Project no.: **NMP3-CT-2004-500253**

ExtreMat IP

New Materials for Extreme Environments

Instrument: Integrated Project

Thematic Priority: Nanotechnology and nanoscience, knowledge-based multifunctional materials, new production processes and devices - NMP

Final Activity Report

Period covered: from	2008-12-01	to	2010-09-30	Date of preparation	2010-11-29
Start date of project:	2004-12-01			Duration:	70 month
Project coordinator name	Dr. Christian Linsmeier				
Project coordinator organisation name	Max-Planck-Institut für Plasmaphysik (IPP)			Revision	a

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Publishable executive summary

Project data

Contract number:	NMP3-CT-2004-500253-2
Project acronym:	ExtreMat IP
Project Title:	New Materials for Extreme Environments
Priority:	NMP3 Nanotechnology and nanosciences, knowledge-based multifunctional materials, new production processes and devices – 'NMP'
Project Logo:	ExtreMat
Web site:	www.extremat.eu The project website will be kept online beyond the end of the project.
Total cost:	approx. 35 Mio €
Community Contribution:	17,4 Mio €
Project duration	1.12.2004 – 30.09.2010

List of participants

37 Partners participate in the ExtreMat Consortium:

Research Centres

IPP (DE), AIT (AT), CEA (FR), DEMOKRITOS (GR), DLR (DE), FZJ (DE), JRC (EU/NL), NRG (NL), PSI (CH), UKAEA (GB),

Research Institutes

CEIT (ES), EMPA (CH), FhG-IFAM (DE), IMSAS (SK), INASMET (ES), INCAR (ES), IPP Prague (CZ),

Universities

Ecole Federale Polytechnique Lausanne (CH), Polytechnico Torino (IT), Technical University Vienna (AT), University of Alicante (ES), University of Oxford (GB), Warzaw University of Technology (PL)

Industry

Ansaldo Energia (IT), ATL (GB), Empresarios Agrupados (ES), EADS (DE), FN (IT), AREVA (FR and DE), MT Aerospace (DE), MERL (GB), NNC (GB), PLANSEE (AT), SIEMENS (DE), SGL (DE), Bayern Innovativ (DE)

Main goals

The goal of the ExtreMat IP is to provide and to industrialize knowledge-based materials and their compounds for top-end and new applications in extreme environments that are beyond reach with incremental materials development only.

The materials which are to be developed in this project shall

a) provide durable complex protection mechanisms for sensitive structures and devices operated in extreme environments;
b) provide the capability of removing extreme heat fluxes, often at very high temperature level;
c) endure radiation doses far beyond the capability of materials now available;
d) be processed into complex heterogeneous compounds that can be operated in extreme environments.

Key applications for these new materials are in the sectors of space, electronics, advanced fission and fusion applications. Further use of these materials is expected in spin-off fields, such as brake applications and energy conversion.

Key issues

To realize the goals of the project a set of radically new materials capable of withstanding extremely aggressive environments are required.

The present project constitutes a radically new approach towards these goals. The critical mass for real breakthrough-oriented RTD is assembled by pursuing the common materials issues for a range of applications in which materials have to sustain several aspects of an "extreme environment". In such applications, typically not only one loading factor occurs but combinations of several, see Fig. 1, leading to extreme and complex loading conditions of materials. The common point is that in such harsh environments the performance of existing materials is limited by the *multiple and complex functions* the materials have to provide.

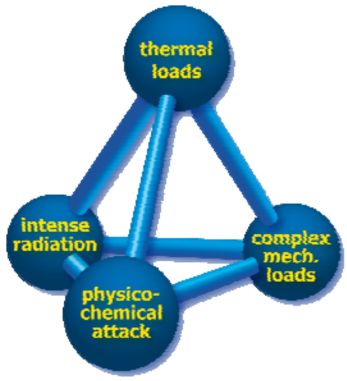

Fig. 1: **Extreme environment** is a combination of severe loading conditions at the limit of materials capabilities

New horizons of performance of materials operating in extreme environments are only accessible by merging the materials-related expertise now dispersed in different fields of applications, e.g. space industry, turbine manufacture, electronic device industry, power generation research, and radiation facilities. *Identification of the common loading aspects of materials allows pooling of basic and specific knowledge.*

This basis has yielded a new synthetic approach towards materials engineering and processing technology. This multi-sectorially oriented IP strategy aims at meeting the common need for radically new materials.

Project organization and technical approach

The Project contains four subprojects dedicated to the required functionalization of the materials and to the integration of these materials into multi-functional compounds, Fig. 2.

Three subprojects are geared towards new materials with a very high degree of functionalization (SP1, 2, 3).

- **Subproject 1**: Self-Passivating Protection Materials
- **Subproject 2**: Heat Sink Materials
- **Subproject 3**: Radiation Resistant Materials.

One subproject, **Subproject 4** forms the technological and systems engineering bracket and provides optimum interfacial technologies to combine the highly functionalized materials of the Subprojects 1 to 3 into multi-functional compounds and components for operation in extreme environments. Thus not only new materials are developed, but the embedding into this 4th subproject warrants the industrial relevance of the materials development and provides multifunctional added value compounds from these new materials.

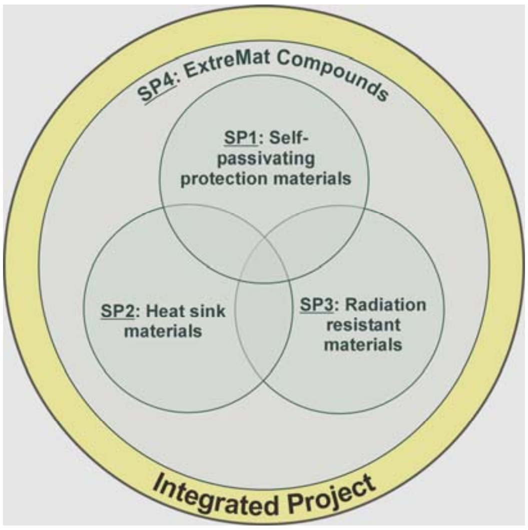

Fig. 2: ExtreMat IP structure

Each of these subprojects is structured into three, resp. four specific work packages.

All subprojects follow the same project-phasing structure, shown in Fig. 3, which ensures systematic and timely integration of all activity components into the project evolution.

The project phases and implemented milestones are synchronized to ensure that all elements of the work are available in time to allow the integration of the knowledge gained within the materials related subprojects into the compound / component-oriented contexts of subproject 4 which will have a strong application specific orientation.

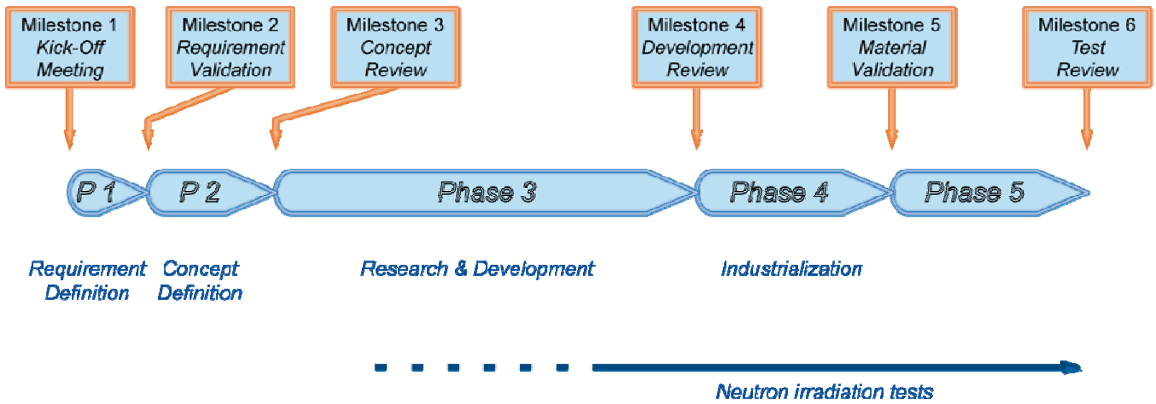

Figure 3 Project Phases

Thus for the first time this project assembles the critical mass needed for these breakthrough developments by joining the expertise, facilities and forces, which are presently scattered into the incremental improvement work for singular applications without connection to the underlying needs and common materials science issues.

Expected Achievements / Impacts

The expected achievements associated with the ExtreMat IP are to provide and industrialize the following types of knowledge-based materials and compounds for top-end and new applications in extreme environments that are beyond reach with incremental materials development only:

1. self-passivating protection materials (no erosion under oxygen, hydrogen up to 2000°C)
2. new heat sink materials (remove heat fluxes of 20 MW/m^2 at up to 1000°C)
3. radiation-resistant materials (withstand 150 dpa at 750°C; low radioactivation)
4. ExtreMat compounds (technology for functionalized compounds with very high durability)

These new breakthrough materials will allow radical innovation in the following fields of application:

- space applications: near zero-erosion of protection; multiple reuse of components (protection, thrusters); fail-safe and controlled behaviour under off-normal conditions
- new electronic device applications: a) new compact 3-d microchip architectures;
- b) high reliability, compact power electronics
- neutron-based systems: passive safety and very high temperature operation for coupled hydrogen production (VHTRs); economic realization of nuclear fusion reactors as new energy source
- *spin-off related innovation*: key fields are energy conversion (materials for hydrogen generation, functional interlayers for gas turbine components, heat exchanger materials), transport (materials for new brake systems), particle beam target heat sinks (new heat sink materials and compounds)

Results achieved

During the ***first project year*** the first two project phases have been concluded successfully and the R&D phase of the project has been launched. The requirement and concept definition phases yielded 31 industrial *User Requirement Specifications* regarding materials and compounds for applications and spin-off subjects covered by the project activities. Based on these definitions of the industrial user needs the project partners elaborated the corresponding 15 *Materials Requirement Specifications* which describe in detail the requirements to materials and compounds to be developed during the R&D phase. The Scientific Industrial Committee and the project partners have evaluated the Materials Requirement Specifications and steered the selection of the materials concepts which are to be pursued in the main R&D phase. The results are documented in the *Concept Evaluation and Selection Reports*.

The ***second project year*** was devoted to the first part of the R&D phase of the project (project phase 3). During this phase, materials and compound development was carried out based on the 15 *Materials Requirement Specifications* which were elaborated at the end of project phase 2.

Processing of materials was initiated at lab-scale levels. After determination of the material properties, for the most promising materials the processes were optimized and up-scaling efforts were started. The irradiation conditions and the test matrix of the neutron irradiation campaign were developed. Sample production for neutron irradiation was carried out and samples are delivered to NRG as the responsible partner for assembly of the irradiation capsules.

During the period three Training Activities were organized with main focus on creating awareness of the ExtreMat activities within expert communities and the objective of training young researchers in the fields of ExtreMat subjects.

The ***third project year*** was completely devoted to the R&D phase of the project (project phase 3). Moreover during phase 3 some of the concepts have been discarded, since achieved results demonstrated their unsuitability for complying with requirements; a few new concepts have been added, too.

After characterization of the material properties, for the most promising materials the processes were optimized and up-scaling efforts were started. The irradiation conditions and the test matrix of the neutron irradiation campaign were finalized. Sample production for neutron irradiation was completed and samples have been delivered to NRG as the responsible partner for assembly of the irradiation capsules and the organization of the neutron irradiation. With selected materials from all subprojects the development of processes for compound formation was enhanced and suitable joining techniques have been investigated. Procedures and techniques for the environmental testing of materials and compounds have been improved in interactions between material producers and partners responsible for testing.

During the third project year three Training Activities were organized.

The ***fourth project year*** was completely devoted to the Industrialization phase of the project (project phase 4). The industrialization phase activities focus on the up-scaling of selected successful materials concepts elaborated during the RTD phase 3. The selection of the industrialized concepts is based on the 20 *Materials Industrialization Concepts* which were elaborated mainly by the industrial partners at the end of phase 3, taking into account feasibility studies and risk minimization.

Based on the neutron irradiation test matrix, the irradiation capsules were loaded and mounted in the HFR reactor, Petten, and the neutron irradiation was started. With selected materials from all subprojects and bonding/joining technologies from SP4, compounds and mock-ups were produced. Testing and characterization of the new materials were performed in strong collaboration between partners responsible for development testing.

During this period four Training Activities were organized with main focus on training young researchers in the fields of ExtreMat subjects and towards the outreach into commercial markets and industry in order to create awareness of the achievements of the ExtreMat project. Another major event in disseminating ExtreMat results towards industry was the presentation of the ExtreMat project at the Hanover Industry Fair on an own stand.

During the ***final period (22 months)***, the project phase 4 “Industrialization” terminated for the limited number of partners who remained active for this task. Project phase 5 was carried out, during which the neutron irradiation campaign was finalized.

The neutron irradiation at HFR Petten, which was interrupted in summer 2008 due to a defect in the cooling circle of the HFR, was re-started February 2009. The ExtreMat-I irradiation capsule was removed from the reactor in April 2009, after completing the planned irradiation schedule. The ExtreMat-II irradiation capsule was removed from the reactor in September 2009. After cooling down, post-irradiation examinations were carried out for the samples with low enough activity to allow for the characterization. Post-irradiation examinations at other partners than NRG were prepared by organizing the transport of activated samples.

During all phases of the project, the Scientific Industrial Committee and the project partners have critically evaluated the progress reports and steered continuously the activities which were carried out at the partners' institutions.

Coordinator contact details

Max-Planck-Institut für Plasmaphysik (IPP)
Boltzmannstr. 2

D - 85748 Garching
Germany

Coordinator:	Project Manager:
PD Dr. Christian Linsmeier linsmeier@ipp.mpg.de Tel.: +49 89 3299 2285	Elmar Neitzert elmar.neitzert@ipp.mpg.de Tel.: +49 89 3299 1626

1 Section 1 – Project objectives and major achievements

1.1 General project objectives

Goal:

The goal of the ExtreMat - IP is to provide and to industrialize knowledge-based materials and their compounds for top-end and new applications in extreme environments that are beyond reach with incremental materials development only.

The materials which are to be developed in this project shall

a) provide durable complex protection mechanisms for sensitive structures and devices operated in extreme environments;
b) provide the capability of removing extreme heat fluxes, often at very high temperature level;
c) endure radiation doses far beyond the capability of materials now available;
d) be processed into complex heterogeneous compounds that can be operated in extreme environments.

Key applications for these new materials are in the sectors of space, electronics, advanced fission and fusion applications. Further use of these materials is expected in spin-off fields, such as brake applications and energy conversion.

Objectives:

The overall objective of the ExtreMat - IP is to provide and industrialize the following types of knowledge-based materials and compounds for top-end and new applications in extreme environments that are beyond reach with incremental materials development only:

a) self-passivating protection materials (no erosion under oxygen, hydrogen up to 2000°C)
b) new heat sink materials (remove heat fluxes of 20 MW/m^2 at up to 1000°C)
c) radiation-resistant materials (withstand 150 dpa at 750°C; low radioactivation)
d) ExtreMat compounds (technology for functionalized compounds with very high durability)

1.2 Project's relation to the state of the art:

The state-of-the-art situation is described with respect to the classes of materials forming the RTD parts of the project (subprojects):

Protection materials (Subproject 1)

State-of-art: Fibre-reinforced CMCs, e.g. C fibres, C-Si-SiC matrix: mainly low thermal conductivity materials due to inhomogeneous structure; large scatter of properties; erosion due to burn-out of C fibres and/or C matrix by oxygen; chemical erosion of C by hydrogen; instabilities in high-temperature behaviour due to excess Si; high cost (cost of fibres, cost of matrix impregnation)

Refractory metals: good mechanical properties at high temperature, reproducible production routes; very high reactivity and erosion even under low oxygen partial pressure.

Silicides: poor thermal cycling and low-temperature properties; good passivation at very high temperatures (> 1000° C), "pesting" and instability at intermediate temperatures.

Strategy of the ExtreMat project: Develop C-based highly graphitic bulk materials with very high thermal conductivity, doped at nanoscopic and microscopic level with elements acting as catalysts of graphitization and as inhibitors of both, chemical erosion by hydrogen and oxidation. Expectation of significant cost reduction compared to fibre-based composite materials. Doping of C-matrix of C/C composites. Expectation of highly improved thermal conductivity and resistance to chemical erosion by hydrogen and to oxidation; develop different SiC-based protection materials for application in extreme environments: SiC- based multilayer ceramics, short fibre C_f/SiC by Liquid Silicon Infiltration, long fibre SiC_f/SiC and SiC-based coatings. Expectation of highly improved oxidation resistance, high temperature stability, high thermal shock resistance, controlled thermal conductivity and low cost (for multilayer ceramics and C/SiC); develop W-based materials with atomically dispersed oxide-forming metal content to allow formation of stable complex oxide scale already in the medium temperature range (> 600° C); develop CrRe alloys with high thermal shock and oxidation resistance and with lower cost compared with Pt-based alloys.

Results of subproject 1 (Self-passivating protection materials): Different methods to obtain doped isotropic graphites and CFCs with fine and homogeneous dopant dispersion and required properties for the envisaged applications were developed and optimized. Best results were achieved with Ti-doped graphites and Ti-doped 3D CFCs for fusion first wall application, where materials with very high thermal conductivity (> 200 W/mK in all directions for graphite, close to 300 W/mK in one direction for CFC), relatively high mechanical strength, strongly reduced chemical erosion (at least for Ti-doped graphite) and highly improved resistance to off-normal thermal loads (ELMs, disruptions) were obtained. Mock-ups were manufactured by joining these materials to a CuCrZr heat-sink (within SP4); these mock-ups were tested at thermal cyclic loads up to 20 MW/m^2 (SP4): the one with Ti-doped graphite survived 100 cycles at 20 MW/m^2 on one tile plus 2 additional cycles at 21.8 MW/m2, after which no total failure of the tile occurred; and the one with Ti-doped 3D CFC survived 62 cycles at 20 MW/m^2 (testing was only stopped due to a malfunction of the clamping mechanism to the cooling structure). C-SiC-B_4C composites with self-passivating behavior against oxidation and reasonable mechanical properties were produced for space applications. Re-entry tests on these material showed that burn-off did not start until 1400°C, and at 1500°C lifetime is limited to ~10 cycles. Deposition of a HfO_2 coating resulted in a tremendous improvement of oxidation resistance. SiC-based multilayer ceramics with different composition and architecture were processed and characterized. For the most promising concepts the processing way was further optimized and the definitive method for processing multilayer SiC (containing only dense layers) was transferred to FN for industrialization. These materials fulfil all requirements for their application as TPS of re-entry vehicles, while being significantly cheaper than SiC_f/SiC materials. Short fibre C_f/SiC composites with improved performance were manufactured by liquid silicon infiltration and the processing optimized. Large C_f/SiC brake discs, some of them with and oxidation protective coating, were manufactured. The friction and wear tests performed on these discs gave very promising results, showing a higher friction coefficient than that of the commercially available C_f/SiC material and a lower wear. 2D and 3D long fibres SiC_f/SiC composites were manufactured by CVI and the process optimized. CVD-based high temperature SiC-lubricant coatings for CMCs (C/SiC and SiC/SiC) bearings were developed and optimized for application in re-entry space vehicles. 27 re-entry missions under bearing loads, 1550°C and movements could be demonstrated. Sputtered graded multilayer protective coatings based on SiC and HfO_2 have been developed and tested. CrRe alloys for application in satellite thrusters were investigated concerning hot forming, machinability, weldability and high temperature resistance under vacuum. The results led to the conclusion that CrRe35 is a promising concept but has some specific weakness which affects key properties. Therefore, the CrRe activities were stopped in month 30. Thin film deposition of ternary and quaternary W-based alloys was performed and the oxidation resistance of these films tested. Best results were obtained with the quaternary alloy WSi3Cr10Zr5. The overall results indicated that quaternary alloys show better passivation behaviour than ternary while containing more W; active elements (Y/Zr) do not form oxide layers, but improve oxide scale adhesion; the surface oxide consists of Cr_2O_3; and different oxide phases formed, but no WO_3 which means that the passivation is successful. Thick CVD W coatings on Eurofer steel substrates involving CVD erbia as tritium permeation barrier and copper as stress absorbing interlayer were produced; the coatings delaminated due to thermal mismatch, despite modelling and re-engineering of the interface region. Modelling of the oxidation process of W-36Si-20Cr and W-36Si-20Cr-11Zr systems was done using the experimentally obtained oxidation rate constant. The concentration profiles of W, Si and Cr in the alloy during oxidation at 1000°C were calculated. The materials development within SP1 was supported by application oriented environmental tests and special characterizations within WP1.4 as well as within SP3 and SP4.

Heat sink materials (Subproject 2)

State-of-art: Use of pure highly conductive metals like Cu, Au, Ag is limited because of poor mechanical properties, especially at high temperatures and no possibility to control CTE according to substrate needs. Alloying may lead to improvement of mechanical properties, but mostly on cost of thermal conductivity. The adjustment of CTE is possible only in composite approach. Cu-based, Ag-based composites with highly conductive low CTE reinforcements have a potential to provide much higher thermal conductivity and controlled CTE; the main road blocks however exist in the creation of stable interfaces between matrix and reinforcements with low interfacial thermal resistance and sufficient strength to withstand high thermal stresses due to CTE mismatch - these obstacles prevent the breakthrough of economic new-generation heat sink materials. AlSiC composites are at market level, but of very poor machinability and rather low thermal conductivity.

Strategy of the ExtreMat project: The main aim was to develop two new basic types of heat sink composites:

A. Heat sinks based on copper matrix reinforced with ceramic and intermetallic (nano) particles or fibres for use in high temperature applications (up to 1000°C); where excellent dimensional stability combined with good thermal conductivity play a primary role. This work was devoted to ***Workpackage 2.1 – "High temperature heat sinks"***. The objective was to achieve thermal conductivity comparable to Cu (300 W/mK) with a heat flux removal capability of up to 20 MW/m^2. Additionally, the coefficient of thermal expansion (CTE) in plane direction at the interface needed to be adapted to the CTE of the protection material which is typically $4...9 \times 10^{-6}K^{-1}$.
B. Heat sinks based on highly conductive phases (diamond, highly graphitised carbon fibres, carbon nanotubes with theoretical thermal conductivity 800-6000 W/mK) embedded in appropriate metallic matrix for use in applications, where extreme heat conductivities combined with tailored CTE are the main requirements. This work was devoted to ***Workpackage 2.2 – "High conductivity heat sinks"***. The CTE in plane direction at both interfaces needed to fit the CTE of the adjacent structures, typically $4...9x10^{-6}K^{-1}$.

Further requirements to both material groups included structural stability during thermal cycling within working temperature range (e.g. n > 5 x 10^6), joinability (e.g. brazability), control of internal stresses, dimensional stability, and acceptable costs. To overcome the limitations of current materials optimum architecture of novel composites was modelled and the interfaces between constituents were tailored at nanoscopic level in ***Workpackage 2.3 "Nanoscopic interface design and modelling".*** The main aim of this WP was to develop stable interfaces in the whole range of working temperatures without degradation of thermal conductivity.

The work based on integrated approach comprising new processing methods aimed at keeping high thermal conductivity and minimizing thermal stresses throughout all synthesis steps; atomistic modelling and interfacial engineering at nanoscopic level in order to reduce interfacial thermal resistance; tailored reinforcement architectures to balance local CTE, (high temperature) strength, and thermal conductivity requirements.

Results of subproject 2 (Heat sink materials):

The requirements for targeted applications for novel heat sink materials were defined in **8 User Requirement Specifications** (URS) comprising: avionic modul, launcher propulsion, rotating X-ray anode, divertor in fusion reactor, novel SiC chip, opto-, power- and microelectronics components. Based on these requirements **25 state-of-the-art studies** and SWOT analysis on potential materials for novel heat sinks, incl. review on testing methods, existing modelling approaches and activities concerning development of optimum interfaces were elaborated. **35 alternative material and technological concepts** capable to meet the targeted requirements were suggested by research partners, most promising of them were further developed on lab-scale with the aim to receive well-defined small specimens for characterization and relevant environmental testing. The main attention was given to copper and aluminium matrix composites reinforced with diamond, tungsten wires, continuous SiC or carbon fibres and highly conductive graphite flakes or carbon nanofibres. The liquid (gas pressure infiltration, squeeze casting) and solid state manufacturing techniques (hot pressing of coated reinforcements, spark plasma sintering, powder metallurgical methods) were optimised from the technical as well as economical point of view in order to meet all targeted requirements. The interfaces in composites were modified via ultra thin surface coating of reinforcements and/or mutual reaction with matrix at nanoscopic level in order to reduce thermal resistance of the interface. Material properties were characterized with respect to thermal conductivity, CTE and level of internal stresses. Almost all material systems underwent thermal cycling testing in order to characterise their structural stability. The modelling studies were performed to optimize composite architecture with an aim to minimise internal stresses and maximise thermal conductivity. The samples for testing of heat sink performance in near to application conditions were suggested and most of them also manufactured from most promising developed materials. Selected Cu-W and CuCrZr-W and Cu-SiC composites underwent irradiation test in SP3.

