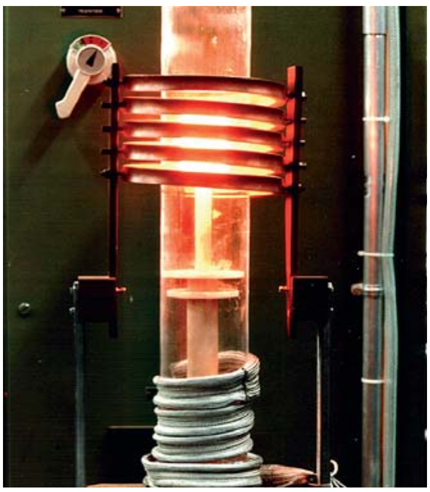
New C/SiC/B_4C composites with high oxidation resistance were developed for space applications. No mass change was observed after 10 oxidation tests of 1 h in air at 1400°C due to the formation of a thin borosilicate layer. Re-entry tests showed that burn-off starts at ~ 1400°C, and at 1500°C the lifetime of the material is ~10 cycles. Coating with HfO_2 resulted in a significant improvement: no mass loss at 1400°C, at 1500°C the mass loss is below 5% after 30 cycles.

Thermal protection shields

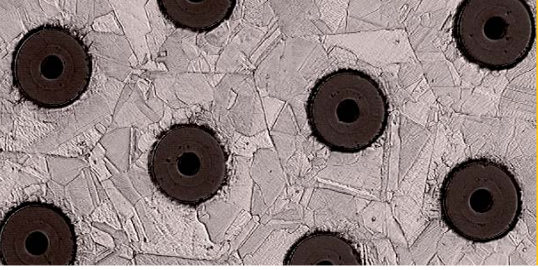
SiC-based multilayers developed within SP1 fulfil all requirements for application as thermal protection systems in space vehicles. After exposure to air at 1600°C for 30 h they retained 100% of their strength, and survived 100 re-entry tests at 1500°C. The introduction of a porous layer resulted in a reduction of the through thickness thermal conductivity by maintaining the good mechanical properties. Coating with Y_2O_3-SiO_2 provides an effective protection against high temperature oxidation/corrosion under O_2/H_2O atmosphere. The industrialization of this material is almost concluded: the first 4 steps (slurry, tape casting, stacking, de-binding) were successfully transferred, and the sintering step is in progress.

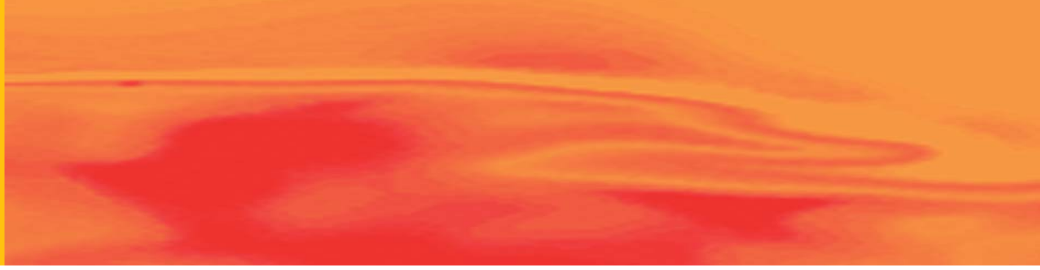
High temperature lubricants

CVD-based high temperature SiC lubricant coatings on CMC bearings for space vehicles were developed, optimized and tested under re-entry conditions, surviving 27 missions at 1550 °C with > 22 000 movements under 7.3 kN load. These results are the basis for a currently running, very promising re-entry space vehicle development. The industrialization and reproducibility of the coating procedure was successfully demonstrated.



Re-entry tests of new heat shield materials for future European space shuttles (picture: FZJ)

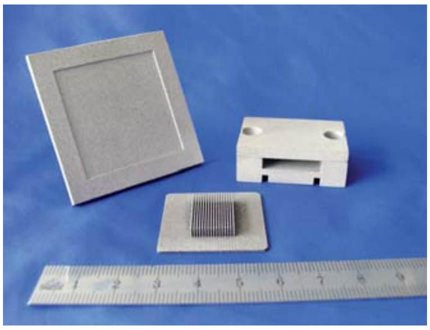




Sub-project 2:
Heat Sink Materials

The main motivation of sub-project 2 is to develop novel composites for heat sinks in advanced applications such as high temperature reactors or modern electronic devices. These materials are expected to remove excessive heat generated during long-term performance, withstand large temperature changes without disintegration and deterioration of properties and reduce complex thermo-mechanical stresses after bonding to supporting or protecting structures by tailoring their thermal expansion. The research effort was oriented towards two basic types of heat sinks:

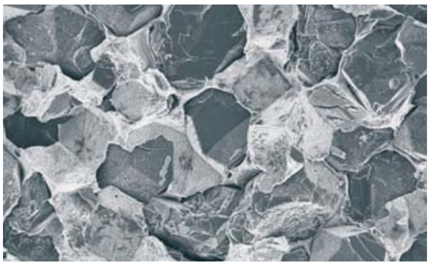
František Simančík
IMSAS, Bratislava, Slovakia
Coordinator of sub-project 2



Examples for heat sinks and housings made of silver-diamond composites (picture: EPFL)

- → composites based on a copper matrix reinforced with continuous SiC, carbon or refractory fibres for use at temperatures up to 1000 °C in applications where excellent dimensional stability, combined with good thermal conductivity, play a primary role and
- → composites based on extremely conductive phases like diamonds, graphite flakes or pitch-based carbon fibres embedded in an appropriate metallic matrix for use in applications, where extreme heat conductivities, combined with tailored thermal expansion, are the main requirements

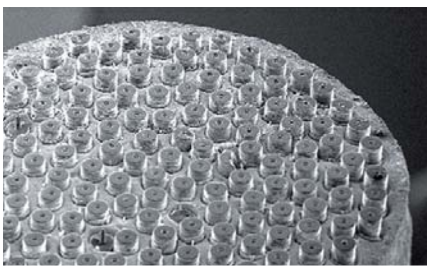
Several unique materials have been developed within ExtreMat so far, including silver-based diamond composites with extreme thermal conductivity exceeding magical 900 W m^{-1} K^{-1}, thermally stable tungsten wire reinforced copper with „isotropic" thermal expansion, which is repeatedly able to withstand heat fluxes higher than 20 MW/m^2, as well as low-cost aluminium matrix / graphite flake composites with excellent machinability for manufacturing of complex shape components.



Copper-diamond composite as heat sink material (picture: Plansee)

Advanced technological methods comprising gas pressure infiltration of melts, squeeze casting, isostatic pressing, spark plasma sintering, etc. were worked on for manufacturing of these special composites. The development was supported by tailoring of interfaces between constituents at nanoscopic level in order to stabilise them in the whole range of working temperatures without degradation of thermal conductivity. Novel non-destructive advanced characterisation techniques, including synchrotron tomography, neutron diffraction or thermal mapping, were proposed and utilised to study the stability of composites under extreme loading conditions. The experimental results helped to suggest new realistic models for further material optimization and component design.

> „ The obtained results give very promising prospects not only for fields targeted within the ExtreMat project but also for many other applications where thermal management is of highest importance.
>
> František Simančík



Cu-SiC composite made by PVD coating of SiC fibres with copper and hot isostatic compaction (picture: IPP, DLR)

The improved performance of matured materials has been successfully verified in testing of various prototype heat sinks such as the cooling plate for IGBT power electronics modules, the protection wall for fusion reactors, the housings of electronic packages or of laser slab crystals.

ExtreMat Project Consortiu

Universities – Ecole Polytechnique Fédérale de
Warszawska, Poland • Politecnico di Torino, It
sity of Oxford, United Kingdom • Technische U

Research Institutes – Centro de Estudios e
Spain • Consejo Superior de Investigaciones
CAR), Spain • Fraunhofer-Institut für Fertigung
Pulvermetallurgie und Verbundwerkstoffe, Ge
Swiss Federal Laboratories for Materials Test
plazmatu AVCR, Czech Republic • Ústav ma
Slovakia

Research Centers – AIT Austrian Institute of
l'énergie atomique, France • Deutsches Zent
European Commission, DG JRC Institute for E
Jülich GmbH, Germany • Max-Planck-Institut
tre for Scientific Research "Demokritos", Gre
v.o.f., The Netherlands • Paul Scherrer Institu
ergy Authority, United Kingdom