The plans for further industrialisation use of developed materials were established in **8 Material industrialisation concepts** (MIC) regarding Cu/SiC monofilaments, Cu/W wires, Cu/VGCNF, Al/diamond and Al/SiC, Ag/diamond and Cu/diamond, Cu/diamond PM, Al graphite flakes and Cu(Al)/short C fibres. Gas pressure infiltration, squeeze casting, hot diffusion bonding and spark plasma sintering (SPS) were approved as most feasible processing techniques for further industrialisation.

Performance of developed materials was evaluated on various mock-ups prepared for selected electronics, space and fusion applications (divertor, thermal lid, IgBt-base plate, RF package, laser bar insert, etc.) and tested by industrial partners in close cooperation with SP4 (service related component testing). The performance and characterisation data were supplied to WP 2.3 partners for validation of material and component models.

Following main results may be considered as **highlights** of SP2 research:

- ***New developed materials including:***
  - thermally stable Cu –W composites with „isotropic" CTE (~6 ppm/K) capable to withstand termocycling at extreme heat fluxes (~20MW/m^2)
  - machinable high conductivity carbon fibre heat sinks with thermal conductivity exceeding 700 W/mK)
  - low cost machinable graphite flake composites (TC~400W/mK, CTE~9ppm)
  - unique Ag based-diamond composites with extremely high thermal conductivity ~over 900 W/mK
- ***Novel technologies for making composites***
  - spark plasma sintering,
  - continuous PVD coating,
  - pressure infiltration and squeeze casting
- ***Potential applications with improved performance***
  - power electronic packages (IGBT plate, thermal lid)
  - part of divertor for fusion reactor
  - optoelectronics components (slab crystal housing, laser bar insert)
- ***Novel techniques used for advanced characterisation of composite materials***
  - thermal mapping for characterisation of structural uniformity
  - synchrotron tomography and neutron diffraction for characterisation of internal stresses
- ***New models for fibre and particle reinforced composites***

Radiation-resistant materials (Subproject 3)

State-of-art: Low-activation ferritic/martensitic steels exist at experimental level and exhibit stability only up to moderate temperatures (T_{max} = 550°C); this leads to low energy conversion efficiency in energy applications. ODS ferritic steels and refractory materials will have better stability at higher operation temperatures. Main issues are low-temperature embrittlement, loss of ductility leading to instability during operational temperature ramps, and high-temperature creep resistance. C- and SiC-based materials show potential for very high-temperature application (of the order of 1000°C), but advanced composites are unstable under high dose irradiation (degradation of thermal conductivity, loss of dimensional stability).

Strategy of the ExtreMat project: Modelling-based approach to determining optimum architecture and dispersion of nanoparticles in metal matrices. Development of lab-scale ODS ferritic steels and W-base materials for irradiation and pre- and post-irradiation characterization. Investigation of the neutron irradiation behaviour of materials developed within SP3, of specifically tailored protection materials from SP1, of high-strength, high-temperature heat sink materials from SP2, and of small mock-ups from SP4.

Results of Subproject 3 (Radiation resistant materials): During Phase 1 the SP3 partners contributed to the preparation and evaluation of four Users Requirement Specifications (URSs), which led to the preparation of four Material Requirement Specifications (MRSs) for oxide dispersion strengthened (ODS) steels, tungsten-based materials, nanostructured monolithic ceramic materials and carbon fibre reinforced ceramics, respectively. In addition, all needed numerical and experimental tools for R&D activities during subsequent Phases have been assembled and tested by the SP3 partners. During Phase 2, Alternative Concepts (ACs) for further activities have been identified by the SP3 partners. R&D activities were performed during Phases 3, 4 and 5. Main results are summarized just below.

A new interatomic potential for pure tungsten has been developed, which is consistent with density functional theory predictions in terms of migration and formation energies of point defects. The new interatomic potential developed for pure tungsten has been fully parametrized for further molecular dynamics (MD) simulations of radiation damage in pure tungsten. A new method for connecting this new long-range potential to the universal short-range one has been proposed. MD simulations of atomic displacement cascades, structural defects and migration of edge and screw dislocations in pure tungsten, by using all available interatomic potentials, have been successfully performed. The ductile-to-brittle transition (DBT) in various pure tungsten materials has been investigated by means of fracture tests. A DBT was observed at a well-characterised temperature for each strain rate. The strain-rate variation of the DBT temperature in all the tungsten materials investigated was found to fit an Arrhenius law with an activation energy of approximately 1.05 eV. Dislocation-based modelling of the DBT in single crystalline tungsten was successfully performed, as well as electron back-scattered diffraction studies aimed at mapping the strain field around crack tips. A comprehensive model that explains the anomalous radiation damage effects occurring in iron and steels at elevated temperatures was developed. It showed 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. This work revealed for the first time the fundamental link between the nature of radiation damage observed experimentally at temperatures approaching 500°C, the diffusionless phase transitions occurring in iron and iron-based alloys at about 900°C, the anisotropic elastic properties of iron and iron-based alloys that become particularly significant at these elevated temperatures, magnetic fluctuations, and the loss of mechanical strength of steels at elevated temperatures.

Oxide dispersion strengthened (ODS) reduced activation ferritic (RAF) steels, with the chemical composition of Fe-(12-14)Cr-2W-0.3Ti-0.3Y_2O_3, have been produced by mechanical alloying followed by either hot isostatic pressing (HIPping) or hot extrusion. Both materials were found to exhibit a bimodal grain microstructure, high tensile strength and good ductility, but relatively poor Charpy impact properties. The microstructure and Charpy impact properties have been significantly improved by applying thermo-mechanical treatments, such as hot rolling, hot pressing or cold pressing. On the other hand, commercial pure W and W-1%La_2O_3 materials have been submitted to high-speed hot extrusion (HSHE) at Warsaw University of Technology (SP1 partner) in an attempt to improve their ductility. Pure W was found too brittle for the HSHE process. This process allowed plastic deformation of the W-1%La_2O_3 material but no significant improvement in the DBT temperature. The potentiality of using equal channel angular pressing (ECAP) for producing nanostructured ODS ferritic steels has been demonstrated. Following its application to the commercial PM2000 ODS ferritic steel, it was applied to the new 14 Cr ODS RAF steel developed within ExtreMat, in an attempt to improve the ductility of these materials. Unfortunately, no significant decrease of the grain size was obtained by applying this method, even at temperatures above 500°C. It was therefore attempted to apply high-pressure torsion (HPT) to the commercial PM2000 ODS ferritic steel and to the new 14Cr ODS RAF steel. Smaller grain size materials with a higher microhardness were obtained by means of HPT. However, the quantities that can be produced by using this technique are very small.

High purity, nanometric SiC powder particles destined to the preparation of high quality, advanced SiC-based materials within SP1 and high purity, dense, nanostructured, monolithic SiC have been produced. High heat flux experiments were performed on unirradiated and available irradiated specimens, in the aim to define testing parameters to be used for further high heat flux experiments on neutron-irradiated specimens.

Various types of specimens have been prepared by the various ExtreMat partners from the most promising metallic and ceramic materials developed within the project. Two neutron irradiation campaigns in the high flux reactor (HFR) were started in February and May 2008, respectively. Unfortunately, due to technical problems, the neutron irradiations were stopped in summer 2008 and restarted in February 2009. The ExtreMat I irradiation (low-dose, low temperature neutron irradiation) ended on April 26, 2009. Specimens were irradiated at 300 and/or 550°C. The ExtreMat II irradiation (high-dose, high temperature neutron irradiation) ended on September 08, 2009. Specimens were irradiated at 600 and/or 900°C to about 4 dpa (in steels). Both irradiations included ceramic and metallic specimens. 493 specimens in total have been neutron irradiated in the HFR. Post-irradiation experiments (PIEs) on metallic and ceramic materials that were neutron-irradiated in the HFR have been shared between NRG, the EPFL, the CEA and FZJ. Post-irradiation characterization of the physical properties of the less radioactive ceramic specimens has been completed. All other neutron-irradiated specimens are still in the cooling phase, i.e., they are still too radioactive to be tested. Transports from NRG to the PSI, the CEA and FZJ of a part of the neutron-iradiated specimens are being organized. The transports should take place by the end of 2010, and PIEs on these specimens should be performed in 2011-2012.

Post-irradiation tensile testing of specimens of the MA956 and MA957 ODS ferritic steels neutron-irradiated in the Phénix reactor at temperatures in the range 410-550°C to various doses up to 80 dpa revealed significant irradiation-induced hardening and reasonable ductility. Results have been analyzed in terms of irradiation-induced α' precipitation. The microstructure and creep behaviour of the commercial PM2000 ODS ferritic steel has been characterized, before and after ion irradiation. It was found that irradiation creep is important up to about 680°C. At higher temperatures thermal creep becomes predominant. The irradiation creep data obtained for the helium-implanted, commercial PM2000 ODS ferritic steel were compared to data reported in the literature for other ODS ferritic steels as well as for ferritic and ferritic/martensitic steels. It was found that the size of dispersoids has no significant effect on irradiation creep and that the irradiation creep of ODS steels is comparable to that of non-ODS materials. SiC and SiC/SiC specimens have been irradiated in the SINQ facility (the Swiss Spallation Neutron Source) with a mixed spectrum of high-energy protons and spallation neutrons. Testing of reference (unirradiated) specimens was performed by means of three-points bend tests, small ball punch tests and Extended X-ray Absorption Fine Spectroscopy (EXAFS). Helium-implanted specimens of SiC were also characterized using EXAFS. The Fourier transform of EXAFS spectra revealed a strong decrease of the coordination number of atoms in the second shell, corresponding to the next neighbour silicon atoms, with increasing irradiation dose.

Industrial evaluation of the materials developed within SP3 with respect to applications chosen for investigation has been continuously performed. Data requirements for industrial application, R&D, manufacturing and design have been identified. In particular, the materials requirements as well as the candidate materials for Generation IV reactors, more especially for very high temperature reactors (VHTRS), have been reviewed. The longer-term industrialisation of the materials developed within SP3 was also addressed. A review of existing standards for experimental testing of non-metallic materials has been also performed. Activities related to the development of a database to store the test results from the neutron irradiation programme carried out in the HFR and to share these amongst the partners within the project were also addressed.

Compound technology including barriers (Subproject 4)

State-of-art: The diffusion barrier technology for high-temperature application is insufficient: main existing route comprises oxide-forming alloys by segregation processes. Naturally grown oxide layers on the basis of these concepts are of rough structure. During operation the layer growth continues until stress-induced spallation and component failure occur. Another existing route is refractory thin-film metals for very specific barrier application (e.g. carbon interdiffusion).

Bonding and interface technologies are highly application-specific; heat flux removal of up to 20 MW/m^2 through the interface is required (high-heat-flux components for plasma facing applications in nuclear fusion); the problems are reproducibility of parameters over various batches, availability of a technology suitable for large and complex components and cost reduction; moreover state-of-the-art components, using CuCrZr as heat sink material, are suitable for nuclear fusion research facilities (including ITER), but they can not operate at the high temperature (around 600 °C) required for nuclear fusion reactors (DEMO and future power plants). A problem in other applications is to obtain lower processing temperatures, reduced interdiffusion, and stability under very high cycle numbers. In joining of protection materials to metallic alloys for space applications (like in re-entry vehicles), tailoring of the surfaces for chemical compatibility and therefore joining is still a challenging task, as well as the resistance to stresses induced by thermal load and shock. Electronic applications need the formation of compounds comprising a heat spreader and an active electronic component with an interlayer, being both electrically insulating and thermally conductive. Available technologies based on brazing/soldering of ceramic sheets are limited by costs and thermal cycling resistance; processes involving the deposition of the ceramic layer as a coating need to be more flexible (adaptable to different geometries) and cheaper, but they are not yet available.

Methods of testing and characterizing under application-relevant conditions are available but not always satisfactory; accelerated testing needs to be established.

Strategy of the ExtreMat project: Develop oxide thin-films as barriers against hydrogen diffusion, integrated (buried) within multilayer systems where necessary. Develop thin-films (single layer or multilayers) acting as diffusion barriers and/or adhesion promoters during bonding processes based on high temperature brazing or diffusion bonding. Develop thick interlayers able to reduce (or to withstand instead of weaker materials) residual stresses in bonding of heterogeneous materials. Develop deposition processes for thick layers of protective materials, for thick graded interlayers and for electrically insulating layers. Develop application-specific bonding technology in order to apply the new materials developed in other SPs and the barriers and interlayers developed in SP4 to compounds and components for specific applications.

Results of Subproject 4 (ExtreMat compounds):

- CVD Erbia diffusion barrier films against Tritium diffusion (permeation reduction factor of 10^3 to 10^4) developed and industrial applicability demonstrated producing large flat samples and long tubes (up to 400 mm); application in breeder blanket modules of nuclear fusion reactors.
- CVD technology for Rhenium deposition, tested between CFC and W for nuclear fusion and on Ni-based superalloys as interdiffusion barriers for NiCoCrAlY coatings.
- Si_3N_4 CVD films for protection of graphite components in silicon processing facilities, demonstrated on real components that operated for more than 1 year in a commercial production plant.
- CVD W and SiC on diamond grit: Coatings on diamond grit for improved wetting by molten metals (and enhanced thermal transfer) in diamond based Metal Matrix Composite heat sinks. Batches of diamond grit as well as infiltrated preforms delivered to SP2, for production of heat sinks with Cu and Al matrices (improved heat transfer between diamond particles and metallic matrix demonstrated).
- CVD technology for deposition of W layers on C-based protective materials and on Cu-alloys, thicker than the state-of-the-art ones.
- Processes for thermal conductivity improvement of plasma sprayed W and W-Cu composites and graded layers.
- Wetter promoters for brazing: Films to be used as wetter promoters for brazing developed (TiN_x and TiC by sputtering and Si_3N_4 by CVD), as well as characterisation devices (High Temperature Sessile Drop Device specially designed within this project); $TiN_{0.3}$, TiC_x and Ti by sputtering as well as Si_3N_4 and TiC by CVD chosen and active cooled plasma facing mock-ups prepared (4 of them showed good performance up to 15 MW/m^2 high heat flux testing without tiles failures).
- Use of low CTE interlayers in brazing of flat tile plasma facing compounds:
  - Supported by FEM calculations
  - Demonstrated with Mo as interlayer and state-of-the-art industrial NB31 CFC
  - Tested with SP2 Wf/Cu MMC as interlayer and state-of-the-art industrial CFC
  - Tested with SP2 SiCf/Cu MMC as interlayer and W tiles
  - Tested with Mo as interlayer and SP1 Ti-doped graphite and CFC
  - Tested with SP2 Wf/Cu MMC and TiN, TiC and Si_3N_4 coated NB31 CFC
  - Tested with SP2 Wf/Cu MMC and SP1 Ti-doped graphite
  - Successful at least up to 15 MW/m^2 high heat flux testing on almost all mock-ups and up to 20 MW/m^2 on best ones
- Direct brazing of W to CuCrZr heat sinks for intermediate heat flux applications in nuclear fusion; active cooled PFC mock-ups successfully tested up to 15 MW/m^2 high heat flux; 2 mock-ups delivered for nuclear irradiation and, subsequently, high heat flux testing.
- Monoblock mock-ups with Wf/Cu MMC heat sink tube, produced by gas pressure infiltration of molten Cu in Wf preforms, with simultaneous formation of MMC heat sink tube and joining to the W tiles; successful up to 20 MW/m^2 in high heat flux test; technology potentially suitable beyond ITER, for application at high temperature as expected in future commercial nuclear fusion power plants.
- Monoblock mock-ups with CuCrZr tube reinforced by Wf/Cu MMCWf/Cu MMC consolidated by HIPping on CuCrZr tubes, then W tiles brazed; successfully tested up to 10.5 MW/m^2.

- CMCs to metals joining process (for space applications): surface modification of C_f/C and C_f/SiC by Cr sputter deposition, then brazing with TiCuSil alloy to Nimonic 80 Ni-based superalloy; compounds failed in shear tests inside the CMC, not at the joint; similar results achieved also with a composite brazing technique (C-fibres addition inside the brazed joint).
- CMC joining process for nuclear fusion applications: mechanical joint plus glass ceramic sealing developed and compounds tested in neutron irradiation; self-propagating high temperature synthesis, ingnited by microwaves, tested as alternative joining process.
- Contribution to characterization of materials and compounds from other SPs: high heat flux tests for nuclear fusion applications, tests of heat sinks for electronic applications (thermal cycling, "pressure cooker", thermal conductivity) and deuterium permeation measurements through diffusion barriers.

Work performed, results obtained and main achievements

The first two project phases formed the main part of the first 12 months period (phase 1: months 1-4; phase 2: months 5-10; phase 3: planned for months 11-36). The work performed in the first 12 project months followed consequently the described objectives and yielded the expected results. The milestones were reached and the work for the project phase 3 (RTD phase) has been launched. All deliverables listed in Annex I (rev. i), pages 65-72, for the project phases 1 and 2 have been produced.
The second project year period (months 13 to 24) lied completely within the project phase 3 "Research & Development" which started in month 11. The work performed followed the results from phases 1 and 2 which determined the *Alternative Concepts* to be investigated within the work packages to develop materials and components which comply with the *Material Requirement Specifications*. The work performed was reported and evaluated continuously at the *Subproject Meetings* and by the *Scientific Industrial Committee*. All milestones were reached in the first part of phase 3. All deliverables listed in Annex I (rev. k), pages 53-56, for the project phase 3 have been produced.
The third project year (months 25 to 36) formed the last part of the project phase 3 "Research & Development", which lasted from month 11 until month 36. The work performed followed the results from phases 1 and 2 which determined the *Alternative Concepts* to be investigated within the work packages to develop materials and components which comply with the *Material Requirement Specifications*. The work performed was reported and evaluated continuously at the *Subproject Meetings* and by the *Scientific Industrial Committee*. All milestones were reached during phase 3 with some technical adjustments to the materials concepts pursued and the schedule of the neutron irradiation. All deliverables listed in Annex **I (rev. l), pages 56-59**, for the project phase 3 have been produced.
The fourth project year (months 37 to 48) formed the project phase 4 "Industrialization", which lasted from month 37 until month 48. The work performed followed the results from phases 1, 2, and 3 which determined the *Alternative Concepts* to be investigated within the work packages to develop materials and components which comply with the *Material Requirement Specifications*. Finally, the *Materials Industrialization Concepts* document the selected concepts for the industrialization activities. The work performed was reported and evaluated continuously at the *Subproject Meetings* and by the *Scientific Industrial Committee*. All milestones were reached during phase 4 with some technical adjustments to the materials concepts pursued. A major deviation from the schedule was caused by the interruption of the operation of the HFR, Petten, delaying the activities related to the neutron irradiation and

respective post-irradiation characterization by at least eight months. Due to this *force majeure* event, the new project schedule aims at an extension until 11/2010 in order to finalize the neutron irradiation activities. Deliverables listed in Annex I (rev. o), pages 56-59, for the project phase 4 have been produced.
The final project period (months 49 to 70) comprised the phase 4 “Industrialization” for a limited number of partners, and phase 5 in which the neutron irradiation campaign was finalized. The work performed was reported in the technical documents. Both phase 4 and phase 5 milestones according to the project planning were reached during the reporting period. The neutron irradiation campaign was carried out and terminated successfully. The status of the irradiation-related activities was reported and evaluated by the project partners. Deliverables listed in Annex I (rev. q), pages 55-58, have been produced.

Contractors involved

All contractors IPP; AEN; ATL; CEA; CEIT; EADS; EMPA;EPFL; FN; FZJ; IFAM; IMSAS; MERL; NRG; PLANSEE; POLITO; PSI; SIEMENS; UALICANTE were involved in the activities. Section 2 features tables for each subproject which show the involvement of the contractors in the respective subprojects and workpackages.

1.3 Training Activities

Training activities in ExtreMat had four major objectives:

Internally:
- to train ExtreMat partners in a proper and effective protection of the know-how generated in a EU-funded research project
- to train the staff of ExtreMat consortium partners in the proper use of methods and knowledge generated within ExtreMat

Externally:
- to facilitate the immediate pick-up of materials design and processing know-how in the materials industry and a quick innovation cycle of market-ready new materials
- to facilitate the rapid integration of the new technologies into new products and systems

During the ExtreMat project, 10 targeted training activities with more than 300 participants in total were organised:

1. Tutorial Course “C&SiC Composites for Nuclear Applications”,
   20 September 2006, Petten, The Netherlands (main organizer NRG)
2. Tutorial Course “Plasma-Facing Materials and Components for Fusion Applications”,
   9 October 2006, Greifswald, Germany (main organizer IPP)
3. Conference “High-performance diamond-based composites: Innovations in superabrasives and thermal management”,
   10 November 2006 Dübendorf, Swizzerland (main organizer EMPA/PLANSEE)
4. ExtreMat Training “Thermophysical Workshop”,
   22-23 January 2007, Jülich, Germany (main organizer FZJ).

5. ExtreMat Training "Space Materials for high temperature applications", 26-27 June 2007, Torino, Italy (main organizer BI / POLITO)
6. ExtreMat Training "Novel heat sink materials for power electronics applications", 10 October 2007, Nuremberg, Germany (main organizer BI)
7. Workshop on "Protecting and Exploiting Intellectual Property generated in ExtreMat", 3-4 April 2008, Munich, Germany (main organizer BI)
8. Session "ExtreMat – New materials for Extreme Environments"
   within the public "Forum Tech Transfer – The Direct Route to the Market" at the Hanover Industry Fair,
   22 April 2008, Hanover, Germany (main organizer IPP)
9. Tutorial Course on "ExtreMat materials R&D" at the 1st Conference on New Materials for Extreme Environments,
   2 June 2008, San Sebastian, Spain (main organizer INASMET / IPP)
10. Evening Seminar for Professionals on "Materials for Extreme Environments", 16 October 2008, Garching, Germany (main organizer BI).

1.4 Interaction with other EU projects and Technology Platforms

Common meetings with other European projects and organizations as well as presentations of the ExtreMat project at European initiatives were organized.

In particular:

1. Link Meeting with the RAPHAEL-IP, 6 March 2006, Garching
2. Presentation of the ExtreMat project at the EFDA STAC Meeting, 16 March 2006
3. Presentation of the ExtreMat project at the Launch Event of the EuMaT Technology Platform, 27 June 2006, Brussels
4. HYTHEC / ExtreMat First Technical Exchange Meeting, 5 December 2006, Saclay
5. Meeting "Fusion / Fission Synergies in Materials", organized by AREVA-D on 7 February 2007 in Erlangen, with participation of representatives from ExtreMat IP, Raphael IP, and EFDA
6. Common meeting with the KMM NoE ("Knowledge-based Multicomponent Materials for Durable and Safe Performance"): "2nd KMM-NoE Integration Conference" on 24/25 October 2007, Vienna
7. Participation and presentation of ExtreMat results in the meetings of the EFDA Topical Group "Materials Science" (2008-01-21/22 and 2008-04-02, Garching, Germany).
8. Based on the results of the ExtreMat project and in close interaction with EFDA, a Coordination and Support Action "FEMAS-CA (Fusion Energy Materials Science Coordination Action)" launched at 1 October 2008 in the frame of FP7 (Euratom)

9. Information meeting between Ch. Linsmeier and Marjorie Bertolus (Deputy Coordinator) of the FP7 project “F-BRIDGE”, and exchange of project ideas, Marseille, 2008-10-03.
10. Continuous contact and interactions with the EuMaT Technology Platform (participation in the Steering Committee of EuMaT), coordination of the EuMaT WG 4 “Knowledge-based multifunctional materials and materials for extreme conditions”, co-chaired by M. Basista (WUT) and Ch. Linsmeier (IPP) [until October 2008]
11. Participation and presentation of ExtreMat results in the meetings of the EFDA Topical Group “Materials Science” (Jan. 2009, Garching, Germany, and July 2009, Stockholm, Sweden).
12. To harmonize the documentation of data resulting from the neutron irradiation campaign between different EU projects, the respective SP3 partners attended the necessary RAPHAEL, GCFR, EUROTRANS, ELSY and EISOFAR meetings.