Industry – Ansaldo Energia S.p.A., Italy • Ar
NP SAS, France • AREVA NP GmbH, German
Deutschland GmbH, Germany • Empresario
S.p.A. Nuove Tecnologiee Servizi Avanzati, Ita
Engineering Research Laboratory Ltd, United
ited, United Kingdom • Plansee SE, Austria •
Germany

Sub-project 3: Radiation Resistant Materials

The overall objective of this sub-project is to develop radiation resistant, high-temperature, low activation materials for structural and protective applications. Activities are being focused on oxide dispersion strengthened ferritic steels and tungsten-base materials. The neutron irradiation resistance of the most promising materials developed within SP1, SP2 and SP3, and of small mock-ups produced within SP4 is also being investigated within this sub-project.

Nadine Baluc
EPFL, Villingen PSI, Switzerland
Coordinator of sub-project 3

im

e Lausanne (EPFL), Switzerland • Politechnika
taly • Universidad de Alicante, Spain • Univer-
Universität Wien, Austria

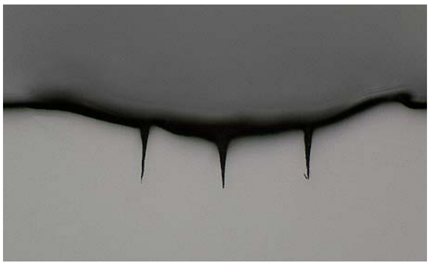
Investigaciones Técnicas de Gipuzkoa (CEIT),
Científicas Instituto Nacional del Carbón (IN-
gstechnik und Angewandte Materialforschung,
ermany • Fundacion Inasmet, Spain • Empa,
ting and Research, Switzerland • Ustav fyziky
teriálov a mechaniky strojov SAV, Bratislava,

Technology GmbH, Austria • Commissariat a
rum für Luft- und Raumfahrt e.V., Germany •
nergy, The Netherlands • Forschungszentrum
für Plasmaphysik, Germany • National Cen-
ece • Nuclear Research & Consultancy Group
ut, Switzerland • United Kingdom Atomic En-

rcher Technicoat Ltd., Great Britain • AREVA
y • Bayern Innovativ GmbH, Germany • EADS
s Agrupados Internacional, S.A., Spain • FN
aly • MT Aerospace AG, Germany • Materials
Kingdom • National Nuclear Corporation Lim-
SGL Carbon GmbH, Germany • Siemens AG,

New steels for nuclear applications

Recent activities have been focused on the manufacturing of improved oxide dispersion strengthened (ODS) steels. In particular, it was attempted to improve the microstructure and mechanical properties of the Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 ODS ferritic steel by applying thermo-mechanical treatments (TMTs), such as hot pressing or hot rolling, and heat treatment. It was found that the microstructure is significantly improved, i.e. homogenized, by the TMTs, as well as the Charpy impact properties. The best ductile-to-brittle transition temperature (DBTT) of about 10°C was obtained for the ODS ferritic steel compacted by hot extrusion at 1100°C and submitted to hot rolling at 700°C and heat treatment at 1050°C for 1 hour.



Quantitative analysis of plastic zones around crack-tips in tungsten (picture: UOXFORD)

Understanding material failure

A comprehensive model that explains the anomalous radiation damage effects occurring in iron and steels at elevated temperatures was developed. This model shows that the anomalous generation of <100>-type dislocation loops observed in iron, iron-based alloys and steels at elevated temperatures is related to the loss of strength of steels observed in the same temperature range.

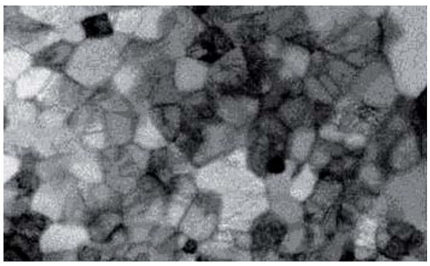
Miniaturization of materials testing

In the field of small specimen test technology, micropillars oriented along the <111> direction have been prepared from the annealed PM2000 ODS ferritic steel. A very good agreement between the yield stresses measured from compression tests on these micropillars and tensile tests on standard specimens was found. Size effects cannot be excluded for other materials, however, but even if present they can be calibrated and the results can be used for damage assessments.

Neutron irradiation tests

Two neutron irradiation campaigns in the High Flux Reactor (HFR) in Petten have been started, one at low temperatures (300°C, 550°C) and low doses (0.7-1.4 dpa in stainless steels) and another one at high temperatures (600°C, 900°C) and large doses (3-5 dpa in stainless steels).

Each irradiation matrix contains about 450 specimens of the most promising materials developed within ExtreMat.



Microstructure of a Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 ODS ferritic steel prepared by powder metallurgy techniques (picture: EPFL)

Both irradiation campaigns should be achieved in 2009 and post-irradiation examination of the specimens should take place in 2010.

> Very promising oxide dispersion strengthened ferritic steels have been developed within this sub-project. I see potential applications in advanced nuclear installations (advanced fission reactors, thermonuclear fusion reactors, spallation neutron sources, accelerator driven systems) as well as in non-nuclear installations (solar thermal reactors, solar chemical rectors, gas turbines, coal gasification systems).
>
> Nadine Baluc

Sub-project 4: ExtreMat Compounds

The fourth subproject has a central function in making ExtreMat a true "Integrated" Project, thanks to its prevalent orientation towards practical industrial applications of new materials: this is being realized through the preparation of ExtreMat compounds, incorporating different new materials and interfaces; joining itself is done through newly developed concepts, and testing in relevant conditions completes the logical process.

Carlo Gualco
AEN Genova, Italy
Coordinator of
sub-project 4

> Producing compounds with the new materials, providing tailored interface technologies and offering industrially relevant testing capabilities, significantly contributed to the demonstration of practical exploitability of the new ExtreMat materials. Excellent commitment from other subprojects ensured continuous and timely flow of materials and information. I am personally grateful to all partners for the willingness to cooperate that I always found during so many direct contacts.
>
> Carlo Gualco

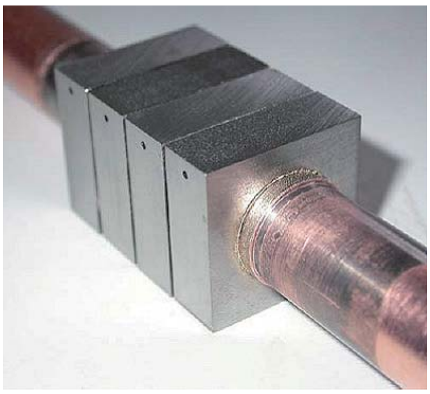
Joining, stress and interlayers

Extensive FEM calculations confirmed that low CTE stiff interlayers being developed within this project can reduce residual stresses deriving from high temperature brazing of protective materials to heat sinks in nuclear fusion applications.

This joining concept, after preliminary successful demonstration on state-of-the-art materials with simple molybdenum interlayers, allowed the production of actively cooled plasma facing modules, including new Ti-doped graphites and carbon fibre composites as well as W-fibres/Cu and SiC-fibres/Cu metal matrix composites interlayers from sub-projects 1 and 2, respectively. New films acting as wetting promoters for brazing alloys (based on TiC_x, TiN_x and Si_3N_4 compositions with optimized stoichiometry) have been deposited on CFC tiles to be joined.

Monoblock manufacturing

Actively cooled modules with so-called monoblock geometry (tubular heat sink passing through protective tiles) have been prepared by gas pressure infiltration of molten copper in tungsten fibres preforms and simultaneous joining to W or CFC tiles; state-of-the-art CuCrZr alloy heat sinks are completely replaced, opening the way for higher temperature applications, as expected in future nuclear fusion power plants (technology beyond ITER).



Tungsten wire reinforced copper tube, produced and joined to tungsten monoblock tiles in a single gas-pressure infiltration process (picture: IMSAS)

Extensive testing

High heat flux tests performed in powerful facilities, like the electron beam JUDITH and the ion beam GLADIS, finally underlined the success of ExtreMat materials and joining concepts: most tested modules survived 100 thermal cycles at 15 MW/m² whereas some of them showed no significant degradation after 100 thermal cycles at 20 MW/m², that favourably compares with state of the art materials and technology.