2 Section 2 – Workpackage progress of the period

2.1 Subproject 1: Self-passivating Protection Materials

SP1 is divided into ***4 work packages*** (WP) and ***21 project partners*** contribute to this SP:

Partners	**WP 1.1: C-based materials**	**WP 1.2: SiC-based Materials**	**WP 1.3: Refractory based Materials**	**WP1.4: Environmental tests and industrial evaluation**
CEIT (SP coord)	**X**			
INCAR	**X**			
UALICANTE	**X**			
SGL	**X**			
POLITO		**X**		
MTA		**X**		
FN		**X**		
DEMOKRITOS		**X**		**X**
EADS		**X**	**X**	
ATL			**X**	
WUT			**X**	
PLANSEE			**X**	
IPP			**X**	**X**
NNC				**X**
AIT				**X**
AEN				**X**
EA				**X**
AREVA-FR				**X**
FZJ				**X**
JRC				**X**
MERL				**X**

X: WP partners **X** WP leader

2.1.1 WP 1.1: Carbon-based Materials

2.1.1.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
CEIT	Development of nm-dispersed doped isotropic graphite by mixing of C-raw materials with nanopowders				77,3
INCAR	Development of doped CFCs				46,6
UALICANTE	Development doped CFC and doped Iso Graphite for fusion/fission application				49,7
SGL	Development of nanodispersed doped mesophase by co-pyrolysis				33,7

Objectives

Main: Development of nm-dispersed doped isotropic graphites and CFCs with high thermal conductivity and high thermal-shock resistance (passive stability under off-normal events), and self-passivating effect under hydrogen and oxygen exposure up to 2000°C.

Objectives of **Phase 1**:
- To identify the real needs of end-users for carbon-based materials and to define the requirements the materials have to fulfill for the envisaged applications.
- To define R&D concepts for Phase 2 according to the material requirement specifications in view of selecting the most promising R&D concepts at the end of Phase 2.

Objectives of **Phase 2:** to select the most promising C-based materials concepts according to the material requirement specification identified in Phase 1, and to define the R&D activities for Phase 3 taking into account the risk involved, the resources requirements and the schedule.

Objectives of **Phase 3:** to develop the C-based materials concepts selected in Phase 2 and to test samples of these new materials under the conditions required by the applications to demonstrate their compliance with the Materials Requirement Specifications. The concepts shall undergone the SIC evaluation and be validated/rejected during and after each of the two SP1 meetings planned for this period.

Objectives of **Phase 4:** industrial up-scaling of the most promising concepts developed during Phase 3, according to the guidelines proposed in the Material Industrialization Concepts prepared at the end of Phase 3. As previously, the concepts shall undergone the SIC evaluation and be validated/rejected during and after each of the two SP1 meetings planned for this period.

Description of work

Phase 1: review on promising dopants for passivation (CEIT); review of promising methods for obtaining doped isotropic graphites with nanoscopic dopant dispersion by different processing routes (CEIT, UALI); review on the state-of-the-art of CFCs (INCAR); proposal of R&D concepts for Phase 2 in view of selecting out of them the most promising R&D concepts at the end of Phase 2; evaluation and validation of User Requirements Specifications (URD) and Material Requirement Specifications (MRS) related to fission applications of C-based materials provided by other ExtreMat partners (SGL).

Phase 2: definition of selection criteria for alternative concept proposals on C-based materials and selection of proposals for material concepts. At the end of Phase 2, revision of the MRS and of alternative concept list, and contribution to the planning of Phase 3. The R&D work during Phase 2 includes:
- Selection of appropriate dopants for relevant applications; selection of different carbon raw materials according to the MRS; study of different methods to introduce dopants in different C-raw materials; start of processing of doped isotropic graphites by different methods (CEIT, UALI).
- Identification of promising carbon precursors for CFCs; identification of synthesis parameters and routes for introducing dopants; evaluation of methods for introducing dopants into matrix precursor (INCAR).

- Development of concepts for manufacturing doped 3D-CFC and Iso Graphite; definition and preparation of samples for infiltration trials (2D and 3D CFC); evaluation and validation of Alternative Concepts for doped isotropic graphites and CFCs as regards fission and fusion applications (SGL).

Phase 3: start of R&D work on the alternative concepts proposed during Phase 2 to demonstrate their compliance with the MRS:
- Development and optimization of different methods to obtain doped isotropic graphites with nanoscopic dopant dispersion and required properties (CEIT, UALI)
- Development and optimization of processes to obtain doped CFCs with nanoscopic dopant dispersion and required properties (INCAR and SGL):
- Development of 2D and 3D fabric for impregnation trials with pitch/dopant matrix-systems (SGL).
- Preparation of samples for neutron irradiation, for tests/characterization by WP1.4 partners and for SP4

Exchange of information/technologies and materials between WP1.1 partners to estimate possibilities for industrial up-scaling.

Phase 4: industrial up-scaling of the most promising concepts developed during Phase 3, according to the guidelines proposed in the Material Industrialization Concepts prepared at the end of Phase 3:
– Delivery of Ti-doped and reference undoped samples (graphite and CFC) for compound manufacturing in SP4 (all partners)
– Optimization of properties of most promising doped graphite and CFC concepts, and continue sample exchange with WP1.4 (all partners)
– To evaluate the capability for up-scaling of the most promising materials and processing paths for industrial production (all partners under the guide of SGL).

2.1.1.2 Results

In **Phase 1** the following User Requirements Specification (URS) involving carbon-based materials were identified:
- URS on Self-Passivating Protection Materials for Air breathing Propulsion (EXM-SP1-SPE-EAD-0002)
- URS for Plasma Facing Compounds (EXM-SP4-SPE-ARI-0005)
- URS for C-based Protective Materials (used in PFCs for Nuclear Fusion Devices) (EXM-SP1-SPE-ARI-0012)
- URS for Advanced Fission Applications (EXM-SP1-SPE-FAF-0001)
- URS for Brakes on Aircraft and High Speed Racing Vehicles (EXM-SP1-SPE-MER-0001)
- URS for High Temperature Lubricants (EXM-SP1-SPE-MAN-0001)

These URSs resulted in the elaboration of Material Requirements Specification (MRS) for Carbon-based Materials. It was decided to concentrate the future R&D work on the applications related to the ExtreMat main topical groups, i.e. C-based plasma facing materials for the strike point area of the ITER divertor (TG4), structural elements of the reactor core and control rods of a VHTR (TG3) and thrusters chambers in reusable air breathing propulsion systems (TG2). For the spin-off applications (Brakes on Aircraft and High Speed Racing Vehicles, and High Temperature Lubricants) it is expected that the material development performed for the 3 mentioned applications will result also in useful materials.

Furthermore, reviews on the state-of-the-art were prepared by all partners.

During **Phase 2** a preliminary list of Alternative Concepts (AC) for materials development, meeting the identified Material Requirements Specifications was prepared. According to these ACs, appropriate dopants were identified, different carbon raw materials for graphite production were selected and first experiments on the processing of doped isotropic graphites starting from different carbon raw materials and different nm-sized dopant powders were performed at CEIT, while UALI explored two approaches to obtain self-sintering doped precursor (co-pyrolysis of petroleum residues with a metal containing precursor, and co-pyrolysis of petroleum residues with dispersed metal carbide nanoparticles). INCAR identified mesophase pitches as precursors for CFCs with improved thermal conductivity and developed a method to produce mesophase pitches with potential to be converted into a continuous process. SGL prepared samples (2D CFC) for infiltration trials at INCAR and provided fine grained carbon raw material ready to press to CEIT.

At the end of Phase 2 the list of AC was updated and afterwards evaluated and validated by the Scientific Industrial Committee (SIC). These Alternative Concepts Proposals were the basis for the R&D work to be performed during Phase 3. The MRS for Carbon-based Materials were updated including the results of this evaluation/validation.

During **Phase 3** different methods to obtain doped isotropic graphites and CFCs with nanoscopic dopant dispersion and required properties for the envisaged applications were developed and optimized. Ti-, Si- and Zr-doped graphite samples, Ti- and Si-doped 2D-CFC, undoped 3D-CFC as well as C-SiC-B_4C composites were produced, characterized and sent to WP1.4 partners for further tests/characterization. Samples for neutron irradiation were manufactured and sent to NRG (SP3).

During **Phase 4** optimization of the most promising methods to obtain doped isotropic graphites and CFCs with nanoscopic dopant dispersion and required properties was performed in view of their industialization. Larger samples of optimized Ti-doped graphites and Ti-doped 3D-CFCs were sent to ARI for the manufacturing of plasma facing mock-ups within SP4; high heat flux tests were performed on these components at FZJ. Steps towards industrialization were done.

The main results obtained at WP1.1 during the whole project can be summarized as follows:

Ti-doped isotropic graphites (4 at.%Ti) using AR as raw material were manufactured and optimized for fusion application by CEIT and UALI. Typical properties of the graphites prepared by CEIT were: total porosity ≈ 10% (all closed), flexural strength ≈ 90-110 MPa, E ≈ 11-12 GPa, thermal conductivity at RT 200-220 W/mK. The carbide distribution was homogeneous. The Ti-doped isotropic graphites (4 at.%Ti) from UALI were produced using self-sintering doped mesophase produced by co-pyrolysis of petroleum residues with a dopant precursor; best results were achieved by mixing dispersed TiC nanoparticles in the petroleum residue. Typical properties were: open porosity 2%, bending strength 75 MPa and thermal conductivity at RT 180 W/mK. On these materials a reduction of chemical erosion by hydrogen down to targeted level was achieved (reduction from 8% to 2% for 30 eV D at 630 K; from 18% to 6% for 200 eV D at 820 K). The main mechanism for this reduction was TiC enrichment at the surface due to preferential erosion of C. A smooth and homogeneous TiC-enriched surface network was obtained protecting the underlying carbon from further erosion. High heat flux (HHF) tests of these materials under disruption relevant conditions with an energetic electron beam at FZJ resulted in significant reduction of particle emission and macroscopic damage compared to pure C, no cracks deep into the material. Brazing tests of these Ti-doped graphites to a CuCrZr block was successfully performed at ARI using a Mo interlayer (similar CTE than C). CEIT prepared 8 samples (27×20×5 mm) for the manufacturing of plasma-facing mock-ups and subsequent HHF cyclic testing within SP4. One of these mock-ups survived 100 cycles at 20 MW/m^2 on one tile plus 2 additional cycles at 21.8 MW/m^2, after which no total failure of the tile occurred. The other tiles withstood screening or several cycles at 20 MW/m^2 before failure, which was due to complete detachment or to detachment plus cracking. The reason for the better behaviour of the first tile is attributed to its higher thermal conductivity (222 W/mK) together with acceptable strength (σ_f 85 – 95 MPa). 10 samples of dimensions 27×20×7 mm were prepared also by UALI for the manufacturing of 2 plasma-facing mock-ups and subsequent HHF cyclic testing within SP4. The results were also satisfactory, surviving 100 cycles at 15 MW/m^2 and screening at 20 MW/m^2. These results demonstrate that Ti-doped graphites are promising armour materials for ITER, able to compete with present (undoped) CFC candidate materials.

Zr-doped graphites were also developed by CEIT and UALI using similar techniques. The results were satisfactory but the performance was lower than the one obtained for Ti-doped graphites.

Ti-doped 3D CFCs were developed by INCAR mainly for fusion applications. Best results were obtained with a new carbon fibre preform supplied by SGL within the last project year. Up to four densification cycles were performed to reduce the porosity of the resultant materials (~17 vol. %). The dopant was fairly well dispersed in the material (mainly nanometric size). The thermal conductivity at RT was slightly below 300 W/mK in one direction. On these materials also chemical erosion measurements, HHF tests and brazing tests to CuCrZr were performed. The reduction of chemical erosions is noticeable but less than the one observed for the Ti-doped graphites due to the lower Ti-content (0.6 to 1.7 at.% Ti). Samples of dimensions 27×20×7 mm were also prepared for the manufacturing of 3 plasma-facing mock-ups and subsequent HHF cyclic testing within SP4. The results were also satisfactory, surviving 100 cycles at 15 MW/m^2 and several cycles at 20 MW/m^2. On the mock-up prepared with the material with the highest thermal conductivity testing was only stopped due to a malfunction of the clamping mechanism to the cooling structure, but no visible failure was appreciated after 62 cycles at 20 MW/m^2.

Steps towards industrialization of Ti-doped graphite was performed by CEIT, upscaling the manufacturing process to samples of significantly larger size (60 x 60 x 15 mm, i.e. at the limit of CEIT's facilities) to check the possibilities for industrial production. The intended size would permit the manufacture of a plasma-facing actively cooled MU with monoblock geometry. The task could not be finisched since no total reproducibility of results compared to smaller samples could be achieved during the remaining time.

C-SiC-B_4C composites were developed by CEIT and UALI for space applications. CEIT used SGL 1 μm C-powder as raw material, achieving following typical properties: P_{tot} 24-31%, σ_f 110-150 MPa, E ~33 GPa, K 31-60 W/mK. Good oxidation resistance was found up to 1400°C for 10 h in air, as well as after 10 heating and cooling cycles with and without 1 h dwell at 1400°C. High heat flux tests with an energetic electron beam were performed at FZJ on these materials, and the threshold for particle emission was ~ 2 MJ/m^2 (0.4 GW/m^2 for 5 ms). Re-entry tests were performed by ARCS on this material, showing that burn-off did not start until 1400°C, and at 1500°C lifetime is limited to ~10 cycles. HfO_2 coatings were deposited on this material by DEMOKRITOS within WP1.2. The corresponding re-entry tests represented a tremendous improvement of oxidation resistance, demonstrating that HfO_2 on this composite provides an excellent barrier against oxidation at high temperatures (up to ~ 1500 ºC).

2.1.1.3 Deviations from the work programme, and corrective actions taken

There were no major deviations from the work programme.

2.1.1.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.1.2 WP1.2: SiC-based Materials

2.1.2.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
POLITO	SiC based protective coatings				101,0
DEMOKRITOS	Liquid Silicon Infiltration of Short Fibre C-SiC Composites				53,8
EADS	Multilayered SiC and SiC/oxide synthesis and industrial upscaling				12,2
FN	Long-fibre SiC/SiC composites and industrial up-scaling				12,9
MTA	Multilayered SiC and SiC/oxides ceramics with oxidation resistance				21,6

Objectives

Main: Development of SiC-based protection materials for application in extreme environments

Objectives of **Phase 1**:
- To identify real needs of end-users for SiC-based materials; to define materials requirements for the envisaged applications.
- To define R&D concepts for Phase 2 according to the material requirement specifications (MRS) in view of selecting the most promising R&D concepts at the end of Phase 2.

Objectives of **Phase 2**: to select the most promising SiC-based materials concepts according to the MRS identified in Phase 1, and to define the R&D activities for Phase 3 taking into account the risk involved, the resources requirements and the schedule.

Objectives of **Phase 3**: to develop the SiC-based materials concepts selected in Phase 2 and to exchange samples with other partners (mainly from WP1.4) to tests the developed materials under the conditions required by the applications. The concepts will be evaluated by the SIC and validated/rejected during and after each of the two SP1 meetings planned for this period.

Objectives of **Phase 4**: industrial up-scaling of the most promising concepts developed during Phase 3, according to the guidelines proposed in the Material Industrialization Concepts prepared at the end of Phase 3. As previously, the concepts shall undergone the SIC evaluation and be validated/rejected during and after each of the two SP1 meetings planned for this period.

Description of work

Phase 1: provision of User Requirements Specifications (URS) on thermal protection systems for space vehicles (POLITO), on air breathing propulsion systems (EADS) and on CMC for different applications (fusion, aerospace, high temperature lubricants etc.) (MTA); reviews on the state-of-the-art of different SiC-based materials systems; proposal of R&D concepts for Phase 2 in view of selecting out of them the most promising R&D concepts at the end of Phase 2; evaluation and validation of the URS and of the MRS of SiC-based materials related to aerospace applications (POLITO, MTA, EADS and FN).

Phase 2: definition of selection criteria for alternative concept proposals on SiC-based materials and selection of proposals for SiC-based material concepts. Evaluation/validation of the alternative concept list by the partners acting as SIC members. At the end of Phase 2, revision of both MRS and alternative concept list, and contribution to the planning of Phase 3. The R&D work during Phase 2 includes:
- First laboratory experiments on processing of SiC-based multilayer ceramics (POLITO).
- First experiments on CVI processing of short fibre SiC/C_f composites (EADS).
- First experiments on processing and characterization of dense 2D and 3D SiC/SiC_f composites (MTA).
- Experimental verification of the production of tailored protective SiC layers by magnetron sputtering (DEMOKRITOS).

It was decided not to follow the line of SiC-based composites synthesized by SHS proposed by DEMOKRITOS because during Phase 1 it was realized that these materials did not comply with the MRS.

Phase 3: start of R&D work on the alternative concepts proposed during Phase 2 to demonstrate their compliance with the Materials Requirements Specifications (MRS):

– Processing of multilayer ceramics with different composition and architecture (POLITO, FN).
– Processing by liquid silicon infiltration of C_f/SiC composites with improved performance (EADS).
– Processing by CVI of 2D and 3D long fibres SiC_f/SiC composites with improved performance (MTA).
– Development of a sputtered graded multilayer coating based on SiC, suitable for protection of materials (DEMOKRITOS).

Phase 4: industrial up-scaling of most promising material concepts developed during Phase 3, as proposed in the Material Industrialization Concepts (MIC) prepared at the end of Phase 3:

– Industrial processing of SiC-multilayer by tape-casting (FN, POLITO).
– Verification of deposition method of protection/lubricant coatings (by slurry, CVD) with respect to industrialization of process. Final testing of coating behaviour (ageing, thermo-shock) (MTA).
– Processing of plane C_f/SiC specimens with oriented short fibre for brake or aerospace applications. Definition of design criteria for large scale sample; manufacture and testing of this sample (EADS).
– Sputter deposition of SiC and HfO_2 based coatings on SiC or C based materials from WP 1.2 and WP1.1 partners (DEMOKRITOS).

2.1.2.2 Results

In **Phase 1** the following User Requirements Specification (URS) involving SiC-based materials were identified:

- URS for Thermal Protection Systems for Space Vehicles (EXM-SP1-SPE-POL-0001)
- URS for Satellite Propulsion (EXM-SP1-SPE-EAD-0001)
- URS for Air breathing Propulsion (EXM-SP1-SPE-EAD-0002)
- URS for Oxidation- & Abrasion Resistant Coatings on CMC (EXM-SP1-SPE-MAN-0005)
- URS for Advanced Fission Applications (EXM-SP1-SPE-FAF-0001)
- URS for CMC for Fusion Application (EXM-SP1-SPE-MAN-0002)
- URS for Gas Turbines Combustion Chambers, Flame Holders and Vanes (EXM-SP4-SPE-ARI-0008)
- URS for Brakes on High Speed Rail Vehicles (EXM-SP1-SPE-MER-0002)
- URS for High Temperature Lubricants (EXM-SP1-SPE-MAN-0001)
- URS for Hydrogen Generation (EXM-SP1-SPE-NNC-0002)

These URSs resulted in the elaboration of Material Requirements Specification (MRS) for SiC-based Materials. These requirements are defined with reference to the following applications envisaged for SiC-based materials: Thermal Protection Systems for Space Vehicles (TG2); Self-Passivating Protection Materials for Satellite Propulsion (TG2); Self-Passivating Protection Materials for Air-breathing Propulsion (TG2); Oxidation and Abrasion Resistant Coatings for CMC (SiC-based) (TG2); Very High Temperature Reactor (VHTR) (TG3); CMC materials for fusion blankets (TG4); Gas Turbines Combustion Chambers, Flame Holders and Vanes (TG5); Brakes on High Speed Rail Vehicles (TG5); High Temperature Lubricants (TG5).

Furthermore, reviews on the state-of-the-art were prepared by all partners.

During **Phase 2** a preliminary list of Alternative Concepts (AC) for materials development meeting the identified MRS was prepared. According to these AC, laboratory tests were performed at POLITO to develop more complex architectures for SiC-based multilayer ceramics and evaluated their feasibility, and processed also multilayer ceramics based on Al_2O_3, $MoSi_2$ and ZrO_2. EADS manufactured first short fibre C_f/SiC composites by Liquid

Silicon Infiltration (LSI) using a pyro-carbon CVD coating to protect the fibres against Si attack and demonstrated the concept of in-situ forming protective coatings. MTA: produced CMC material based on crystalline SiC-fiber and crystalline SiC-matrix produced by CVI using different 2D and 3D SiC fibrous fabrics with and without various fiber coatings as well as various SiC matrix infiltration processes. DEMOKRITOS performed a detailed literature review on SiC-based protective layers produced by magnetron sputtering, and verificated experimentally their production, taylored for the specific applications.

At the end of Phase 2 the list of AC was updated and afterwards evaluated and validated by the members of the SIC. These AC Proposals are the basis for the R&D work to be performed during Phase 3. The MRS for SiC-based Materials were updated including the results of this evaluation/validation.

During **Phase 3** SiC-based multilayer ceramics with different composition and architecture were processed and characterized. For the most promising concepts the processing way was further optimized and the definitive method for processing multilayer SiC (containing only dense layers) was transferred from POLITO to FN for industrialization. Short fibre C_f/SiC composites with improved performance were manufactured by EADS by liquid silicon infiltration and the processing optimized mainly by targeted orientation of the fibres aiming mainly at increasing the bending strength. 2D and 3D long fibres SiC_f/SiC composites were manufactured at MTA by CVI and the process optimized. In addition, CVD-based high temperature SiC-lubricant coatings for CMCs (C/SiC and SiC/SiC) bearings were developed and optimized for application in reentry space vehicles. Sputtered graded multilayer protective coatings based on SiC and HfO_2 have been developed. Samples of most of the developed materials were sent to WP1.4 partners for further test and characterization. In addition, sample delivery to NRG (SP3) for the neutron irradiation campaign was finished.

During **Phase 4** SiC-based multilayer ceramics with different composition and architecture were optimized by POLITO. The industrialization of the whole process was successfully performed by FN. POLITO proved that an Y_2O_3-based surface coating was effective for the protection of multilayer SiC from oxidation/corrosion at high temperature under O_2/H_2O atmosphere. EADS optimized the processing of short fibre C_f/SiC composites by liquid silicon infiltration (LSI), increasing mechanical strength (3PB) up to 140 MPa by short fibre orientation; this value is nevertheless still too low for space applications. Thus, a component for brake application was designed, manufactured and sent to MERL (WP1.4) for testing. MTA optimized CVD-based high temperature SiC-lubricant coatings for CMCs (C/SiC and SiC/SiC) bearings for application in reentry space vehicles, and coated complex shaped ceramic component were successfully tested under simulated re-entry mission like conditions. For the CVD coating process applied for bearing lubricants, an industrial processing level could be reached. DEMOKRITOS optimized sputtered graded multilayer protective coatings based on SiC and HfO_2. Oxidations and re-entry tests of several HfO_2 coated SP1 materials resulted in significant improvement of performance.

The main results obtained at WP1.2 during the whole project can be summarized as follows:

SiC-multilayers developed by POLITO and sucdesfully industrialized by FN proved to fulfill the material requirements for self passivating Thermal protection Systems (TPS) of re-entry vehicles in terms of density, hardness, strength and modulus in the temperature range of the application, thermal expansion, thermal stability, self-passivating behaviour, thermal shock resistance and capability of sustaining high heat flux. Such a material survived 100 re-entry simulations. Failure mechanism was found to be different from that displayed by conventional ceramics owing to delamination phenomena involving energy dissipation. The integration of porous layers within the multilayer architecture was found to enhance the delamination phenomena and to decrease the through-thickness thermal conductivity. This material represents a cheap option with respect to conventional currently used CMCs containing long fibres. The same material coated with a Y_2O_3-SiO_2 layer is also promising for turbine engine application.