Heat flux test of plasma facing compounds for fusion reactors in the Garching LArge DIvertor Sample test facility GLADIS (picture:IPP)

New tritium diffusion barriers

Chemical vapour deposition of erbium oxide allowed the production of barriers against tritium diffusion for nuclear fusion applications, able to reduce permeation through Eurofer low activation steel of a factor 10^3 to 10^4. The targeted application is for components of the breeder blanket modules of future nuclear fusion power plants, i.e. in the parts of the reactor where tritium fuel will be produced from lithium under neutron irradiation.

Brazing thermal protection shields

Surface modification with chromium of C_f/C and C_f/SiC composites allowed successful brazing to nickel based alloys, aiming at thermal protection of space re-entry vehicles; shear strength tests showed that produced joints are stronger than the composites.

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Resume and Outlook by the Project Coordinator

The ExtreMat Integrated Project realized for the first time a concerted European research and development effort for materials solutions in application fields where limits are reached by conventional materials. The project is based on the idea of bridging application fields by materials solutions and thus stimulating break-throughs in pushing the limits for extreme loading conditions.

Christian Linsmeier
IPP, Garching, Germany,
Project Coordinator

ExtreMat assembled a consortium of 37 European partners from industry, SMEs, universities and research institutions. The combined efforts in searching common materials solutions for cutting-edge applications established new relations and co-operations which will last beyond the duration of the project lifetime.

Integrated approach

The true integrated approach of ExtreMat is realized in the collaborative nature of the research activities. Nearly all research efforts were carried out by two or more partners who collaborated in all stages of the activities, from problem description, definition of materials solutions, and elaboration of alternative concepts to realize new materials concepts, through the research and characterization efforts.

Throughout the whole project duration, the activities were steered and critically assessed by the industry partners, in this way ensuring that the development efforts are focussed on the problem solution. Only the ExtreMat integrated approach made it possible to develop new components, combining new materials, new bonding technologies. In addition, final dedicated application-related testing was realized on a time scale of only four years.

Scientific exchange

Project activities were not limited to collaborative research efforts, but comprise regular semi-annual workshops ("Progress Meetings"). Typically, around 100 participants discussed in topical sessions during four days their latest results, decided on the next goals and the necessary research steps.

During the late phase of the project, an "International Conference on New Materials for Extreme Environments" was organized and attracted more than 140 participants, almost half of them not affiliated with ExtreMat consortium members. Selected contributions of this conference were published in a peer-reviewed volume of "Advanced Materials Research".

The impetus of the ExtreMat project on the materials science community is also reflected in the topic "Materials for Extreme Environments", which typically comprises 4-5 symposia and is now an established part of the bi-annual conference "Euromat". Both during the ExtreMat Conference and in direct contacts with US colleagues, the idea of the cross-application materials development concept of ExtreMat also spread across the Atlantic. Now similar research consortia are under discussion in the USA.

Prosperous industrial contacts

Knowledge dissemination activities during the ExtreMat project not only addressed the scientific community in more than 200 publications, but also extended into industry and SMEs. Besides a number of ExtreMat Training Events, the consortium was present at the Hannover Messe 2008. The entire project activities were presented at an own 200 m^2 stand in the hall 'Research & Technology'. The industrial partners - as well as the research organizations and universities - established numerous contacts in business and science.

Thanks to these contacts, individual project developments moved a step closer to industrial application. Potential users identified innovative materials which they can harness to their project goals. The intensive follow-up contacts after Hannover Messe - in particular on the part of the industrial partners - accelerated this process.

The 'ExtreMat spirit'

Finally, and certainly as a benefit which cannot be judged high enough, the ExtreMat project created a fruitful atmosphere in which a wealth of concerted activities and new partnerships developed. Although the number of partners (37) from all over Europe (12 nations) is high, the consortium never fell apart into independent groups, but developed a group spirit which will certainly carry on and already stimulated a number of smaller joint activities.

Facts & Figures

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NMP3-CT-2004-500253

Start date:	2004-12-01
Duration:	70 Months
Budget:	approx. 35 million €
EU funding:	17.4 million €

Contact:
Christian Linsmeier (Coordinator)
Max-Planck-Institut für Plasmaphysik
(IPP), Garching, Germany;
linsmeier@ipp.mpg.de

www.extremat.eu

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