C_f/SiC composite material with oriented short fibres was developed by EADS. Different fibre orientation techniques were investigated aiming at improving the mechanical properties for air-breathing propulsion application. Fibre orientation by Fibre-Patch-Preforming (FPP) was discarded, while the carding technique provided satisfactory results. The obtained composite material mainly contained C fibres and SiC as well as very small amounts of free Si contents (< 2 %). The density of the material was 2.25 g/cm^3, showing homogenous microstructure with low porosity. The mechanical strength (3PB) of the LSI - C_f/SiC with oriented short fibre could be increased up to 140 MPa, which is nevertheless still too low for space applications. Therefore, the spin-off application ‘brakes for high speed rail vehicles, race cars and aircraft’ was selected since the developed material seemed very promising for this application. 6 up-scaled brake discs with 75 mm diameter and 10 mm thickness were manufactured for friction and wear tests at MERL. The used composite material was the optimized short carbon fibre C_f/SiC material of 2nd generation without targeted short fibre orientation. Furthermore, 2 old disc samples were added for the planned test friction and wear test campaign for comparison. One brake disc of each material quality included an oxidation protection coating. The friction and wear tests performed on these discs gave very promising results, showing a higher friction coefficient than that of the commercially available C_f/SiC material and a lower wear.

CVD SiC-based lubricant coating on C/SiC bearing components and SiC/SiC parts were successfully developed by MTA. Complex shaped rudder like ca. 450x250 mm ceramic component was coated and tested under simulated re-entry mission like conditions up to 27 missions at 1550 °C with > 22 000 movements and under a load of 7.3 kN. The CVD coating process applied for bearing lubricants was verified to be reproducible for complex shaped as well as simple shaped parts (like flat panels or tubes), independent from the dimension of the components, as far as there is no limitation of the CVD inner furnace chamber. Therefore, an industrial processing level could be reached. This development could directly be taken as basis for a very promising and future relevant re-entry space vehicle, basis of the FLPP (Future Launcher Preparatory Program) space project, managed by ESA (European Space Agency) and implemented into the planning and establishment of a hot re-entry mission simulating test campaign with a CMC control surface with ceramic bearing elements.

HfO_2 layers of thickness varying between 5 and 15 µm on SiC developed by DEMOKRITOS demonstrated to be an attractive way to significantly improve the oxidation resistance of SiC. Oxidization tests were performed at FZJ in air in the temperature range 1100 to 1450°C. During oxidization re-crystallization of HfO_2 takes place and $HfSiO_4$ is formed, enhancing the bonding with the SiC substrate and further increasing oxidization resistance. Re-entry tests up to 1400°C for up to 100 cycles were performed at AIT, showing that the coating remains intact and no oxidization of the SiC substrate is observed. In addition, re-entry tests up to 1550°C performed on HfO_2 coated C/SiC/B_4C composites (supplied by CEIT) showed significant improvement of oxidization resistance performance compared to uncoated samples.

2.1.2.3 Deviations from the work programme, and corrective actions taken

During Phase 2 the SHS technique proposed by DEMOKRITOS was proven not to be applicable for the intended applications. For this reason this approach was discarded and the sputtering techniques were adopted by DEMOKRITOS for the production of tailored protective coatings.

2.1.2.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.1.3 *WP1.3: Refractory based Materials*

2.1.3.1 *WP overview on objectives, involved partners, planned work and manpower*

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
EADS	Processing of bulk CrRe alloys				19,6
ATL	Industrial upscaling of nanodispersed doped CVD coatings on W-base				22,8
IPP	Atomic synthesis of W-based alloy coatings, modelling of reactivity				38,7
PLANSEE	Manufacture of actively cooled stainless steel samples with thick W-VPS coating for irradiation testing				6,0
WUT	Nanoscopic characterization and modelling of W-based and Cr-Re alloys				41,0

Objectives

Main: Development of self-passivating refractory based protection materials.

Objectives of **Phase 1**:
- To identify the real needs of end-users for refractory-based materials and to define the requirements the materials have to fulfill for the envisaged applications.
- To define R&D concepts for Phase 2 according to the material requirement specifications (MRS) in view of selecting the most promising R&D concepts at the end of Phase 2.

Objectives of **Phase 2**: to select the most promising refractory-based materials concepts according to the MRS identified in Phase 1, and to define the R&D activities for Phase 3 taking into account the risk involved, the resources requirements and the schedule.

Objectives of **Phase 3:** to develop the refractory-based materials concepts selected in Phase 2 and to exchange samples with other partners (mainly from WP1.4) to tests the developed materials under the conditions required by the applications. The concepts will be evaluated by the SIC and validated/rejected during and after each of the two SP1 meetings planned for this period.

Objectives of **Phase 4:** industrial up-scaling of the most promising concepts developed during the previous R&D (Phase 3), according to the guidelines proposed in the Material Industrialization Concepts prepared at the end of Phase 3. As previously, the concepts shall undergone the SIC evaluation and be validated/rejected during and after each of the two SP1 meetings planned for this period.

Description of work

Phase 1: provision of User Requirements Specifications (URSs) on self-passivating protection materials for satellite propulsion (EADS), and on W-based plasma facing protection materials for the first wall of fusion reactors (IPP); review on the state-of-the-art of the different material systems and identification of requirements for nanoscopic characterization and modelling; proposal of R&D concepts for Phase 2 in view of selecting out of them the most promising R&D concepts at the end of Phase 2; evaluation and validation of the URSs and of the MRS of self-passivating refractory-based materials (IPP, EADS and PLANSEE).

Phase 2: definition of selection criteria for alternative concept proposals on refractory-based materials and selection of proposals for materials concepts. Evaluation/validation of the alternative concept list by the partners acting as SIC members. At the end of Phase 2, revision of both MRS and alternative concept list, and contribution to the planning of Phase 3. The R&D work during Phase 2 includes:
- First experiments on Cr-Re processing by different techniques (EADS).
- Evaluation of feasibility of W-alloy sputter-deposition and their oxidation behaviour measurements (IPP).
- Evaluation of viability of potential routes for production of required materials by CVD (ATL).
- Investigation of W-VPS coating on CFC bulk material with a layer thickness ~200µm (PLANSEE).
- Evaluation of modelling capabilities at macro-, micro- and atomic level for the required materials (WUT).

Phase 3: start of R&D work on the alternative concepts proposed during Phase 2 to demonstrate their compliance with the Materials Requirements Specifications (MRS:
– Processing and characterization of CrRe material by different techniques (EADS).
– Thin film deposition of W-Si and ternary W-based alloys and oxidation experiments (IPP).
– Development of CVD coatings: W-based on steel, Al and Si based on CrRe (ATL).
– Microstructural and analytical characterisation of CrRe bulk materials and W-based thin films. Modelling of diffusion process and oxidation resistance of W-based material (WUT).

Phase 4: industrial up-scaling of the most promising material concepts, as proposed in the Material Industrialization Concepts (MIC) prepared at the end of Phase 3:
– Evaluation of synthesis methods for thick film deposition and bulk material for fusion application in view of industrialization of the thin film W-based alloys. Thin film deposition of quaternary W-based alloys and investigation of their oxidation behaviour (IPP).
– Coating of components with CVD W for interface testing and analysis at IPP and WUT (ATL). If successful, design of an increased size furnace for producing thick W coatings.
– Microstructural and analytical characterization of optimised coatings and joints for fusion applications; modelling of oxidation resistance of WSi-based materials at relevant temperature range (WUT)

2.1.3.2 Results

In **Phase 1** the following User Requirements Specification (URS) involving refractory-based materials were identified:
- URS on Self-Passivating Protection Materials for Satellite Propulsion (EXM-SP1-SPE-EAD-0001)
- URS for W-based Plasma Facing Protection Materials for the First Wall of Fusion Reactors (EXM-SP1-SPE-IPP-0001)
- URS for Plasma Facing Compounds (EXM-SP4-SPE-ARI-0005)

These URSs resulted in the elaboration of Material Requirements Specification (MRS) for self passivating refractory-based materials. These requirements are defined with reference to the following envisaged applications: Future generation satellite thrusters applied in satellite propulsion systems (TG2); Tungsten-based plasma facing material for the divertor and main chamber of ITER and future fusion reactor concepts (TG4).

Furthermore, reviews on the state-of-the-art were prepared by all partners.

During **Phase 2** a preliminary list of Alternative Concepts (AC) for materials development meeting the identified MRS was prepared. According to these AC, a pre-selection of the 3 most important concepts related to Cr-Re processed by induction melting (based material, coating, high temperature forging) and 2 high potential but high risk concepts (nanostructuring, additional diffusion of the coating) was performed by EADS. IPP demonstrated the feasibility of W-Si and W-X_1-X_2 preparation by magnetron sputtering and the possibility of measuring their oxidation behaviour by thermobalance. ATL deposited thick CVD pure W coatings on Eurofer and CFC substrates; routes to CVD W and W_xSi_y with additional elements such as Er, Ni, Y, Cr and Al were investigated by literature search and thermodynamic calculation; CrRe samples were aluminised at two different CVD conditions. PLANSEE investigated W-VPS coating on CFC bulk material with a layer thickness in the range of 200µm by different destructive examinations. WUT analized the modelling possibilities of oxidation resistance of W-based alloys, ab initio modelling of CrRe alloys, nanostability of Cr-Re alloys and aluminizing process of Cr-Re alloy.

At the end of Phase 2 the list of AC was updated and afterwards evaluated and validated by the members of the SIC. These AC Proposals are the basis for the R&D work to be performed during Phase 3. The MRS for self-passivating refractory-based Materials were updated including the results of this evaluation/ validation.

During **Phase 3** CrRe alloys were investigated by EADS concerning hot forming, machinability, weldability and high temperature resistance under vacuum. The results led to the conclusion that CrRe35 is a promising concept but has some specific weakness which affects key properties. Further investigations would be necessary to optimize the material, which were not feasible within the current time and budget frame. Therefore, it was proposed to stop the CrRe activities in month 30 (May 2007) and to shift the remaining time and budget to WP2.1. Thin film deposition of the ternary W-based compounds W-Si-Ni and W-Si-Zr were performed at IPP and their oxidation resistance tested. Even though W-Si-Ni and W-Si-Zr showed higher oxidation rates than W-Si-Cr, cracks during heating, oxidation and cooling down were strongly reduced using Ni or Zr additives. Thus, alloys of W-Si-Cr-Zr were proposed for further investigations during Phase 4. A new batch of thick CVD W coatings on Eurofer steel substrates prepared by ATL delaminated again due to thermal mismatch, despite modelling and re-engineering of the interface region. The source of this problem could not be identified and this investigation was shifted to Phase 4. Modelling of the oxidation process of W-36Si-20Cr and W-36Si-20Cr-11Zr systems was done by WUT using the experimentally obtained oxidation rate constant. Furthermore, WUT performed very useful nanostructural characterisation of W-Si and W-Si-Cr coatings before and after oxidation, of W/Cu/Eurofer steel joints, and of CrRe samples after different aluminising processes followed by oxidation.

During **Phase 4** thin film deposition of the quaternary W-based compounds W-Si-Cr-Zr was started. Compared to the best results of ternary alloys, the oxidation rate of this alloy was further decreased. Possible processes to manufacture these alloys at industrial scale were identified. A new batch of thick CVD W coatings involving CVD erbia as tritium permeation barrier was produced. Copper as a stress absorbing interlayer was deposited, but there was insufficient time to complete these samples due to delays in their production. This will be done within the next six months. Modelling of the oxidation process of W-36Si-20Cr and W-36Si-20Cr-11Zr systems has been done using the experimentally obtained oxidation rate constants. The concentration profiles of W, Si, Cr in the alloy during oxidation at 1000°C were calculated.

The main results obtained at WP1.3 during the whole project can be summarized as follows:

To investigate the self-passivating properties of binary and ternary W-alloys, deposition and oxidation experiments were carried out at IPP on pure W, the binary alloys W-Si and W-Cr, and the ternary alloys W-Al-Cr, W-Si-Al, W-Si-Cr, W-Si-Ni, W-Si-Y, and W-Si-Zr. The fraction of the used components was varied within the investigated systems. The performance of the ternary alloys was always higher than the one of binary systems. Si was identified as an essential component of ternary alloys. Best results were found using Cr or Y as third alloying component in the ternary system. At lower temperatures the performance of Y additions was higher than the one of Cr, but at 1000 °C Cr containing alloys showed the best self-passivating properties. Quaternary alloys W-Si-Cr-Zr and W-Si-Cr-Y were also investigated resulting in better passivation behaviour than ternary alloys while containing more W. There is a two step-oxidation: it seems that different oxidation mechanisms are rate determining at different temperatures. Different oxide phases are formed, but no WO_3 which means that the passivation is successful. The surface oxide consists of Cr_2O_3. Since the development was carried out on thin films as model for thick coatings or bulk material for fusion first wall application, other manufacturing processes have to be identified to produce such materials. As most promising system the ternary alloy WSi10Cr10 (in wt.%) was identified and should be used basically for industrialization activities.

The oxidation process of WSi36Cr20 and WSi36Cr20Zr11 alloys was modelled by WUT using calculated values of diffusion coefficients and experimentally obtained oxidation rate constant. The concentration profiles of W, Si, Cr and Zr in the alloys during oxidation at 1000°C for 22h were calculated. The results obtained were in a good agreement with experimental results.

Thick CVD W coatings for fusion application were deposited on Eurofer by ATL using a Cu interphase deposited by magnetron sputtering (IPP) or/and electroplating on Eurofer substrates. Also thin layers of erbia, directly deposited on the Eurofer substrate or on the Cu interface, were tested. After the CVD deposition of the thick W coatings on theses samples the system showed always interfacial delamination. Therefore this activity was stopped during Phase 4. It was concluded that the successful erbia permeation barriers would be better used in areas that did not require a tungsten overlayer. This might include tritium breeder modules where the layer is in contact with the PbLi breeder material, or other applications where hydrogen and deuterium are used such as the hydrogen generation industry.

EADS developed of a suitable production technology for CrRe and measured the resulting material properties in order to replace costly platinum alloys for satellite thrusters and to evaluate the envisaged suitability of the new material for fusion application. Induction and arc molten CrRe based alloys were produced achieving very comparable properties. At 1200°C the ductility of this material dropped to zero after reaching a maximum at 1000°C. Exposure to thermal shock resulted in limited cracking on CrRe35 samples and severe cracking on CrRe18 samples. The hot forming, machinability, weldability and high temperature resistance under vacuum of CrRe alloys was investigated. It was found that the CrRe35 material is very interesting with promising properties, but some specific weakness which affects key properties. Thus, the CrRe activities were stopped after month 30.

W-VPS coatings were deposited by PLANSEE on CFC bulk material using spraying parameters which result in a density of about 90%. Several samples were manufactured for the neutron irradiation campaign. This activity stopped at the end of Phase 2, as planned.

2.1.3.3 Deviations from the work programme, and corrective actions taken

During Phase 3 it was realized that the CrRe35 material concept proposed by EADS exhibitsed a pronounced loss of ductility in static tensile testing at temperatures well above 1000°C. In addition, there was a distinct weight loss at temperatures >1600°C with a risk of surface cracks. Finally, the low strength and brittle failure behavior after welding as well as the difficult machining completes the picture of a very interesting material with promising properties but some specific weakness, which affects key properties. Thus, it would not be sufficient to start an industrialization phase after month 36. Further investigations would be necessary to optimize the material, which are not feasible within the current time and budget frame. As a solution, it was proposed to stop the CrRe activities at month 30 and to shift the remaining time and budget to WP2.1 to develop an aerospace demonstrator using the high thermal conductivity material already developed in this WP. The activities of the other WP1.3 partners related to this concept were also stopped.

CVD W coatings on Eurofer steel substrates delaminated due to thermal mismatch. The source of this problem remained unidentified also during Phase 4, and there was insufficient time to complete these samples due to delays in their production.

2.1.3.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.1.4 WP1.4: Environmental Tests and Industrial Evaluation

2.1.4.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
NNC	Industrial evaluation of fission and spin-off hydrogen generation applications				**10,0**
AEN	Evaluation of C- and SiC-based materials for fusion and gas turbine applications				11,5
AIT	Tests in the Re-Entry Chamber				17,7
EA	Nanostructure characterization of SP1 materials by neutron and X-ray techniques				6,4
AREVA-Fr	Industrial evaluation of fission and hydrogen generation (spin-off) applications				7,9
DEMOKRITOS	Industrial evaluation of materials for advanced fission applications				34,7
FZJ	High heat flux/thermal shock and off normal oxidation tests				42,5
IPP	Ion beam, plasma exposure tests: chemical erosion by H atoms/ions				46,3
JRC	Interfacial Toughness Assessment of Tungsten-Armoured Eurofer Steel				21,2
MERL	Friction/wear tests for brake applications (spin-off). Industrial evaluation of brake applications (spin-off)				22,3

Objectives

Main: To perform environmental tests and specific characterizations as well as to provide industrial evaluation of materials and concepts from the other WPs of SP1.

Objectives of **Phase 1**: To identify the real needs of end-users for SP1 materials and to define the requirements the materials have to fulfill for the envisaged applications. To explore the potential for application of materials from WP1.1-1.3 with existing and evolving industrial systems. To define the application specific environmental tests requirements, and their harmonization with existing codes.

Objectives of **Phase 2**: Experiments to verify the required environmental test parameters, characterization methodology, and adequacy of modelling tools to evaluate experiments and to predict operational behaviour of materials. Evaluation/validation of the alternative concepts proposed by WP1.1-1.3 by the partners acting as SIC members, and definition of the tests and characterization activities for Phase 3.

Objectives of **Phase 3**: to provide industrial input to the chosen SP1 material investigations and advise on practical and industrial requirements for the selected applications. To investigate potential spin-off applications. To perform environmental tests and nanostructural characterization on materials from WPs 1.1-1.3 for their evaluation w.r.t. the envisaged applications.

Objectives of **Phase 4**: to provide industrial input to the chosen SP1 material investigations and advise on practical and industrial requirements for the selected applications. To investigate potential spin-off applications. To perform environmental tests and nanostructural characterization on materials from WPs 1.1-1.3 for their evaluation w.r.t. the envisaged applications. To provide required information for the longer-term objectives of industrialisation

Description of work

Phase 1: provision of User Requirements Specifications (URSs) on plasma facing compounds (AEN), on SiC-based protective materials for application in gas turbines combustion chambers (ARI), on C-based Protective Materials used in PFCs for Nuclear Fusion Devices (AEN and IPP), on advanced fission applications (AREVA-F), on brakes on aircraft and high speed racing vehicles (MERL), on brakes on high speed rail vehicles (MERL), and on hydrogen generation (NNC); mapping of performance and parametric requirements for the testing of materials from WPs 1.1, 1.2, and 1.3 in the re-entry chamber from AIT; the same for off-normal oxidation tests and high heat flux tests at FZJ; the same for ion beam and plasma exposure under fusion conditions at IPP; the same for corrosion and thermo-mechanical tests at JRC; review on the applicability of different neutron and X-ray scattering techniques for the materials to be developed in SP1 (DEMOKRITOS)

Phase 2: list of environmental tests and especial characterization techniques available. Evaluation/validation of the AC of WPs 1.1, 1.2 and 1.3 by the partners acting as SIC members. At the end of Phase 2, revision of all MRS of SP1 and contribution to the planning of Phase 3. The testing and characterization work includes definition of the baselines for all subsequent testing and characterization activities and identification of required modifications in corresponding devices.

Phase 3 and 4: advice on practical and industrial requirements and issues of application including spin-offs, and provision of such input to the experimental investigations within SP1. The work related to the environmental tests will focus on the realization of adequate tests and characterization of materials from WPs 1.1-1.3, analysis of the results and discussion of them with materials providers for evaluation. The work includes:

– Evaluation of industrial implementation of chosen SP1 materials and technologies for advanced fission applications (NNC, EA, AREVA-F), fusion applications (AEN, IPP) and spin-off applications (AEN, MERL)
– Performance of erosion measurements on C-based and W-based materials (IPP)
– Performance of re-entry tests on SiC-, WSi- and coated CrRe-based materials (AIT)
– Performance of interfacial fracture toughness tests at RT and HT on VPS and CVD W-coatings (JRC)
– Performance of oxidation/high heat flux tests on C-, W- and SiC-based materials (FZJ)
– Performance of friction/wear tests on doped carbon, SiC/SiC_f and SiC/C_f materials (MERL)
– Performance of brazing tests on doped graphites and CFCs. Thermal shock tests on SiC multilayers (AEN)
– Nanostructure characterization of SP1 materials by neutron and X-ray techniques (DEMOKRITOS)

2.1.4.2 Results

In **Phase 1** the following User Requirements Specification (URS) involving protection materials for ExtreMat application were identified:

- URS for Plasma Facing Compounds (EXM-SP4-SPE-ARI-0005), prepared by AEN.
- URS for C-based Protective Materials (used in PFCs for Nuclear Fusion Devices) (EXM-SP1-SPE-ARI-0012) , prepared by AEN and IPP
- URS for Gas Turbines Combustion Chambers, Flame Holders and Vanes (EXM-SP4-SPE-ARI-0008), prepared by AEN
- URS for Brakes on Aircraft and High Speed Racing Vehicles (EXM-SP1-SPE-MER-0001), prepared by MERL
- URS for Brakes on High Speed Rail Vehicles (EXM-SP1-SPE-MER-0002), prepared by MERL
- URS for Advanced Fission Applications (EXM-SP1-SPE-FAF-0001), prepared by AREVA-F
- URS for Hydrogen Generation (EXM-SP1-SPE-NNC-0002), prepared by NNC
- These URSs resulted in the elaboration of Material Requirements Specification (MRS) for the corresponding family of materials.

Furthermore, a mapping of performance and parametric requirements for the testing of materials from WPs 1.1, 1.2, and 1.3 was performed for the following test facilities:

- AIT: re-entry simulation chamber.
- FZJ: high heat flux/thermal shock and off-normal oxidation tests facilities.
- IPP: ion beam exposure (high current ion source) and plasma exposure (ASDEX Upgrade) experiments.

- JRC: corrosion and thermo-mechanical tests facilities.

DEMOKRITOS prepared a review on the applicability of different neutron and X-ray scattering techniques for the materials to be developed in the other WPs from SP1.

During Phase 1 the partners of WP1.4 acting as SIC members (AEN, EA, AREVA-F, FZJ, IPP, and NNC) evaluated and validated the User Requirements Specifications and the Materials Requirement Specifications of SP1.

During **Phase 2** the partners involved in environmental testing prepared a table summarizing the testing and specific characterization capabilities of SP1, including type of test or technique, test conditions, kind and dimensions of samples, and field of application. The intention of this table is to serve as basic information for the exchange of samples for testing and characterization between all SP1 and ExtreMat partners. AIT identified test conditions for re-entry applications, selecting the German HOPPER study as reference. AEN performed brazing tests on 13 different types of already existing doped and non-doped graphite provided by CEIT from earlier work. DEMOKRITOS defined possible material characterization through scattering techniques, and performed initial preparation of the experimental campaigns at Large Scale Facilities FZJ performed first off-normal oxidation tests in the facilities THERA (I + II), INDEX and OZOX on C-based materials, as well as high heat flux tests on existing W and C-based materials in the electron beam test facilities “JUDITH” and “JUDITH II”. IPP fixed experimental parameters for ion beam exposure (high current ion source) and plasma exposure (Asdex Upgrade) and performed test measurements on pure graphite and on W_xSi_y layers; Monte Carlo codes for ion-solid interactions (TRIDYN, SDTRIM) were established and tested. JRC proposed a comprehensive R&D programme for the mechanical characterisation of the interfacial fracture toughness of bimetallic beams prepared from W-armoured Eurofer steel components for plasma facing applications. MERL performed tests on current leading brake industry materials that will serve as a reference for the materials generated by the SP1 partners.

At the end of Phase 2 the partners acting as SIC members (AEN, EA, AREVA-F, FZJ, IPP, and NNC) evaluated/validated the alternative concept lists of WPs 1.1, 1.2 and 1.3 according to the application corresponding to the Topical Group of their concern, and revised all MRS of SP1, which have been updated including the results of this evaluation/validation.

During **Phase 3** and **Phase 4** environmental tests and especial characterizations were performed on most of the SP1 materials developed within Phase 3, providing an essential contribution for the evaluation/validation of the proposed materials concepts. Erosion measurements at IPP on C-based materials by hydrogen bombardment; re-entry tests at AIT on SiC multilayers and short fibre C_f/SiC composites; interfacial fracture toughness tests at JRC on VPS W-coatings and mechanical testing of SiC multilayers; oxidation and high heat flux tests at FZJ on doped C-based materials, SiC multilayers and C-SiC-B_4C composites; friction/wear tests on doped C-materials, C_f/SiC and C-SiC-B_4C composites; brazing testsat AEN on doped graphites and CFCs; SANS measurements on doped graphites, as well as neutron reflectivity measurements at DEMOKRITOS on oxidized SiC coatings and on oxidized W-Si layers were performed.

The results of these tests and characterization on SP1 materials have been partially reported in the description of the results of the previous WPs. In the following, these results are summarized:

<u>Re-entry tests</u> were performed by AIT on different SP1 materials: SiC multilayer material manufactured by POLITO was identified as a promising candidate for highly demanding

thermal protection applications, both in aerospace and in various other fields of application. The EADS short fibre C_f/SiC material was also identified as a promising low-cost alternative for a number of applications, though aerospace may not be the prime application. SiC material coated with HfO_2 by DEMOKRITOS proved to be very stable against oxidation and survived 100 cycles at 1.450°C without major mass loss. C/SiC/B_4C composite materials from CEIT were tested at various temperatures, ranging from 1200°C to 1500°C. Burn-off started at 1400°C, and at 1500°C lifetime was limited to ~ 10 cycles. A combination of CEIT's materials with DEMOKRITOS-made HfO_2 coating showed a tremendous improvement of oxidation resistance: no mass loss at 1400°C, and the mass loss at 1500°C after 30 cycles was still below the threshold of 5%.

Brazing tests to pure Cu on doped and undoped graphites and CFC from WP1.1 were performed by AEN. Good wettability by the brazing alloy was verified in all cases, with the formation of a suitable reaction layer; in the case of CFC, the brazing alloy has been able impregnate the fibres near the CFC surface, achieving a potentially very good joint. Ti-doped graphites showed cracks and detachments when brazed to pure Cu, most likely due to excessive residual stresses. To avoid this, compounds of Ti-doped graphites from CEIT and UALI and of INCAR 3D CFC with CuCrZr blocks were manufactured introducing and a low CTE (Mo) interlayers, resulting in the absence of crack formation and very good thermal shock resistance. On the basis of these results, the following actively cooled plasma-facing mock-ups for fusion application were prepared and delivered to FZJ for HHF tests:

- 2 mock-ups with Ti-doped CEIT's graphite
- 2 mock-ups with TiC-particles and Ti-butoxide doped UALI's graphites respectively
- 2 mock-ups with undoped and doped 3D INCAR's CFC

Small Angle Neutron Scattering (SANS) and Ultra Small Angle Neutron Scattering (USANS) measurements were performed by DEMOKRITOS on graphite samples provided by CEIT, UALI and INCAR. The data gave information about open and closed porosity, pores size distribution, mean pore size and total porosity surface. These parameters were correlated with the fabrication process and the different dopants used in each case. The SANS measurements were accompanied by nitrogen porosimetry measurements for some of the samples and very good agreement was found between SANS and N2 porosimetry results.

Neutron reflectivity measurements were performed by DEMOKRITOS on oxidised SiC and W-Si coatings coatings for different oxidization conditions and times. The analysis of the data gave information about the density, thickness and roughness of the resulting layered structure

Residual stress measurements were performed by DEMOKRITOS using neutron diffraction on Mo/Cu, Mo/CuCrZr brazed tiles and free Mo blocks as well as on CuCrZr tube brazed with W tile, all provided by AEN. The residual stresses were determined in the three directions, characteristic for each sample geometry, in the side of Mo (for Mo/Cu and Mo/CuCrZr tiles) and in the side of W (for CuCrZr/W) at different dis-tances from the interface.

Off-normal oxidation tests were performed at FZJ on doped carbon based materials in air (most experiments at 700°C). The oxidation resistance of all materials was compared with that of a standard nuclear graphite (Sigri V483T5). It was shown that doping with Ti or Zr does not significantly improve the oxidation resistance. Experiments indicate that a highly oxidation protecting doping has to be applied in concentrations of some ten percent. SiC-multilayers were found to be very efficient in protection from oxidation (tested up to 1500°C in air). This holds also for multilayers containing Y_2O_3 additives too. Uncoated and SiC coated short fibre C_f/SiC were tested in air under high flow rate at INDEX (at 1000°C) and at

THERA (up to 1500°C). SiC coatings protect at high temperatures and high flow rates reasonably from erosive oxidation (dust formation). HfO_2 coatings deposited by DEMOKRITOS on this material are an efficient protection at 700°C in air, but not at 1000°C. High temperature oxidation of C_f/SiC with SiC coating at 1500°C/air can be accompanied by a decomposition of the Si containing structures.

High heat flux / thermal shock tests and measurement of thermal conductivity were performed also at FZJ. Doped graphite materials, doped CFC and SiC-based materials, were tested under thermal shock loads from 0.2 to 2.4 GW/m^2 for ~4-5 ms. The SiC-based materials, either dense or made of a mixture of dense and porous layers, provide comparably low erosion and cracking resistance. Nevertheless at cyclic thermal loads between 350°C and 1650°C, characteristic for re-entry scenarios which is the main application field of these materials, the SiC-based materials showed a good performance and no macroscopic damage. Regarding doped C-based materials, Ti-doping increased drasticallythe thermal shock resistance under typical disruption conditions. In addition thermal diffusivity, specific heat and subsequently thermal conductivity was determined on doped C-based materials. These measurements are coincident with the thermal shock tests indicating a stepwise improvement of the material quality and erosion resistance with increasing thermal conductivity.

Chemical erosion of C-based materials under hydrogen bombardment was measured at IPP with mono energetic ion beams and IC plasmas at 30 and 200 eV at RT and elevated temperature simulating the conditions expected in a fusion reactor. The results show that the different grades from Ti-doped graphites from CEIT meet the requirements for the reduction of the erosion yield with fluence for 30 / 200 eV at elevated temperature. Also the Ti-doped material from UALI used for the plasma-facing mock-up shows reduced erosion yield, meeting also the requirements for 200 eV at elevated temperature. The doping of 0.5 at% Ti of the CFC material from INCAR is still too low and too inhomogeneous to effectively reduce the erosion yield under all conditions. The surface morphology for all fine-grain graphites showed a needle structure with carbide grains on top, desired for the passivating effect. The doped CFC from INCAR exhibited a low Ti concentration in the matrix, which was too low for showing a significant reduction of the erosion yield.

Bulk WSi_2 and the ternary W-based film with 32 at% Si and 25 at% Cr from IPP (WP1.3) were eroded at IPP with a 500 eV deuterium ion beam at room temperature and 600°C. Post-analyses clearly showed that at 600°C Si and Cr were diffusing out to the surface where they were eroded. The post-analyses enabled determining the real composition of the eroded material from the ternary tungsten-based layer and therefore the erosion yield for each element.

Mechanical characterisation of the interfacial fracture toughness of bimetallic beams was performed by JRC on W-armoured Eurofer steel components for plasma facing applications. Different types of delamination tests (Mode I & II, mixed mode) were performed at RT in air. In cooperation with AEN, interfacial shear strength measurements were carried out on brazed W/CuCrZr compounds. Measurements were done at RT and elevated temperature (400°C). Corresponding tests on brazed W/Cu/CuCrZr compounds, where the Cu/CuCrZr was to be probed, failed owing to the excessive ductility of the Cu (alloys) and the outstanding toughness of the interface. Moreover, flexural properties of SiC-multilayers were determined. 3PB and four-point bending 4PB tests with different sample sizes and geometries have been performed at RT and at 1550°C. The main conclusions from this work can be summarized as follows: There is good agreement with the RT results previously obtained by POLITO. The incorporation of porous interlayers reduces the scatter in flexural strength and the sensitivity to statistical size effect. The flexural strength itself is hardly affected by the incorporation of porous layers, while the flexural modulus is not affected at all. Little strength loss observed at 1550°C (dense: 25%, porous: 16%), as compared to RT. The results suggest that more than the current 2 out of 11 porous interlayers should be incorporated to strike an optimum balance between strength and damage tolerance.

Friction and wear tests were performed by MERL on different SP1 materials in view of testing the performance of these materials for brake application. Best results were obtained with C_f/SiC and C_f/C materials provided by EADS. 5 C_f/SiC and 2 C_f/C brake discs were tested. The C_f/C disc material gave low friction results. The C_f/SiC disc with highest fibre content and density had a stable friction coefficient close to the commercially available brake disc material. Its wear resistance was also comparable. It could be demonstrated that the oxidation coating influences the friction results and assures protection from wear. Best results were obtained on brake disc #2 (coated): it showed small scatter in friction coefficient responses, a friction behaviour almost independent of the sliding speed, higher friction coefficients than the commercially available C_f/SiC material, and low wear. This material is thus very promising for brake application.

During and after the SP1 meetings taking place during the project, the partners of WP1.4 acting as SIC members (AEN, EA, AREVA-FR, FZJ, IPP, and NNC) evaluated/validated the alternative concepts of WPs 1.1, 1.2 and 1.3 according to the results shown by the respective partners in view of the application corresponding to the Topical Group of their concern.

2.1.4.3 Deviations from the work programme, and corrective actions taken

There were no major deviations from the work programme.

2.1.4.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.2 Subproject 2 – Heat sink materials

SP2 is divided into ***3 work packages*** (WP) and ***16 project partners*** contribute to this SP:

Partners	**WP 2.1: High-Temperature Heat Sinks**	**WP 2.2: High Conductivity Heat Sinks**	**WP 2.3: Nanoscopic Interface Design and Modelling**
AIT	**X**	**X**	
CEIT			**X**
DLR	**X**		
EADS	**X**	**X**	
EMPA		**X**	**X**
EPFL		**X**	**X**
FZJ	**X**		**X**
IFAM	**X**	**X**	
IMSAS (SP coord)	**X**	**X**	
INASMET	**X**		
IPP			**X**
PLANSEE	**X**	**X**	
SIEMENS	**X**	**X**	
TUW	**X**	**X**	**X**
UALICANTE		**X**	
WUT			**X**

X: WP partners **X** **WP** leader

2.2.1 WP 2.1: High temperature heat sinks

2.2.1.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
IFAM	**Coordination of work package** Development of particulate reinforced PM heat sinks based on Cu				20,0
AIT	Development of short fibre reinforced heat sinks				0,7
DLR	Development of heat sinks reinforced with continuous monofilaments and wires				53,3
EADS	Suggestion of potential space applications and evaluation of alternative material concepts Development of Cu-SiC- fibre heat sinks				30,2
FZJ	Thermomechanical and environmental testing				14,3
IMSAS	Development of heat sinks reinforced with continuous W wires				59,4
INASMET	Development of nanoparticle or VGCNF reinforced heat sinks				38,0
PLANSEE	Suggestion of potential applications for high temperature heat in microelectronics and evaluation of alternative concepts				4,0
SIEMENS	Suggestion of potential applications for high temperature heat in power electronics and evaluation of alternative concepts				13,0
TUW	Development of CuW gradient materials (FGM)				5,0

Objectives:

Develop, characterise and test new heat sink materials based on high conductivity matrix reinforced with ceramic and intermetallic (nano)particles or fibres for use in high temperature applications, where excellent structural stability combined with good thermal conductivity play primary role (aircraft engines, HT reactors, heat exchangers, energy converters, brake discs, welding electrodes, etc.).

Phase 1: Define real user needs on high temperature heat sinks, elaborate up-dated detailed state-of-the-art and patent review, formulate alternative concepts for the research approach.

Phase 2: Select most promising research concept capable to meet R&D requirements on a basis of requirement validation and feasibility studies performed on alternative potential approaches; identification of potential spin-off projects.

Phase 3: Systematic full scale research, development and optimisation of high temperature heat sink material(s) according to the selected materials concepts and related processing techniques: Cu/SiC monofilaments (DLR, EADS), Cu/W wires (IMSAS, DLR), Cu-SiC particles (IFAM) and Cu/VGCNF (INASMET).

Environmental testing of developed materials under real application related conditions (EADS, FZJ, SIEMENS), suggestion and optimization of component design.

Phase 4: Final optimisation of materials and processing techniques developed in phase 3 and verification of their performance in mock-up testing

Description of performed work

Work performed in this WP was coordinated in close interaction with WPs 4.1, 4.2, 4.3, 3.2.

Phase 1: Industrial participants (EADS, PLANSEE, SIEMENS) defined real user needs including technical and commercial requirements on high temperature heat sinks in User requirement specification

(URS). These requirements were transformed into development requirements (material requirement specification MRS), which were validated by the Scientific industrial committee (SIC). Simultaneously all research participants performed the detailed state-of-the-art study, patent review and critical assessment of existing visions concerning manufacturing and application of high temperature heat sinks in the range of the responsibility and competence of each participant.

Phase 2: Evaluation studies including manufacturing and testing of lab-scale specimens were performed in order to validate the feasibility of proposed concepts to meet the defined requirements. The main attention was given to copper matrix composites reinforced with continuous ceramic (SiC) or carbon fibres (DLR, EADS, IMSAS) and with ceramic nanoparticles (IFAM, INASMET). The potential of liquid (pressure infiltration) and solid state manufacturing techniques (diffusion bonding of coated fibres, sintering of coated nanoparticles) for manufacturing of proposed heat sinks was evaluated from the technical as well as economical point of view. The most promising research concept capable to meet all specified requirements with known and acceptable risk were proposed for further systematic research in phase 3.

Phase 3: Systematic full scale R&D on selected promising composite materials:

- ***Cu + SiC monofilament composite*** via PVD coating and HIP, optimisation of fibre volume fraction, fibre spacing and fibre distribution, investigation of the chemical composition of the fibre/matrix reaction zone and preparation of equipment for industrialization of coating process (PVD Inline Hollow Cathode Technology) .
- ***Cu matrix composite reinforced with W wires****:* optimisation of gas pressure infiltration process parameters (temperature, time and pressure), optimisation of volume fraction of reinforcement, fibre diameter and architecture, production of Cu/W composites by vacuum diffusion bonding (in case of degradation of W wires on high temperature during infiltration) and testing of the thermal conductivity in reference laboratory.
- high temperature resistance and conductive ***metal matrix composite based on copper reinforced with Vacuum Grown Carbon Nano-Fibers*** (VGCNF) via electroless plating + sintering using industrially feasible manufacturing techniques
- ***Co + diamond PM composites***: studying of various intermediate layers (alloying with Cr and Ti), reduction of residual porosity, increasing the volume content of diamond in order to tailor CTE and TC, optimisation of the particle size and distribution, optimisation of the processing parameters and sample testing in reference laboratory.
- ***FGM material based on a Cu/W composite***

The selected materials were optimised for applications in compounds defined in URS mostly in heat sinks for fusion reactor (divertor).

The developed materials underwent large application oriented testing programme in order to obtained basic material properties (strength, CTE, TC, etc.). Selected material specimens were prepared and supplied to SP3 for testing under intense neutron irradiation (DLR, IMSAS). Optimum usage of developed materials in future components was proposed in close cooperation with SP4. Several specimens were supplied for the test of mutual joinability under extreme loading.

Phase 4: Selected materials and processes were further optimised to improve their industrial feasibility:

- HIP-joining of W-armour with OF-Cu and CuCrZr (DLR)
- Gas pressure infiltration of 3D W-reinforcements with copper matrix (IMSAS)
- 2-D reinforced Cu/SiC composites with optimized fibre-matrix PVD bonding layer (IPP) compacted via vacuum hot pressing (EADS)
- hybrid SiC/graphite Cu matrix composites manufactured using finer copper and SiC powders (IFAM)
- Cu-40% vol. VGCNFs composites with the boron doped copper coating (INASMET)

Selected composites with optimal arrangement of tungsten wires (cooperation with FZJ, IPP) were used for production of divertor monoblocks (DLR, IMSAS) and flat tile mock-ups. The mock-ups were tested at FZJ.

2.2.1.2 Results

Cu+ SiC monofilaments:

- Laboratory unidirectional composites with CuCrZr matrix containing 9 to 38 vol.% of SiC monofilaments were prepared and optimised by DLR. Processing route was magnetron sputtering of Cu onto fibres followed with hot isostatic pressing (HIP). Obtained fibre strengthening (composite UTS ~500-800 MPa) was however lower than expected if compared to previous results with titanium matrix composites. Therefore different designs of fibre/ matrix interface (with carbide forming Ti-, Cr-interlayer) were investigated. The interfacial strength was tested by fibre push-out test. The interface fracture predominantly occurred in the C-coating of SiC. The thermal conductivities were in the range between 170-300 W/mK depending on fibre volume fraction, whereas the conductivity was slightly increasing with testing temperature up to 600°C
- Equipment for continuous PVD coating of SiC monofilaments with Cu using hollow cathode with vacuum sluices, modules for fibre cleaning by heating, plasma etching and automated fibre transport system was build up and successfully tested by EADS. The device was able to coat the fibres with a thin Ti layer (suggested by IPP for establishing mechanically stable interface between fibres and matrix) and a Cu layer with about 10µm thickness. Because of relatively low production capacity (about 5 m coated fibre with a Cu layer of 10µm thickness in 1 hour) an experimental set-up for an alternative production route via vacuum hot pressing of fibres coated with a thin bonding layer between Cu-foils was suggested and tested. For this production route inline PVD was used for first coating of SiC fibres with thin bonding layer of Ti (100 to 200 nm) and subsequently with thin layer of the Cu matrix material on the top of Ti to prevent its oxidizing at the atmosphere. This manufacturing route was suitable for production of plane specimens, reinforced in two dimensions. The developed vacuum hot pressing technique enabled manufacturing of Cu/SiC samples in the dimension of 50 x 50 x 4 mm, 2-dimensionally reinforced with 4 unidirectional layers of SiC fibres in 0/90° orientation.
- Alternatively hot rolling of SiC monofilament between two Cu-foils was suggested and successfully performed by CEIT. This technique was further optimised in order to provide the possibility for industrial manufacturing of unidirectionally reinforced Cu-SiC tapes.

Cu reinforced with tungsten wires:

- Several technologies for production of Cu/W composites including vacuum diffusion bonding (DB), pressure-less infiltration and gas pressure infiltration (GPI) were tested by IMSAS. GPI was recognized as relatively simple and appropriate technique when compared with time consuming vacuum diffusion bonding. New experimental autoclave and filament winding machine were constructed for manufacturing advanced composites with special wire architecture. The infiltration parameters were optimised to avoid any fibre degradation due to excessive heat flux during processing. Various arrangements of tungsten wires in copper matrix were then produced and characterised, incl. Cu/W cloth (circumferential winding), Cu/W cloth plate, Cu/W cross ply, Cu/W composite with circumferentially wound W wire, etc. Thermal conductivity of all samples was higher than required 200 W/mK. CTE depended on architecture and wire volume fraction. The special architecture was found, which allowed controlling of CTE in the range of 4 – 6 ppm/K in all composite directions. Room temperature tensile strength of composites exceeded 2000 MPa; tensile strength at 500°C was more than 800 MPa and room temperature bending strength was 2,5 – 3 GPa. Various divertor monoblock mock-ups made of developed

Cu/W composites with variable volume fracture and orientation of W wires were manufactured at IMSAS and delivered to FZJ for heat flux testing. Additionally, CFC monoblock divertor mock-up with Cu-W composite tube was also prepared and submitted for heat flux testing in SP3. Finally a divertor mock-up with advanced patented design was developed and successfully tested with very promising results. Cu-W composites prepared by gas pressure infiltration proved fully their potential for the current divertor applications.

- A new method was developed to synthesize MMC interlayers consisting of several fibre layers, various fibre orientation, e.g. 0°/0° and 0°/90° and various fibre volume fraction. In the first step the UD-layers were prepared by two subsequent electroplating processes. Afterwards the UD layers were consolidated by vacuum hot pressing in cooperation with MTU aero engines to form the MMC interlayer. This method delivers samples for investigations of the thermal properties perpendicular to the fibre direction. The values of the thermal conductivity of the tested MMCs (0°/0° and 0°/90°, V_f = 8% to 13%), investigated by laser flash (LFA) measurements are above the required value of 200 $Wm^{-1}K^{-1}$. In addition small mock-ups – MMC, W tile and CuCrZr tile - were prepared. Thermal shock tests which were performed by Ansaldo Ricerche S.p.A show good results. MMC interlayer with a uniform distribution of the fibres and a fibre volume fraction of about 12%, W tiles and CuCrZr tile were brazed to a flat tile mock-up by Ansaldo Ricerche S.p.A. Heat flux test were performed in the high heat flux facility JUDITH at Forschungszentrum Jülich GmbH.

  The experimental temperature data and optical investigations of the first mock-up manufactured from W_f/Cu composites indicated that two of four W tiles were optimally bonded / brazed. Their thermal behaviours showed good agreement with the predicted temperatures during the heat flux experiments. At the highest heat flux of 10.5 MW/m2 one of these W tiles remained bonded and showed an optimal stable thermal behaviour. The second monoblock mock-up could confirm the achieved results and showed unstable thermal behaviour after 25 cycles at 10.5 MW/m2. Microscopic investigation of the cross sections of each W tile revealed that the failure was due to insufficient bonding caused by brazing difficulties. Nevertheless the implementation of the novel W/Cu MMC was considered as successful. The fibres remained stable embedded in the matrix, the failure were mostly caused by improper brazing technique.

CuCrZr-matrix composites reinforced with W-wire

- were developed by DLR as an alternative to CuCrZr/SiC using the same technological approach (PVD+HIP). In this case the interfacial strength exceeds the matrix shear stress (τ > 140 MPa). Thermal conductivity of W/CuCr1Zr (Vf = 50%) was ca. 250 W/(mK) in fibre direction and 225-235 W/(mK) transverse to the fibre direction. The utilisation of this composite in the monoblock design of small scaled divertor was proposed and patented. Two divertor mock/ups were manufactured. Both contained a 3mm thick W-wire reinforced CuCr1Zr composite material with the reinforcements running in axial direction between the tungsten body and the CuCr1Zr cooling tube.

Finite element analyses of the stresses in such divertor mock-up were determined making use of the obtained experimental data of this composite material (wire volume fraction 50%). It has been found that most critical stresses occur after cooling down the component from the processing (HIP) temperature to room temperature. According to the calculations the properties of the W-wire reinforced CuCr1Zr transverse to the wires may not be sufficient in terms of the required strength and ductility. In order to be able to produce more reliable parts complicated (transversely wound) tube specimens were produced. Mechanical testing of these specimens showed substantially better mechanical behaviour (strength was a factor of more than 3 higher) than the simple transverse bundle specimens. This principle was considered in optimization of the final divertor mock-up design.

Vacuum Grown Carbon Nano-Fibres (VGCNF) composites:

- Manufacturing process for preparation of Cu-Ni/VGCNFs composite via electroless plating and subsequent sintering via hot pressing was developed at INASMET. First composites containing up to 40vol %.VGCNF were prepared exhibiting no fibre clusters, low oxidation (<0.5%) and low porosity (<1%) level. Homogeneous Cu/VGCNFs composite were additionally fabricated by the electroless plating technique. The presence of VGCNFs clusters was significantly reduced. Higher densification degree was achieved, whereas the use of controlled electrical field on the spark plasma sintering gave better results over conventional hot-pressing. It was possible to increase the amount of VGCNFs in the composites up to 60 vol. % maintaining homogeneous microstructure. The positive role of VGCNF in the reduction of CTE was approved (<10*10-6 mm-1). The fibre/matrix interface was modified by the addition of titanium nanoparticles, the results showed good distribution of titanium and the formation of TiC. The modification of the interface by the incorporation of third metallic elements on the interface has shown an evidence of an increase of thermal conductivity and a reduction of the coefficient of thermal expansion. However the weak Cu/VGCNFs interface was still identified as the main hurdle to be overcome. Promising systems enabling improvement of interfacial bonding were identified combining reactivity and solubility properties of different elements in Cu and C, incl. Ni, Co, B, etc. Ni-Mo-B coating by electroless plating on VGCNFs showed the best performance. Parallelly a facility for coating of discrete particles (short fibres, CNT, VCGNF, diamonds etc.) on large scale level was developed and constructed. This facility was used in industrialisation phase for materials developed within ExtreMat project.

Co + diamond (Cu-SiC) composites

- Co matrix composites containing 40 vol% of diamond (CD) particles were prepared by hot pressing of powdered mixture and their thermal properties were evaluated (IFAM). The presence of diamond particles showed positive effect on the reduction of thermal expansion (CTE~ 10 ppm/K). However, obtained thermal conductivities were rather low due to low interfacial thermal conductance in this system. Since the coating of diamond particles with carbide forming element (Ti) did not lead to significant improvement of thermal conductivity, further research on these composites was stopped. As a successful alternative – Cu matrix composites reinforced with SiC particles were developed using SiC powders coated with Mo by sputtering. Thin Mo coating can reduce the detrimental reaction between SiC and copper by the formation of molybdenum carbides and silicides. The control of the interfacial reaction is crucial to obtain high thermal conductivity combined with a reduced thermal expansion coefficient. Thermal conductivities were obtained in the range of 270-300 W/mK accompanied with CTE~11-12 ppm/K. The quality of interface was tested under thermal cycling. However this test has revealed a low resistance against thermal fatigue. Additional activities (use of boron as possible carbide and silicide former, rapid sintering to reduce detrimental reaction effects) aimed to increase the performance of CuSiC composites led to insufficient success, the obtained thermal conductivities of the prepared composites were always lower than 300 W/mK.

Therefore, the effect of admixing of an additional highly conductive phase, like natural graphite, into Cu/SiC composites was investigated. The use of natural graphite flakes as additional reinforcement allowed the preparation of composites with an interesting thermal conductivity without any carbide-forming elements. Further optimization and improvements of the Cu/Gr/SiC composites were achieved by variation of the content and the ratio of the reinforcements and the processing parameters. The use of a graphite content of 50-55 vol.% resulted in promising thermal properties especially perpendicularly to the pressing direction: TC = 450-500 W/mK combined with an reduced CTE of 8-8,5 ppm/K.

A strong anisotropy of the thermal properties in case of the Cu/Gr/SiC composites must be considered unlike particle reinforced copper. The lowest values of the thermal conductivity are measured in the hot pressing direction (z). The other two directions (x and y) show similarly high values of the thermal conductivity, approximately seven times larger than the values along the z axis. The CTE shows an inverse dependence on these directions: smaller values along the x and y axes and higher values in z direction.

The bending strength of the composites with high graphite contents of 60 vol.% revealed insufficiently low values of about 40 MPa. However, the addition of some SiC particles improved the mechanical performance of these hybrid composites compared to Cu/Gr. A maximum value of about 65 MPa for the bending strength of Cu/Gr/SiC was obtained. The bending strength of the composites increases with a higher volume fraction and smaller particle size of SiC.

FGM Cu-W composites

- Gradient material consisting of layers with variable content of W particles in Cu matrix was developed by TUW. Specimens were prepared by a wet chemical coating routine of fine sub-µm size W-powders with thin layers of Cu. Such composite powders of varying W/Cu composition were further stacked and sintered to graded layers of W/Cu composites producing a W-rich on the one side and a Cu-rich side opposite. Optimizations were performed regarding the increase of the mutual W- and Cu-phase distribution. It had been turned out, the higher the Cu concentration the worse the mutual homogeneity distribution. In fact, the W-powders are highly agglomerated and keep this behaviour originating from the W-powder production. On the other hand, W-rich layers could be produced without any problems; no Cu-pooling or W-enrichment was detectable in such layers. By improving the parameters during the wet chemical deposition of the Cu-phase on the W-powders, a more even distribution could be evolved, ending up in an increased homogeneity between the cu and W in Cu rich layers. The particular layers possessed TC from ~100 W/mK for 70% W particles to ~300 W/mK for 30% W particles, while CTE varied from 4 to 18 ppm/K depending on W content. An interlayer for brazing experiments of CFC tiles to CuCrZr was manufactured from this FGM and supplied to SP4 for compound manufacturing (AEN).

2.2.1.3 Deviations from the work programme, and corrective actions taken

There were no significant deviations from the work programme. Some of the originally proposed research concepts were withdrawn from further investigations as they appeared as non feasible for targeted applications (according to the results of feasibility studies and comments of SIC). It concerned mainly the materials reinforced with nanoparticles, dispersoids or SiC-multifilaments. It was decided that the MRS concerning "Nanoparticle reinforced metals for high temperature heat sinks" prepared in phase 1 will be cancelled. VGC- nanofibres were suggested as alternative instead of nanoparticles by INASMET and Cu-SiCp composites were developed as a valuable alternative instead of Co-diamond composites (IFAM).

After stopping the research on carbon nanotubes in WP2.2 in second project year, TUW focused the research on the development of FGM Cu-W composites with variable content on submicron W-particles for high temperature heat sinks. This development was involved as an activity of WP2.1.
It was further decided to move all activities concerning characterisation of internal stresses - TUW from WP2.1 to WP2.3, where it appears more appropriate, because the characterisation of internal stresses is common for both WP2.1 and WP2.2.

The feasibility studies and SIC evaluation in first year revealed that there is only a little chance to apply composites reinforced with continuous fibres developed within WP2.1 in microelectronic applications. This was a reason, why the activities of PLANSEE in the second research year concentrated more towards high conductivity heat sinks developed in WP2.2. The entire research capacity of PLANSEE planned for WP2.1 was therefore shifted to WP2.2.

2.2.1.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.2.2 WP2.2: High conductivity heat sinks

2.2.2.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
EMPA	**Coordination of workpackage** Development of diamond/metal heat sinks made by squeeze casting				47,0
EPFL	Development of heat sinks based on infiltrated diamond				40,3
AIT	Development of short fibre (particulate) reinforced heat sinks				21,0
EADS	Suggestion of potential space applications and evaluation of alternative material concepts				4,1
IFAM	Development of particulate reinforced heat sinks				39,6
IMSAS	Development of continuous and short HM-carbon fibre heat sinks				39,0
PLANSEE	Suggestion of potential applications for high conductivity heat sinks in microelectronics and evaluation of alternative concepts				48,6
SIEMENS	Suggestion of potential applications for high conductivity heat sinks in power electronics and evaluation of alternative concepts				42,2
TUW	Development of heat sinks with carbon nanotubes (short C fibres HM) Conditioning of diamond-metal interfaces				37,3
UALICANTE	Development of graphite based heat sinks				43,5

Objectives

Main: Develop, characterise and test new heat sink materials based on high conductivity phase (diamond, HM carbon fibre, carbon n'tubes) for use in applications, where excellent thermal conductivity combined with tailored CTE play primary role (power- and microelectronics).

Phase 1: Define real user needs on high temperature heat sinks, elaborate updated detailed state-of-the-art and patent review, and formulate alternative concepts for the research approach.

Phase 2: Select most promising research concept capable to meet R&D requirements on a basis of requirement validation and feasibility studies performed on alternative potential approaches; identification of potential spin-off projects.

Phase 3: Systematic full scale research on approved materials concepts and optimisation of processing techniques for high conductivity heat sinks: (Al/SiC and Al/diamond via SQ-EMPA; Cu(Ag)/diamond via GPI-EPFL; Cu/diamond, Cu/SiC via PM-AIT, IFAM; Cu(Al)/HM carbon fibers via GPI – IMSAS; graphite-SiC/Al (Cu) via GPI-UAL; Cu/ short carbon fibers and graded W/Cu via HP-TUW)

Phase 4: Final optimisation of approved materials concepts and processing techniques developed in phase 3 for high conductivity heat sinks focussed on following MMCs: Al/diamond via SQ-(EMPA); Cu(Ag)/diamond via GPI-(EPFL); Cu/diamond, via PM-(AIT, IFAM); Cu(Al)/HM carbon fibers via GPI – (IMSAS); graphite-SiC/Al via GPI-(UAL); Cu/ short carbon fibers and graded W/Cu via HP-(TUW). Verification of the performance of developed materials in relevant potential applications.

Description of performed work

Work performed in this WP was coordinated in close interaction with WPs 4.1, 4.2, 4.3.

Phase 1: Industrial participants (SIEMENS, PLANSEE) defined real user needs including technical and commercial requirements on high conductive heat sinks for power- and microelectronic applications. Simultaneously research participants performed the detailed state-of-the-art study, patent review and critical assessment of existing concepts concerning manufacturing and application of high conductive heat sinks in the range of the competence of each participant.

Phase 2: Alternative promising concepts were selected for further experimental evaluation. Research partners performed evaluation studies including manufacturing and testing of lab-scale specimens in order to validate the feasibility of proposed concepts to meet the defined requirements. The main attention was given to metallic composites containing highly conductive phase such as diamond (EMPA, EPFL, AIT, IFAM), HM carbon fibres (IMSAS), graphite flakes and foams (UA) and carbon n'tubes (TUW). The potential of liquid (pressure infiltration or squeeze casting) and solid state manufacturing techniques (diffusion bonding of coated fibres, sintering of coated particles) for manufacturing of proposed heat sinks was evaluated from the technical as well as economical point of view. The most promising research concepts capable to meet all specified requirements with acceptable risk were proposed for further systematic research in phase 3.

Phase 3 The main aim was to perform full scale R&D on following composite systems selected in phase 2:

- ***Al + diamond composites*** made by squeeze casting: determination of optimal processing conditions during squeeze casting and subsequent heat treatment to get best interface and composite properties, development of diamond preforms by microwave sintering or alternative methods in order to facilitate the near-net shape manufacturing of test samples or components; potential assessment of squeeze cast Al-SiC and Al-SiC-diamond hybrid composites as lower priced alternative materials
- ***Cu + diamond composites*** made by rapid sintering of coated diamond particles: coating of diamonds with different elements and different coating thickness (large diameters: >100µm), compaction of composite by PM process and rapid sintering, Characterisation of material properties, modification of coating equipment for coating of fine powders (<100µm), optimisation of processing parameters for rapid sintering, optimisation of Cu-diamond interface by introduction of alloying elements, thermophysical characterisation of the material
- ***Cu (Ag) + diamond composites*** made by gas pressure infiltration: production and characterization of composites, optimization of infiltration process (parallel processing, mould material, in situ coating of composites), evaluation of the effect of vol. fraction and size of diamond, optimisation of coating method
- ***Cu + diamond composites*** made by gas hot pressing of powder mixtures: development and selection of the most appropriate intermediate reaction layer (phase composition and thickness), increasing the volume content of the diamond particles to tailor the CTE of the composite, optimisation of the necessary particle size distribution of the diamond powder, reduction of the residual porosity by improving the mixing technology and using alternative consolidation methods (e.g. HIP)
- ***Cu (Al)+ HM carbon fibre composites:*** optimisation of gas pressure infiltration for the preparation of unidirectionally reinforced Cu-C and Al-C composites, optimisation of interfacial properties via alloying of the molten matrix
- ***Cu (Ag) + carbon nanotubes (CNT) composites:*** optimization of the CNT distribution by optimizing the matrix precipitation process, evaluation of alternative mixing operation like high energy ball mixing, infiltration techniques, optimization of the decoration method of the CNT to achieve optimized fibre-matrix interfaces; evaluation of the use of short C fibres instead of C-Nanotubes as cheaper version
- ***graphite – metal heat sinks:*** optimization of procedures to infiltrate metal into graphite flake performs, combine the graphite flake with an adequate complementary reinforcement material (SiC, Diamond, etc) to reduce the thermal expansion of the composite in the z direction, without reducing the TC; thermal treatment of the composites to improve the interface graphite/alloy interface.

The related process techniques (liquid state: gas pressure infiltration GPI and squeeze casting SQ; solid state: diffusion bonding, HIP, SPS) were developed and optimised with an aim to evaluate up-scaling and industrialisation ability of these concepts. Five potential testing "prototypes" derived from product components in micro- and power electronic applications were suggested by the industrial partners (PLANSEE and SIEMENS) for service related testing in

SP4. Samples from the selected materials systems were produced accordingly by the research institutes and delivered to the industry partners for realisation of the components and subsequent evaluation. After validation suited materials and processing concepts were approved for the following industrialisation phase.

Phase 4: The optimised materials (Al, Cu and Ag "reinforced" with ultra-high thermal conductivity carbon-based phases such as diamond, graphite and carbon fibres) - and the related process techniques (gas pressure infiltration GPI, squeeze casting SQ; diffusion bonding, HIP, SPS) developed and characterised in phase 3 on lab scale specimens were further tested on application-near samples for electronic packaging, space application and for optoelectronics in close cooperation with SP4 in particular:

- **Al/diamond** made by squeeze casting SQ for the IGBT base plate application (EMPA, PLANSEE) and for the slab crystal housing defined by EADS made by GPI (EPFL).
- **Cu(Cr,B)/diamond** made by powder metallurgy PM for laser bar inserts and thermal lids (AIT, IFAM).
- **Ag-(Si,Ni)/diamond** made by gas pressure infiltration GPI for thermal lids, and slab crystal housing (EPFL)
- **Cu(Al)/HM carbon fibers** made by GPI for RF-package (IMSAS).
- **Hybride graphite-C_f/metal composites** for the RF-package and the thermal lid(UALI)

The process techniques were further developed and adapted to allow their industrialisation as well as necessary up-scaling.

2.2.2.2 Results

Al + diamond composites

- were successfully prepared via squeeze casting by EMPA. The thermal conductivity of the composite depended on the subsequent solid-state annealing treatment as well as on the diamond conditioning. Using as-received diamonds, a maximum value of 400 W/mK was measured for the composite. Higher thermal conductivities of up to 550 W/mK could be reached using conditioned diamonds, i.e. diamonds that had either been etched in oxygencontaining atmosphere or “graphitised” in inert atmosphere. Composites containing larger (uncoated) diamonds (> 100 µm) were much more sensitive to small variations in the conditions during manufacturing than composites containing smaller diamonds (< 50 µm). Reproducibility of the properties is still unsatisfactory for the composites with larger diamonds. Better reproducibility and better interfacial properties are obtained by using smaller diamond grits. However, the maximum thermal conductivity of such composites is only about 450 W/mK. Stable diamond preforms were successfully produced by using different binders. However, binders can impair the interfacial thermal conductance very significantly. SiC-coated diamonds (made via CVI - supplied from ATL) allowed the production of composites with high thermal conductivity of about 520 W/mK (for diamonds of mesh size 70/80) in as-cast condition, i.e. no subsequent annealing treatment of the composite was necessary. Thermal cycling test proved the good interfacial characteristics of the interface in such composite; the loss in thermal conductivity after thermal cycling test (500 or 1000 cycles between -55°C and +150°C performed by SIEMENS) was less than 2%. No significant in-depth corrosion due to the presence of aluminium carbide was observed after environmental “pressure cooker test” performed by SIEMENS. Systematic series of Al-CD samples have been prepared, by varying the alloy composition and the heat treatment. These samples were evaluated by TU Wien under traction conditions, by synchrotron X-ray diffraction, synchrotron tomography as well as neutron diffraction.

- The coating of diamond by CVI SiC was successfully achieved on 200g of diamond type IIb single crystals. This amount was sufficient for the preparation of a large size sample for IGBT plate demonstrator. A first scale-up tentative of the CVI process was attempted to produce batches of 4 preforms to 127x137x5mm.

Al + SiC composites

- Al-SiC specimens featuring thermal conductivity of up to 270 W/mK were produced by squeeze casting using pure aluminium as matrix instead traditional AlSi matrix (EMPA). A strong bonding between matrix and the SiC particles is obtained in as-cast condition although only very few interfacial aluminium carbide is present in the composite. The obtained thermal conductivity was about 260 W/mK in as-cast condition, which is about 20% above the value of commercial Al-SiC materials which have an AlSi-matrix. Al-SiC composites behave very well in both the thermal cycling test and the pressure cooker test. In case of gas pressure infiltration Al/SiC composites with bimodal particle distribution showed lower thermal conductivity than those made by squeeze casting in the whole range of particle volume fraction due to the dissolution of SiC in the liquid aluminium (EPFL). Conducting electrical resistivity measurements on the two series of Al/SiC composites for comparison between gas pressure infiltration and squeeze casting indicated that the GPI samples had some residual porosity that may have also affected the thermal conductivity. A second result of these measurements was that the squeeze cast samples were virtually free of Si in solid solution..
- Hybrid composites containing both diamond and SiC particles were also manufactured by EPFL. However, the annealing treatment, which is necessary to get sufficient bonding between the aluminium and the diamonds, resulted in incipient melting of the composite since the SiC was partly dissolved and alloying of the aluminium occurred.

Cu + diamond composites

- The Cu-based composites reinforced with diamond particles were fabricated by powder mixing with subsequent pressure assisted consolidation by IFAM. In order to solve the interface problem between copper and diamond different carbide formers added as alloying elements to the copper matrix were investigated. The highest thermal conductivities were obtained with diamond composites based on CuCr or CuB. The control of the interfacial reaction resulting in the formation of a nano-sized carbide layer is crucial to enable the manufacturing of Cu/diamond heat sinks with high thermal conductivities up to about 700 W/mK combined with a coefficient of thermal expansion (CTE) of 7-8 x 10^{-6}/K. The evolutions of the thermal conductivity and the coefficient of thermal expansion as a function of the alloying content of boron in the copper matrix in CuB/60vol% diamond composites were studied. The thermal conductivity achieves a maximum (>600 W/mK) at boron levels of about 0,5 wt.% (2,9 at.%) and decreases slightly at higher boron contents. This decrease of TC is (according to model developed by EPFL) due to two extreme situations, i.e. all boron is at the interface and all boron is in the matrix. The comparison between modelling and experiment suggests that an increasing thickness of the boron carbide layer can cause the reduction of the thermal conductivity of the composites with the increasing boron content.
- Fast pressing techniques, e.g. Spark Plasma Sintering (SPS), offer advantages regarding shorter processing times, higher efficiency and often better properties, compared to traditional hot pressing. The control of the interfacial reaction resulting in the formation of thin and nano-sized Cr_3C_2 or $B_{6,5}C$ layers is crucial to get high thermal conductivities.

The interfacial carbide formation is different in morphology depending on the pressing technology. Obviously, the rapid heating (during the directly heated hot pressing) can cause a smaller critical nucleus radius and a higher number of nuclei for carbide formation resulting in finer and smoother interfacial structures compared to the conventional hot pressing process. This can presumably correlate with the achieved higher thermal conductivity of the composites. The set-up/pressing die configuration has an important influence on the thermal diffusivity/conductivity values of diamond based composites if larger plates are manufactured. Possible temperature gradients in SPS must be evaluated carefully to optimize processing in larger size specimens. For the fabrication of large parts, e.g. base plates for IGBT modules with size of 100x100 or larger the design of the die and the configuration of the pressing set-up have played a crucial role.

- Selected composites were tested at SIEMENS in thermal cycling tests and showed good interfacial bonding and only small decrease in thermal conductivity of about 10-15% and relatively stable CTE after the thermal cycling. Different plates of composites (disks with a diameter of 80mm and a thickness of about 2mm) with the most promising compositions were sent to PLANSEE for further investigation and production/testing of some large test samples for microelectronic applications. In addition some Cu/Cu-diamond/Cu laminates were also produced to improve the machinability and bonding behaviour of the surface of diamond composites.

- Copper-diamond composites based on PVD/CVD (Mo, Cr, W, B) coated diamonds were developed by AIT. An optical method (Diashape) has been used to characterize the coating thickness of various coated diamonds. Different hot pressing processes have been used. The investigated composites based on PVD Mo coated diamonds showed the best thermal performance. Here it was demonstrated, that a thin layer (in the range of 5 to 10nm) resulted in a good thermal contact between matrix and reinforcement. First experiments using boron coated diamonds resulted in rather lower thermal properties if compared with boron alloyed matrix. Using alloying elements (B in the case of 120/140 diamonds and Mo in the case of 325/400 diamonds) the thermal diffusivity values reached closely 200 [$m^2/s10^{-6}$]. Even after thermal cycling these values could be retained. It was also shown that there is a strong dependence between holding time and alloying content. In cooperation with ATL (SP4), CVD W coated diamonds were also successfully tried.

- Further reduction of the CTE in PM composite materials requires increase of the diamond volume content. This can be achieved by a bi- or multimodal diamond distribution. In order to identify the possible size of diamond which can be used without a too large degradation of the thermal diffusivity, samples with different diamond sizes were prepared. The first composites consisting of 60 vol% diamond with different size showed a reduced CTE. However, at the same time the porosity was increased. Surface profiles of diamond composites were measured in order to optimise the diamond size for minimisation of surface roughness.

- A method for measurement of thermal diffusivity on large surfaces was developed (AIT), which allows identification of anisotropy of TC. AIT carried out thermal investigations (local thermal mapping) on large scale samples in order to check their uniformity. An Al-SiC plate (provided by SIEMENS) was analysed, showing that the developed method could be a suitable tool for local thermal mapping. This method was also used for the local mapping of samples with different diamond concentration. By increasing the volume concentration to 60 vol.% a reduction of the thermal properties was observed. At the same time certain inhomogenities were observed on plates with size of 40x40 mm²

- 16 pieces of heat spreaders have been prepared in a near net shape for application oriented test. For this purpose copper-diamond core was covered on both sides with a copper foil and hot pressed using an appropriate punch. The parts have been machined and forwarded to PLANSEE for application tests.

Cu (Ag) + diamond composites

- Aluminium-, silver-, and copper based diamond composites with thermal conductivity of up to 780, 920, and 750 W/mK, respectively, were developed by EPFL using gas pressure infiltration of diamonds with liquid metal. A number of alloying elements to silver and copper matrices was investigated in terms of their potential to confer high thermal conductivity to the metal diamond composites. Critical alloying element addition to Ag and Cu for various active elements was determined. The alloying elements can be added in situ, i.e. by mixing the diamond powder with SiC (in case of infiltration with pure Ag to be alloyed with Si) and with B4C (in case of infiltration with pure Cu to be alloyed with B). In order to improve conductance at the interface the surface of diamonds were roughened by selective reaction with metallic particles like Fe (TUW).
- The resistance to thermal fatigue has been tested on diamond composites with Cu-B, Cu-Cr, Ag-Si, and Ag-Ni matrices. All materials loose slightly in thermal conductivity upon thermal cycling, yet stay at overall high level (>90% of the initial value). The relation between volume fraction of diamond and the coefficient of thermal expansion of Ag-, Cu- and Al-based composites using bimodal particle distributions was explored. It has been further established that the DEM modelling scheme is the most appropriate to predict the thermal conductivity of metal diamond composites for a phase contrast (kp/km) range between 2 and 9. The limit of thermal conductivity has been pushed very close to 1000 W/mK. Several samples having a thermal conductivity between 950 and 1000 W/mK at 35°C have been fabricated.
- A set of samples was produced in net shape according to the specifications defined for of the thermal lid type application with high to very high thermal conductivity. Small series of the “laser bar insert plates” and the “slab crystal housing” were produced. To achieve this new processing route for manufacturing of net shape mould for infiltration of loose diamond powder (made from pressed salt) was developed and optimised.

Cu (Al)+ HM carbon fibre composites:

- Metal matrix composites reinforced with continuous pitch based carbon fibres has been prepared by gas pressure infiltration GPI by IMSAS. Cytec K1100 carbon fibres were chosen for the research since they have highest TC (1000 W/mK) of all pitch based fibres available on the market. Infiltration of carbon fibres with pure Cu was impossible due to the extremely high wetting angle. Successful infiltrations were performed with CuCrZr alloy as matrix metal when C fibres were pre-treated in water dispersion of TiN particles. Experiment yielded sound, pore-free composite however, without any reaction at the interface. Use of binary copper alloys with carbide forming elements which improve wetability to carbon fibre led also to satisfactory infiltration results. Already small amount of such elements (e.g. Cr) ensures good wetability in the system. Microstructural investigations revealed pore-free composites with homogeneous distribution of the fibres. Thermal conductivity of unidirectional copper based composites was very high in longitudinal (up to 700 W/mK), but significantly lower in transversal direction (60– 90 W/mK). Similar situation was in case of CTE: extremely low CTE in longitudinal (-1 up to 1 ppm/K) and high in transversal direction (around 18 ppm/K). One of possible ways how to solve this problem was to pre-constrain unidirectionally reinforced composite in transversal direction with the help of low CTE material. CTE of pre-constrained material was 3 – 4 ppm/K (stable even at 900°C) in longitudinal direction and the CTE in transversal direction was that of the chosen constraint material.

- In case of aluminium based composites an extensive reaction at the fibre/matrix interface took the place during infiltration forming highly undesirable hygroscopic Al_4C_3 carbide. Lowest amount of carbides was detected in composites with AlSi matrix. Thermal conductivity of composites with pure Al matrix was 615 W/mK.
- Composites were thermally cycled by SIEMENS. Thermal fatigue test did not revealed any significant reduction of TC compared to as cast composites. Because of rather low conductivities achieved perpendicularly to fibre alignment the infiltration of 2D fibre preforms was investigated as well (cross ply composites with K1100 and K1392U fibres - various orientations 0°/90° and +45°/-45°). After systematic characterisation a lot of material data is now available for various possible partners who are interested for application of these materials. Parameters of gas pressure infiltration have been completely optimized and process is ready for up-scaling
- Beside composites reinforced with long fibres, IMSAS investigated also the possibility to use short fibre in order to reduce anisotropy of composites. Granoc XN-100-01Z fibres were used as short fibre reinforcement. Matrices with pure copper + 0.2 % Cr, 1 % Cr, AlSi5 and MgAl2 were experienced. C fibre preform fabrication route via polymer binder was developed. High residual stresses were observed in bulk composite samples and a proper thermal treatment for their removal was needed. The stability of composites repeatedly exposed to heating/cooling cycles up to the temperature of 1000 °C was determined.
- Generally a new material with specific combination of properties has been developed. The manufacturing technology was optimized up to an industrially viable procedure. It exhibits good machinability, structural stability, repeatability and has well defined properties. Targeted values in terms of thermal conductivity and thermal expansion were achieved. With long fibres, one needs to take its anisotropy into account and use it as an advantage in future designs. It cannot be ignored or fight with. This seriously limits the fields of application. More homogeneous distribution of properties can be achieved with short fibre reinforcements, however the top properties typical for unidirectional reinforcement are adequately decreased in this case. In those areas where the density is a major issue, Al and Mg matrix variants can play an important role.

Cu (Ag) + carbon nanotubes (CNT)or short carbon fibre composites

- These composites were studied by TUW. First attempts to manufacture bulk material from Cu coated CNT revealed, that such task is too challenging for manufacturing of sound samples. Extremely large CNT surfaces make the creation of optimum carbon-metal interface very difficult. High porosity level and non uniform CNT distribution did not allow evaluation of most important material properties in particular CTE and TC. Therefore the research on this system was stopped within ExtreMat and CNT were replaced with short carbon fibres possessing high conductivity, whereas the manufacturing technique developed for CNT composites was further utilised. It was within the aim of those investigations to use pure matrices and not carbide forming active alloying elements typically used for the GPI processes. Therefore the respective matrices were either pure copper or silver. But in order to address a necessary optimal interfacial bonding, the necessary carbide forming elements were introduced again by pre-coating the C-fibres with different elements like Co, Mo, Ni (for Ag matrix) or W, Cr or B (in the case of a Cu-matrix). Different C-fibres were investigated, like the Cytec HM fibres K1100, with an intrinsic thermal conductivity of 900-1000 W/mK, or the Mitsubishi K13D2U fibres (λ = 800 W/mK) and the DKD short carbon fibres (λ = 400-700 W/mK). The composite materials were then fabricated either by powder metallurgical methods comprising hot pressing for consolidation or by GPI (in cooperation with IMSAS). The results showed that there could be a significant difference between PM manufactured specimens and samples which had been fabricated by GPI means. All GPI samples indicate much higher TC and lower CTE, than the PM ones. Various composites including Cu/W coated long C fibres, Cu/W coated short C fibres, Cu/B coated long C fibres, Cu/B coated short C fibres, Ag/Co coated C fibres, Ag/Ni coated C fibres, Ag/Mo coated C fibres were prepared by GPI for characterisation. The thermo-physical results indicated a strong dependence on the elements used for pre-coating of the fibres. Especially coatings of Co or Mo within the Ag-composite series turned out to be much more effective than Ni. Dilatometry measurements of obtained composites revealed that already low fibre content (~10 vol.%) can significantly reduced CTE value. The level of anisotropy of such composites is much lower than in case of long fibre reinforcement.

Graphite flake – AlSi12 composites

- were made by gas pressure infiltration by UALI. The activities were focused on the optimization of the thermal and mechanical properties of hybrid graphite flakes-SiC particles/Al-12wt%Si composites. Several particle sizes and various volume fractions of SiC were selected for the manufacture and characterization of these hybrid composites. The results obtained indicate that the thermal conductivity along the z axis is five times smaller than those along the other two directions. The flexural strength increased as the relative amount of the soft phase (graphite flake) decreased, reaching a maximum value of about 74 MPa for composites having the highest SiCp volume fraction (40%) and the smallest particle size (13 µm). Thermal conductivity and CTE of graphite flakes-SiCp/Al-12wt.%Si composites were characterized before and after two different thermal tests conducted by SIEMENS (1000 cycling runs in the range of temperatures –55ºC to 150ºC). The effect of the SiC particle size in graphite flakes-SiCp/Al-12wt.%Si composites in both thermal tests (Thermal cycling and pressure cooker test) was also evaluated (samples containing particles SiC of 13, 40 and 60µm). Preliminary results showed that these treatments affected both the thermal conductivity and the coefficient of thermal expansion (CTE) of all the samples. While the thermal conductivity was slightly reduced, the CTE was increased for the significant planes of the samples.
- Alternatively hybrid composites of graphite flakes-carbon fibres/Ag-3wt.%Si were produced by UALI using graphite flakes and carbon fibres of high thermal performance. The effects of the graphite flake size and volume fraction of carbon fibres on the thermal conductivity and flexural strength of the hybrid composites were evaluated. The results showed that the thermal conductivity is significantly higher for the composites containing graphite flakes of larger diameter (values close to 600 W/mK are easily achieved). The flexural strength of the fabricated materials shows a tendency to increase with increasing volume fraction, although all values were still very low, since the maximum flexural

strength was below 20 MPa. The further optimization was directed to the improvement of the interface and hence the adhesion of the metal and the carbon reinforcement, particularly via thermal treatment of the composite materials (in order to induce chemical interactions between metal and carbon fibres in solid state) or via increasing contact time between reinforcement and liquid metal during the infiltration process.

- A simple model capable to estimate the Thermal Conductivity (TC) of graphite flakes-SiC particles/Al-12wt%Si composites has been developed. The model incorporates the two reinforcements in two steps: a) First the SiC particles are incorporated into the alloy by means of the Hasselman-Johnson model, b) then, noting that the composite microstructure essentially consists of alternating layers of graphite flakes and SiC/Al-12wt% composite, the TC was calculated by assuming a network of thermal resistors either in parallel (TC parallel to the graphite planes) or in series (TC perpendicular to the graphite planes).

Main results of industrial evaluation

- Industrially relevant testing of some developed materials was performed by SIEMENS. Altogether more than 400 samples were received. The most stable material against the environmental test was found to be the bimodal AlSiC without Si from EMPA, which thermal conductivity of 315 W/(mK) was much higher than for industrial AlSiC with Si (200 W/(mK)). The flexural strength and Young's modulus were characterised by four-point bending test. In case of AlSiC from EMPA it was done before and after the thermal cycling. Industrial AlSiC with Si in the matrix, as a reference, has the highest characteristic flexural strength of 345 MPa. At the same time the stiffest material was AlSiC from EMPA even after the thermal cycling with 1000 cycles, since it's Young's modulus was 237 GPa. Industrial AlSiC and Al-diamond composites from EMPA and PLANSEE had about 200 GPa.
- The thermal resistance of the manufactured IGBT modules was investigated and compared with that of the standard modules. The module with voids-free system solder was characterised in view of the influence of the thermal cycling at temperatures -40°C / +125°C. It has been found that TC performance of Cu-diamond composites is better than that of conventional AlSiC materials, on the other hand thermal expansion performance of developed composites is superior if compared with plain Cu.
- Temperature distribution was tested on diamond based composites for thermal lid application (new experimental setup was developed). A comprehensive selection of high performance heat spreader based on diamond and graphite composites was collected. They represent state of the art technology usable in different applications. Identification of these applications was carried out and potential lead users were addressed.

2.2.2.3 Deviations from the work programme, and corrective actions taken

There were no significant deviations from the work programme. Some of the originally proposed research concepts were withdrawn from further investigations as they appeared as non feasible for targeted applications (according to the results of feasibility studies and comments of SIC). It concerned mainly the materials reinforced with graphite foam, ceramic particles and carbon nanotubes, which were in further research replaced with cheaper short carbon fibres with high thermal conductivity.

Similarly as in WP 2.1 it was decided to move all activities concerning characterisation of internal stresses - TUW from WP2.2 to WP2.3, where it appeared more appropriate, because the characterisation of internal stresses is common for both WP2.1 and WP2.2.

According to suggestions of industrial partners (SIEMENS) also Al-SiC composite made by squeeze casting were alternatively investigated in phase 3 (EMPA). The motivation was to increase thermal conductivity of current AlSiC materials by using of pure Al matrix instead of Al-Si alloy.

After Dr. Olivier Beffort of EMPA left the project (in Mai 2007), Dr. Ludger Weber of EPFL was nominated as workpackage leader and coordinator of WP2.2.

2.2.2.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.2.3 WP 2.3: Nanoscopic interface design and modelling

2.2.3.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
TUW	**Coordination of workpackage** Modelling of discontinuously reinforced and skeleton heat sinks Characterisation of internal stresses in composites				54,0
CEIT	Thermomechanical modelling of continuously reinforced heat sinks				44,7
EMPA	Conditioning of diamond-metal interfaces				33,6
EPFL	Characterisation and modelling of interfacial thermal resistance				15,7
FZJ	FEM of heat sinks component architecture				16,6
IPP	Atomistic interface modelling and conditioning				98,0
WUT	Microstructural characterisation of interfaces				46,0

Objectives:

Main):
- developing of stable interface between constituents of composite in whole working temperature range without reduction of thermal conductivity
- modelling of interfaces, in terms of chemical and process compatibility and minimum interfacial thermal resistance
- modelling of the overall composite architecture as well as the geometry and particularly the volume fraction of the reinforcement in order to achieve the highest thermal conductivity and to reduce excessive internal stresses
- developing of realistic models for design of heat sink architecture

Phase 1: Collect and assess available information on existing composites and models, elaborate updated state-of-the-art review, formulate concept for the research approach.

Phase 2: Adapt existing approaches for interface and composite modelling, suggest architecture of composite and surface treatment of reinforcement in order to reduce internal stresses and to minimise thermal resistance of the interface.

Phase 3: Systematic research concerning:
- modelling, design and optimisation of interfaces between constituents in composites, adequate interlayers, conditioning or surface modification of reinforcement phase,
- characterisation of internal stresses in developed metal matrix composites
- modelling and design of the composite architecture, the geometry and the volume fraction of the contionuous and particle reinforcements in order to achieve the highest thermal conductivity, low CTE and to reduce internal stresses with respect to targeted applications
- (micro)structural characterisation of developed materials

Phase 4: Optimisation of developed materials and models according to results of systematic experimental testing; suggestion of industrially viable techniques for reduction of interfacial thermal resistance; finalisation of models for design of selected electronic and space components and their verification.

Description of performed work
Work in this workpackage was performed in close interaction with WPs 2.1, and WP 2.2.

Phase 1: Existing information on composite interfaces and composite models was collected and critically assessed with regard to heat sink application. Concept for research approach leading to optimisation of interface with minimum thermal resistance were suggested on the basis of detailed state-of- the- art review (EMPA, EPFL). The appropriate models for thermo-mechanical behaviour of MMC's were selected for the design of heat sink architecture (FZJ).

Phase 2: The architecture (volume fraction, geometry and distribution of reinforcement) were modelled using existing information on composites (FZJ, TUW, IPP, CEIT) with an aim to minimise internal stresses and maximise thermal conductivity according to industrial requirements defined in WP2.1 and 2.2. Three types of composites were concerned: discontinuously reinforced (short fibres, n'tubes, particles - TUW), reinforced with continuous fibres (laminated structures-CEIT, IPP) and skeleton composites (infiltrated percolating structures-TUW). Microstructural investigations were performed on the interfaces of first diamond based composites (WUT, EMPA, EPFL). The feasibility study with surface modification of reinforcing fibres was performed in order to improve interfacial strength (IPP, EMPA) and to reduce thermal resistance of the interface (EPFL).

Phase 3&4: The modelling studies were performed on selected composite materials developed in WP2.1 and 2.2. The architecture (volume fraction, geometry and distribution of reinforcement) was modelled with an aim to minimise internal stresses and maximise thermal conductivity. Microstructural modelling was supported by suitable micromechanical testing (e.g. push-out, indentation, etc.). The main attention was given to diamond reinforced materials and to composites reinforced with continuous W-wires and SiC monofilaments.

The surface of reinforcing phase was further optimized. These activities involved coating of SiC and W fibres with improved Ti based interlayer and galvanic copper plus optimisation of thermal treatment (IPP), investigation of the effect of coatings on interface formation in diamond based composites (EMPA in cooperation with ATL); chemical etching of diamonds before infiltration and investigation of the effect of various metal coatings by TTR measurement (EPFL); determination of reaction products in diamond – metal composites, micrtostructural and analytical characterisations (WUT). The surface conditioning (coating, functionalization, thermal treatments) of the high conductivity filler phases were performed with an aim to reduce thermal contact resistance at an interface and consequently enhance thermophysical properties of the composites. The results from these activities were regularly supplied to WP2.1 and WP 2.2 for synthesis of selected composites. On the other hand the information concerning performance of developing materials was supplied to WP 2.3 for refining of existing theoretical models and to subprojects 4 for updating the work on ExtreMat-compounds.

The architecture of selected mock-up components was proposed according to the results of FEM modelling based on experimentally obtained properties on prepared material specimens.

Experimental determination of residual stresses generated by CTE mismatch of the constituents in diamond-metal composites at RT and during heating cycles via diffraction experiments using conventional X-Ray sources, synchrotron beam lines or neutron radiation; investigation of the relationship between the interface bonding and void evolution was performed by TUW, calculation of residual stresses in W fibre reinforced copper matrix composites was done by CEIT

2.2.3.2 Results

Conditioning of interfaces in diamond composites EMPA, TUW:

Various surface treatments of diamond particles were investigated in order to improve thermal conductance at the diamond-metal interface. The methods included thermal etching in air, oxidation in liquid salt, chemical reaction with Fe or Mn powder or coating with Mo. Effect of quality and diamond particle size on mass loss was investigated. Thermal annealing of Al infiltrated diamonds without pre-treatment was studied for comparison. It has been found that treated diamonds are more reactive than as received ones. The reaction takes place predominantly at (100) planes if compared to (111) ones, although surface treatment can reduce differences observed in non-treated particles. It has been found, that the conditioning of diamond powders by thermal treatment in an oxidative atmosphere may lead to roughening of the diamond surface and influence the reactivity with aluminium matrix. However, this conditioning is very sensitive to small variations during processing and leads to non reproducible results. This requires a development of a method for sorting of diamonds by reactivity. One solution, based on the endorsement of hetero-crystallisation in diamond surface defects, was proposed.

It has been shown (in collaboration with EPMA) that the thermo-chemical etching of the diamond prior to infiltration can improve thermal conductivity of resulting composites. For an aluminium diamond composite the thermal conductivity improved from 630 to 670 W/mK for high quality diamond and from 500 to 640 W/mK for lower quality diamond.

Coating of diamonds with SiC by means of CVD was suggested and proved to be a very suitable method to obtain a very strong interface between these diamonds and the aluminium matrix in as-cast conditions. No further annealing treatment of the cast composite is necessary and no aluminium carbides were observed at or near the interface.

Aluminium nitride coating on diamond was alternatively experienced because of its attractive thermomechanical properties and the ability to selectively coat the faces that have been earlier shown to be weakly interacting with the aluminium matrix (EMPA). In a first step AlN coating acoustic matching interfacial layer was achieved on 111 diamond faces by microwave assisted plasma deposition in aluminium diamond powder bed mixtures under nitrogen atmosphere. In a second step, the use of CVI was investigated for the production of aluminium nitride coating diamond preforms in collaboration with ATL. Based on the thermal decomposition of $AlCl_3$ and NH_3 columnar aluminium nitride coating could be obtained on diamond. The AlN microstructure was found to be columnar with a typical thickness of 5microns. The AlN seems to grow epitaxially both on 111 and 100 faces of diamonds.

Mixtures of Si and diamond can be treated by microwave to produce epitaxial SiC mainly on 100 diamond faces. CVI can be used to coat diamond powder or to produce stable preforms. The polycristalline SiC is however not epitaxial to diamond in this case. Mixtures of Al and diamonds can be treated by microwave under nitrogen to produce epitaxial AlN mainly on the diamond 111 faces. CVI can be applied for the elaboration of AlN coatings on diamonds. The AlN layer is growing epitaxially on 111 and 100 diamond faces.

Evaluation and modelling of interface thermal resistance ITR (EPFL):

Interface thermal resistance between metals and dielectrics was modelled using DEM approach in comparison with Hasselman - Johnson (H-J) model. Assessment of modelling schemes was done using one single composite specimen, whereas the effective phase contrast varied by heat treatment. Thus variation in volume fraction or shape was eliminated, matrix conductivity could be measured independently and calculation was possible for every heat treatment temperature.

It was found that DEM requires an effective inclusion conductivity that is roughly constant for large and small particles. H-J requires a constant effective conductivity of the small particles. For large particles effective conductivity is indicated to raise at low matrix conductivity (high ht-T). DEM yields consistent effective inclusion thermal conductivity of the investigated range of effective phase contrast. The values for intrinsic inclusion conductivity and interface thermal conductance are in agreement with estimated (based on nitrogen content) and directly measured values. DEM is the appropriate modelling scheme for back calculating inclusion related thermal conductivity data. The H-J model yields good estimates up to an effective phase contrast of 4 (@0.6 Vol fraction).

The laboratory TTR apparatus was assembled which enables direct interface thermal conductance measurement on metal-coated dielectric substrates. It has been found that chemically etched diamond yields consistently higher thermal conductivity in composites than the non-etched counterparts. For Al/MBD4 70/80 composites a room temperature thermal conductivity of 675 W/mK was achieved. The JR1-based composites reached roughly 590 W/mK in the best treatment. SiC-coated diamonds seem to be not appropriate for being infiltrated with Ag. The obtained thermal conductivity was low, i.e. only 230 W/mK.

Modelling of composites:

Modelling of thermal conductivity of **particle reinforced composites** and thermomechanical behaviour of ductile matrix was performed by TUW. Diamond composites were investigated as modelled materials.

An analytical Mori–Tanaka model was devised for studying the thermal conduction behaviour of composites reinforced by spherical particles with prescribed size distributions and non-ideal interfacial conductances. A purely analytical model is aimed at allowing low-cost modelling of the thermal conduction behaviour of composites reinforced by particles that follow prescribed size distributions.

Further development of continuum models was carried out. These models account for the inhomogeneous structure of the composite at the length scale of the particulates. A semi-analytical approach based on Mori–Tanaka algorithms for handling non-ellipsoidal particles with inhomogeneous interfacial conductances was extended incorporating the Replacement Tensor Algorithm (MTM/RTA), and tested against multi-particle unit cell models (periodic homogenization). These two approaches provide complementary capabilities in terms of cost and accuracy for studying the thermal conduction behaviour of diamond particle reinforced composites.

Beside this the extensions of the Incremental Mori–Tanaka (IMT) method for describing the thermo-elastoplastic behaviour of ductile matrix composites under cyclic loading were carried out, in particular implementation of mixed hardening models into micromechanically based material routines that use an IMT and debugging and testing of such implementations.

A unit cell models and “windowing” models make use of discrete volume elements that can be employed for both thermal and thermomechanical analysis, with unit cell models requiring periodic unit cells, whereas windowing models can use non-periodic volumes provided their macroscopic symmetry is not markedly lower than orthotropy.

Excellent agreement between results obtained with modified Mori–Tanaka models employing homogeneous (MTM/RTA,hom) and inhomogeneous (MTM/RTA,inh) interfacial conductances, unit cell models (PMA) and three-point bounds (3PB,hom), has been achieved.

Micromechanical methods were developed for describing the thermal conduction and thermo-mechanical behaviour of discontinuously reinforced composite materials. Research work was devoted to the modelling of the thermal conduction behaviour of diamond particle reinforced metal matrix composites and the implementation of mixed hardening models into mean field models for the behaviour of metal matrix composites. These mean field models took the form of a modified Incremental Mori–Tanaka (IMT) micromechanical scheme, which was extended to providing a Chaboche–Marquis mixed hardening model at the level of the composites’ elastoplastic matrix. The numerical behaviour of the algorithm, implemented into a user defined material routine (UMAT) for the commercial FE-package ABAQUS/Standard (Simulia, Providence, RI), was studied in considerable detail in order to assess influences of the control parameters on the accuracy of the predictions of the macroscopic behaviour.

Verification runs were carried out that compared predictions for the cyclic behaviour at vanishing volume fractions with results obtained with a Chaboche–Marquis material model for homogeneous materials that is part of the standard distribution of ABAQUS, and reinforcement volume fraction effects were checked.

Modelling of ***copper matrix composite with continuous tungsten and SCS-6 (SiC) fibres*** was done by **CEIT**. It has been found that SiC-fibre/ Ti-layer/Cu-matrix system behaves like a well bonded system, since both cooling and heating condition have similar radial stresses at the interface. The interface can reduce the residual compressive radial stress around the fibre and introduce tension during the heating process but not strong enough to cause debonding. It has been shown that the crack initiation and propagation is different for square and hexagonal fibre arrangement. This is due to different residual thermal stresses and different stress distribution during loading for different fibre arrangement. The initial damage expected has reached sufficient level to overcome the compressive stress at the fibre/matrix interface when it is heated. Interface debonding, between the fibre and the matrix, is in accordance with previous experimental evidence: the maximum transverse stress before debonding calculated for Cu- 30 vol. % SiC fibres was 57 MPa.

The thermal and mechanical cycling on copper matrix composites reinforced with W fibre was simulated using micromechanical modelling through finite element analysis. A micromechanical model has been developed for the analysis of the thermal residual stresses as a function of fibre volume fraction, which takes into account the thermal history of the composite, the effect of the cooling rate, creep at elevated temperatures for significant periods of time etc. The effects of different interface conditions were modelled with cohesive elements and the transverse stress-strain behaviour at high temperature was evaluated. Chaboche´s model of viscoplasticity was used in the analysis, creep behaviour of copper matrix was introduced as a subroutine into the calculation, thermal residual stresses generated from the manufacturing temperature (900ºC) were considered. Architectural configurations of composites included periodical fibre distribution and random fibre distributions, various fibre volume fraction, unidirectional and cross-ply composites.

Modelling of components:

The 3D-component modelling of heat sink for divertor in fusion reactor was done by **FZJ**. Concentrated efforts were done on two main issues:

1) introduction of a graded W/Cu-interlayer between the plasma facing material and the heat sink material;
2) substitution of the CuCrZr heat sink by a W/Cu heat sink with homogeneous or orthotropic properties and varying composition.

The calculated equivalent stresses in the different materials of the component were compared among each different parameter set. Preliminary set up criteria for an effective comparison were used to qualify the results. Among those, the plastic deformation and the stress level within the soft OFHC-Cu interlayer is probably the most important and becomes least for the introduction of a full W/Cu-gradient between plasma-facing material and heat sink. Nevertheless a clear indication for an improvement of the internal stress field was not obtained and more calculations have to be performed with material data provided and to be provided by the manufacturers.

The data base on relevant material properties for newly developed high temperature heat sink materials within the ExtreMat project, i.e. W and SiC-fibre reinforced Cu composites was enlarged by the ExtreMat partners and these data were compared to earlier produced plasma sprayed W/Cu-materials at FZJ and to the literature data. In regard of the plasma sprayed material a significant improvement of the tensile strength has been achieved and compared to literature data a good correlation for thermo-mechanical and for thermo-physical properties was found.

The expertise on the design of fusion compounds and on the FEM analysis of these compounds was provided for the FEM analysis on fusion compounds developed at DLR and performed at DLR. The simulations were done on a monoblock-design with integrated unidirectional W-fibre reinforced Cu heat sink materials acting as interface between the plasma facing and the heat sink material.

In cooperation with CEIT and based on previously obtained results from thermal shock tests on doped graphite, FEM analyses on the thermal shock behaviour of these materials were performed putting the emphasis on the determination of the onset of particle erosion between 0.4 and 0.6 GW/m2.

Characterisation and optimisation of interface

Characterisation and optimisation of interface in continuous fibre (SiC, W) reinforced Cu was performed by **IPP**. Push-out tests showed that the adhesion between SiC fibre and copper matrix without any interlayer is very low. To increase the fibre-matrix bonding the fibres were coated with Cr and W with a thickness of 300-400 nm before Cu deposition by magnetron sputtering. Push-out tests on these modified fibres showed a significant increase in adhesion compared to the fibres without interlayer. XRD investigations after a heat treatment at 923 K showed a chromium carbide (Cr23C6, Cr3C2) formation and the absence of chromium silicides.

In the case of a W interlayer a W_2C formation is detected but no tungsten silicides. Single-fibre tensile tests were performed to investigate the influence of the reaction zone on the ultimate tensile strength of the fibres. The ultimate tensile strength for fibres without interlayer remains constant at about 2200 MPa after annealing at 923 K. The fibres with chromium and tungsten interlayers, respectively, show a decrease of about 30% of the ultimate tensile strength after the heat treatment at 923 K.

Several interfacial concepts were evaluated in order to find a stable interlayer between the tungsten and copper incl. direct bonding of W to electroplated copper matrix, deposition of thin copper interlayer on W by PVD before electroplating with Cu, deposition of graded transition from W to Cu by PVD with and without additional heat treatment. The properties were investigated through pull-out measurements of single fibres. It was found that the interfacial shear strength increased by a factor 6 by depositing a graded transition between W and Cu plus additional heat treatment. The thermal cycling showed that the graded transition from W fibre to Cu matrix buffers the CTE mismatch during cycling.

To understand the reactions between W and Cu dedicated interdiffusion and segregation experiments were performed. However no segregation of W and Cu at 650°C and no volume interdiffusion of W and Cu at 500°C, 650°C, 800°C and 900°C was determined. Interfacial adhesion of W and Cu is achieved solely through mechanical interlocking.

Various microstructuring concepts (ion sputtering, chemical etching, fibre twisting to 20° and 30° with or without chemical etching etc.) were suggested to modify the tungsten fibre surface in order to enhance interfacial adhesion. In general, an increase of interfacial shear strength due to microstructuring concepts except “Ion sputtering” was indicated. Fibre twisting to 30° showed the highest effect on interfacial adhesion between Cu and W, which increased by a factor 3 compared to the system without microsctructuring. However experimental tensile tests have revealed that the surface modifications slightly weaken the strength of the fibre. Further optimisation is therefore needed.

The reactions between bonding interlayers and substrates have been modelled using the DICTRA-Thermocalc simulation package incl. interdiffusions at CuTi/C interface, as well as Ti/Ta, Ti/Cu and Ti/C interfaces.

The simulation showed that Ti from the Cu-ABA alloy (3 at.% of Ti in Cu) diffuses gradually into graphite substrate during wetting forming a TiC-layer.

Evaluation of internal stresses

Evaluation of internal stresses in particle reinforced composites was performed by **TUW:**

In cooperation with EMPA an Al-Diamond MMC was investigated. The CTE mismatch is transferred from the interface into the metal matrix of the MMC. The consequence is a big amount of residual stresses between the diamonds and the matrix material during heating cycles. Aluminium was chosen for the matrix material because of its carbide forming ability which bears the chance for a good bonding between particle and matrix and thus for high thermal conductivity of the MMC. Al-Diamond based heat sinks after different heat treatments were investigated to show relations between pretreatment and the quality of the interface. The residual stresses in the matrix after quenching experiments from different temperatures where measured. The stress saturation above a critical quenching temperature could be observed. Quenching from temperature above this limit resulted in plastic deformation of the matrix. The decrease of internal stresses after multiple heating cycles resulted in a irreversible debonding between the matrix and the reinforcements which goes along with a reduction of the thermal conductivity. The damage mechanism is analogous to low cycle fatigue damage.

The residual stresses in diamond reinforced MMC (CuB1/CD/60p and AlSi11/CD/60p) during temperature changes were investigated by X-ray diffraction. The stresses were measured in the matrix after quenching from different temperatures at RT. The AlSi11 matrix showed good interface bonding quality but damage after quenching from soldering temperature. The CuB1 matrix was weak at the interfaces compared to the AlSi11 system. Debonding at the interfaces was observed also by SEM images of a fracture surface.

In situ high resolution synchrotron computer tomography was made at ESRF Grenoble at ID19 to investigate debonding and void kinetics in the Al/CD/60p system under thermal load. It has been shown that the voids in the diamond reinforced MMC increase during heating and decrease during cooling. Debonding of the particles from the matrix along the interfaces can be expected due to the big thermal expansion mismatch between the matrix and the reinforcements. The carbides formed in heat treated sample at the interfaces reduce debonding if compared to the as cast material where almost all particles were completely debonded from the matrix.

Combined neutron and synchrotron experiments were made to study the effect of internal stresses on thermal fatigue and void kinetics during and after thermal cycling. In case of the diamond reinforced Aluminium composites (EMPA) high resolution synchrotron tomography showed the small delamination voids at the particle-matrix interfaces and neutron diffraction of a big sample volume provided a good peak distribution in the coarse grained as cast samples. Two systems were compared; Al/CD/60p MMC represents a system of isolated

particles with a dominant macro stress contribution, while in the AlSi7 matrix systems a connected network of reinforcements is formed showing an inverse stress evolution during cycling. A superior thermal fatigue behaviour for a connected network of reinforcements compared with a system of isolated particles was confirmed.
Copper reinforced with SiC and W monofilaments delivered by IPP and DLR was submitted to neutron diffraction experiments showing the residual stresses under application conditions generated by the thermal expansion mismatch between the matrix and the reinforcements. High matrix stress levels during thermal cycling up to application temperature could be observed in fibre direction. In the SiC fibre reinforced composites a significant improvement of the interface bonding quality by Ti coatings is shown by almost constant stress amplitude after multiple cycles. Delamination can be expected for the uncoated SiC fibre due to reduction of the residual stresses after the first cycles. For the W fibre composites, the residual stresses during cycling reveal good interface bonding and no fragmentation.

Structural characterisation

Structural characterisation of materials developed by AIT, IFAM, EPFL and IMSAS was performed by **WUT**. The interfaces in copper alloy infiltrated carbon fibre composites (CuCrZr -C fibre) were examined. Furthermore, the Cu-(Mo)SiC composite and the Cu0.8Cr-diamond composite sintered via PPS were investigated. Finally W-wires treated at different thermal treatments were studied in order to reveal the effect of thermal treatment on possible recrystallisation.

The study of Cu-(Mo)SiC composite allowed to confirm that Mo layer can be an effective barrier against the Si diffusion into Cu matrix. However, there was still a significant amount of discontinuities at the Mo-Cu interface present. The interface in Cu0.8Cr-diamond composite was found (by an indirect observation) to have a very good strength. The formation of an interfacial chromium carbide, was documented.

The microstructures of Ag-diamond composite materials samples received from EPFL were characterized. Electron microscopy study of the Ag-Ni-diamond and Ag-Ni-Si-diamond composites revealed the presence of Ni at the Ag – diamond interface as isolated inclusions on the diamond side of the interface. Cracks were observed in the investigated materials at the diamond / matrix interface, however the origin of these cracks was unclear. In case of the composites with high Si content formation of silicon carbide at the interface was observed.

Results obtained for silicon-diamond preforms developed together with EMPA showed that SiC layer of different character forms on diamond depending on process parameters. Both in conventionally sintered and microwave sintered materials the layers had different morphology depending also on the diamond crystallography. The layers differ in thickness, structure and crystallographic orientation with regards to the substrate diamond. Contaminations from processing were also found in some layers. The SiC layers in conventionally sintered composites were found to be thicker than the corresponding ones in microwave sintered samples. For the composites sintered in air very little aggregates of SiC on diamond were observed. SiC nano-crystals, often in form of whiskers that tend to grow into the Si, not directly on the diamond surface, were found together with silicon oxide fibres.

2.2.3.3 Deviations from the work programme, and corrective actions taken

There were no significant deviations from the work programme. It was decided to move all activities concerning characterisation of internal stresses -TUW from WP2.1 and WP2.2 to WP2.3 in order to improve the data supply to interface development, microstutural characterisation and modelling groups.
The modelling of compounds (FZJ) could not be significantly upgraded because of insufficient material data concerning especially high temperature properties of new developed materials from WP2.1. The FZJ work was therefore concentrated on systematic high heat flux testing of divertor mock/ups with an aim to optimise the architecture and manufacturing technology according to obtained experimental results.

2.2.3.4 List of main deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.3 Subproject 3

SP3 is divided into ***4 workpackages*** (WP) and ***12 project partners*** contribute to this SP.

Partners	**WP3.1: Modelling & Benchmarking**	**WP 3.2: Nanostructured Metallic Materials**	**WP3.3: Radiation-resistant C/SiC Materials**	**WP3.4: Applied Studies, Design Rules**
UKAEA	**X**			
UOXFORD	**X**			
EPFL (SP coord)	**X**	**X**		
PSI		**X**	**X**	
AIT		**X**		
NRG		**X**	**X**	
CEA		**X**	**X**	
AREVA-D			**X**	
FZJ			**X**	
NNC				**X**
EA				**X**
AREVA-F				**X**

X: WP partners **X** **:WP** leader

2.3.1 WP 3.1: Modelling and benchmarking

2.3.1.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 48	**Duration**	48 months

Participants	**Responsibility in the WP (identify in bold WP leader)**	**Efforts (ppm)**
UKAEA	**Modelling of W interaction potentials and radiation damage**	50,0
UOXFORD	DBTT modelling and experimental DBTT benchmarking	35,1
EPFL	Bcc-W: radiation damage modelling and TEM based benchmarking	23,1

Objectives

(1) Analysis of the irradiation-induced defects in pure bcc W
(2) Modelling of the ductile-to-brittle transition temperature (DBTT) of pure bcc W

Description of work

(1) Development of a new interatomic potential for pure bcc W. Simulations of irradiation-induced defects in pure bcc W. Simulations of corresponding transmission electron microscopy (TEM) images. Comparison to experimental TEM images of irradiation-induced defects in pure bcc W.
(2) Production of experimental data for polycrystalline and nanocrystalline bcc W, for input to models of the DBTT of pure bcc W. Modelling of the DBTT of pure bcc W. Verification of the models against experimental data.

2.3.1.2 Results

During Phase 1 the WP 3.1 partners contributed to the preparation and evaluation of four Users Requirement Specifications (URSs) that were identified by the SP3 partners, which led to the preparation by the WP 3.1 (and WP 3.2) partners of a Material Requirement Specification (MRS) for tungsten-based materials. Thesnone documents was submitted for validation to the members of the Scientific Industrial Committee. In addition, all needed numerical and experimental tools for R&D activities during subsequent Phases have been assembled and tested by the three different partners. During Phase 2 Alternative Concepts (ACs) for further activities have been identified by the WP 3.1 partners. R&D activities were performed during Phases 3 and 4.

A new interatomic potential for pure tungsten has been developed, which is consistent with density functional theory predictions in terms of migration and formation energies of point defects. The new interatomic potential developed for pure tungsten has been fully parametrized for further molecular dynamics (MD) simulations of radiation damage in pure tungsten. Investigations of the formation energy of vacancy and interstitial dislocation loops in iron and tungsten were performed. The formation energy for loops was found larger than for voids, whatever the defect size. In iron square <110> loops are more energetically favourable than circular <100> loops. Vacancies move much slower in tungsten than in iron. Therefore, development of atomic displacement cascades in tungsten should be much slower than in iron.

A new method for connecting this new long-range potential to the universal short-range one has been proposed. MD simulations of atomic displacement cascades, structural defects and migration of edge and screw dislocations in pure tungsten, by using all available interatomic potentials, have been successfully performed. The melting temperature at constant pressure of tungsten was found lower with the new interatomic potential than with the former available potential by Ackland and Thetford (1987) and therefore closer to the experimental melting point. The core of edge and screw dislocations appears more extended when calculated with the new interatomic potential than with the Ackland and Thetford potential (1987). MD simulations of the core configuration of edge dislocations in pure tungsten showed that it is compact when the Ackland and Thetford (1987) interatomic potential is being used and extended when the Derlet, Nguyen-Manh and Dudarev potential (2006) is being used. It was therefore possible to move the former one using an applied stress above 100 MPa but not the latter one. A new method for setting-up asymmetric screw dislocations was introduced. Core relaxations showed that the easy asymmetric core is the most energetically favourable core configuration for screw dislocations, whatever the interatomic potential being used, while density functional theory calculations indicate that it should the easy symmetric one. This indicates that none of the tested interatomic potentials is really adequate for describing the screw dislocation core. Small interstitial dislocation loops were also successfully simulated by MD. On the other hand, neutron-irradiated specimens of pure tungsten, which were neutron-irradiated in the High Flux Reactor (HFR) at either 600 or 900°C to a dose of a few dpa, have been transported from NRG to PSI for further post-irradiation examination.

The ductile-to-brittle transition (DBT) in high purity single crystalline and polycrystalline tungsten and in commercial sintered tungsten has been investigated by means of bend tests over a wide range of temperatures and strain rates on un-notched and notched and pre-cracked specimens. At each strain rate, a DBT was observed at a well-characterised temperature. This temperature was found to be practically the same for polycrystalline and single-crystalline tungsten, although the fracture toughness measured for the polycrystalline was by a factor two higher than that measured for the single crystalline material. The strain-rate variation of the DBT temperatures for all the tungsten materials investigated fits an Arrhenius law with an activation energy of approximately 1.05 eV. Chemically vapour deposited tungsten was found brittle up to the maximum investigated temperature of 970°C. Fluorine at the grain boundaries seems to be responsible for that behaviour. Dislocation-based modelling of the BDT in single crystalline tungsten was performed, and modelling results have been compared to results of experiments. A very good agreement was found. Electron back-scattered diffraction (EBSD) studies were performed in the aim to map the strain field around crack tips in pure single crystalline tungsten. It was found that crystal rotations in the plane perpendicular to the bottom of the notch dominate over rotations in other planes. For specimens deformed above the DBT temperature, in-plane crystal rotations revealed a plastic zone extending from the crack tip in {110} planes. The size of the plastic zones and the dislocation densities within them (hence the lattice rotations observed by EBSD) were determined on the basis of the applied loading history, internal stress and velocity-stress-temperature relationship for the dislocations.

A comprehensive model that explains the anomalous radiation damage effects occurring in iron and steels at elevated temperatures was developed. It showed 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. This work revealed for the first time the fundamental link between the nature of radiation damage observed experimentally at temperatures approaching 500°C, the diffusionless phase transitions occurring in iron and iron-based alloys at about 900°C, the anisotropic elastic properties of iron and iron-based alloys that become particularly significant at these elevated temperatures, magnetic fluctuations, and the loss of mechanical strength of steels at elevated temperatures.

2.3.1.3 Deviations from the work programme, and corrective actions taken

none

2.3.1.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.3.2 WP 3.2: Nanostructured metallic materials

2.3.2.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
PSI	**Characterization of ODS steels; post irradiation experiments**				42,1
AIT	Processing of ODS steel specimens				15,1
CEA	Processing of ODS steels, neutron irradiation and post irradiation experiments				77,2
EPFL	Processing of ODS steels and refractory-base materials; post irradiation experiments				57,9
NRG	Neutron irradiation and post irradiation experiments				28,7

Objectives

Development of high temperature, radiation-resistant materials, including nanostructured oxide dispersion strengthened (ODS) steels, ODS ferritic steels and W-based alloys.

Description of work

(1) Development of new high temperature materials: nanostructured ODS steels, ODS ferritic steels and W-based alloys.
(2) Characterization of the developed materials: microstructure, thermal and mechanical properties.
(3) Irradiations of the developed materials: ion irradiations, neutron irradiation, irradiation in SINQ.
(4) Post-irradiation evaluation (PIE) of the irradiated materials.

2.3.2.2 *Results*

During Phase 1 the WP 3.2 partners contributed to the preparation and evaluation of four Users Requirement Specifications (URSs) that were identified by the SP3 partners, which led to the preparation by the WP 3.2 partners of two Material Requirement Specifications (MRSs) for oxide dispersion strengthened (ODS) steels and tungsten-based materials, respectively. These documents were submitted for validation to the members of the Scientific Industrial Committee. In addition, all needed numerical and experimental tools for R&D activities during subsequent Phases have been assembled and tested by the five different partners. During Phase 2 Alternative Concepts (ACs) for further activities have been identified by the WP 3.2 partners. R&D activities were performed during Phases 3, 4 and 5.

<u>Characterization of a commercial ODS ferritic steel:</u> [111]-oriented micropillars of the annealed PM2000 oxide dispersion strengthened (ODS) ferritic steel were deformed in compression tests. As the annealed PM2000 steel contains very large grains, it was possible to prepare small tensile samples from that material in such a way that they were also parallel to the [111] direction. A very good agreement between the yield stresses measured from micropillar tests and tensile tests was found.

<u>Manufacturing and characterization of ODS ferritic steels:</u> ODS reduced activation ferritic (RAF) steels, with the chemical composition of Fe-(12-14)Cr-2W-0.3Ti-0.3Y_2O_3, have been produced by mechanically alloying either Fe, Cr, W and Ti elemental powders or a pre-alloyed powder with 0.3Y_2O_3 powder particles and compacting the mechanically alloyed powder either by hot isostatic pressing (HIPping) or by hot extrusion. The microstructure and

mechanical properties of the produced materials have been characterized mostly by means of scanning and transmission electron microscopy observations, Vickers microhardness measurements, tensile and Charpy impact tests. It was found that the use of a hydrogen atmosphere, instead of argon, during mechanical alloying allows reducing significantly the oxygen content in the materials, leading to improved mechanical properties. Both materials exhibit a bimodal microstructure, made of coarse grains and smaller ones, a ductile-to-brittle transition temperature (DBTT) close to 30°C (at least when prepared from elemental powders and compacted by HIPping), high tensile strength and good ductility. The 14Cr material appears slightly stronger and less ductile than the 12Cr material at all test temperatures. It was also attempted to improve the microstructure and mechanical properties of the ODS ferritic steels by applying thermo-mechanical treatments (TMTs), such as hot rolling, hot pressing or cold pressing. It was found that the microstructure is significantly improved by the TMTs, as well as the Charpy impact properties. For instance, the DBTT of 14Cr steels prepared using a pre-alloyed powder was found to decrease from about 160°C down to about 10°C when the steels are submitted to hot extrusion (instead of HIPping), hot rolling and heat treatment.

<u>Development and characterization of W-base materials:</u> Pure tungsten and W-1%La_2O_3 materials have been submitted to high-speed hot extrusion at Warsaw University of Technology (SP1 partner) in an attempt to improve the ductility of these materials. Pure tungsten was found too brittle for the high-speed hot extrusion process. This process allowed plastic deformation of the W-1%La_2O_3 material but no significant improvement in the DBTT value. Note that all activities on tungsten-base materials have been achieved on Month No. 48.

<u>Severe plastic deformation techniques:</u> The potentiality of using equal channel angular pressing (ECAP) for producing nanostructured ODS ferritic steels has been demonstrated. Following its application to the commercial PM2000 ODS ferritic steel, it was applied to the new 14 Cr ODS RAF steel developed within this work package, in an attempt to improve the ductility of these materials. Unfortunately, no significant decrease of the grain size was obtained by applying this method at temperatures above 500°C. It was therefore attempted to apply high-pressure torsion (HPT) to both types of ODS ferritic steel. Smaller grain size materials with a higher microhardness were obtained by means of HPT. However, the quantities that can be produced by using this technique are very small. The potential use of the other available severe plastic deformation techniques was evaluated.

<u>Neutron irradiations:</u> Various types of specimens have been prepared by a number of ExtreMat partners from the most promising metallic materials developed within the project. Irradiation matrices have been defined, and the irradiation capsule has been designed. The irradiation rig has been then mounted. Due to the refurbishment of the high flux reactor (HFR) for operation with lower enriched uranium fuel and an unexpected lack of professional staff at NRG for a certain period of time, the two planned neutron irradiation campaigns were started with a few months delay in February and May 2008, respectively. Unfortunately, due to a subsequent corrosion-produced leakage problem, the neutron irradiations were stopped in summer 2008. The HFR was repaired and re-started in February 2009. The ExtreMat I irradiation (low-dose, low temperature neutron irradiation) ended on April 26, 2009. Specimens were irradiated at 300 and/or 550°C. The ExtreMat II irradiation (high-dose, high temperature neutron irradiation) ended on September 08, 2009. Specimens were irradiated at 600 and/or 900°C to about 4 dpa (in steels). Both irradiations included ceramic and metallic specimens. 493 specimens in total have been neutron-irradiated in the HFR.

<u>Post-irradiation experiments:</u> Post-irradiation experiments (PIEs) on metallic materials that were neutron-irradiated in the HFR have been shared between NRG, EPFL, CEA and FZJ.

All neutron-irradiated metallic specimens are still in the cooling phase, i.e., they are still too radioactive to be tested. The transports from NRG to the PSI of TEM and Charpy impact specimens of metallic materials, from NRG to the CEA of small angle neutron scattering (SANS) specimens of ODS RAF steels, and from NRG to FZJ of high heat flux specimens of metallic materials, are being organized by the respective Nuclear Safety Authorities. The transports should take place by the end of 2010.

Post-irradiation tensile testing of specimens of MA956 and MA957 ODS ferritic steels, which were neutron-irradiated in the Phénix reactor at temperatures in the range of 410-550°C to various doses up to 80 dpa, revealed significant irradiation-induced hardening and reasonable ductility. Results have been analyzed in terms of irradiation-induced α' precipitation.

Microstructural analyses of the commercial PM2000 ODS ferritic steel in the extruded/annealed condition were performed following creep experiments at various temperatures under ion irradiation. It was found that irradiation creep is important up to about 680°C. At higher temperatures thermal creep becomes predominant. At 400°C an irradiation-induced ordered phase (Fe_3Al) was evidenced. The existence of 1/2<111> interstitial dislocation loops in the ion irradiated PM2000 steel was confirmed. The commercial PM2000 ODS ferritic steel, before and after ECAP, has been also irradiated with helium ions to a dose of about 0.7 dpa. The irradiation-induced hardening (increase in hardness) was found larger for ECAP PM2000 than for annealed PM2000. Generally speaking, there is no significant difference in the irradiation behaviours of annealed PM2000 and ECAP PM2000, The irradiation creep data obtained for the helium-implanted, commercial PM2000 ODS ferritic steel were compared to data reported in the literature for other ODS ferritic steels as well as for ferritic and ferritic/martensitic steels. It was found that the size of dispersoids has no significant effect on irradiation creep and that the irradiation creep of ODS steels is comparable to that of non-ODS materials.

<u>Industrial evaluation:</u> The materials requirements as well as the candidate materials for Generation IV reactors, in particular for very high temperature reactors (VHTRS), have been reviewed. The use of ODS steels at irradiation temperatures below 600°C appears limited, as the oxide particles have no significant effect on irradiation creep at low temperatures but only on thermal creep. Small-grained ODS steels produced by severe plastic deformation are of few interest, as they exhibit significant grain growth at elevated temperatures.

2.3.2.3 Deviations from the work programme, and corrective actions taken

Due to some delays in the achievement of irradiation experiments, due to technical problems, the transports of neutron-irradiated TEM, Charpy impact, SANS and high heat flux metallic specimens from NRG to the PSI, the CEA and FZJ are just being organized. Then, the activity of each specimen will be measured at the PSI, the CEA, and FZJ, and testing plans will be prepared accordingly. Depending on their activity, the specimens should be available for PIEs in 2011-2012.

2.3.2.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.3.3 WP 3.3: Carbon and SiC materials

2.3.3.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
NRG	**High temperature neutron irradiation, post irradiation experiments**				39,4
AREVA - D	VHTR materials characterization				12,0
CEA	High temperature neutron irradiation, post irradiation experiments				28,0
FZJ	High heat flux experiments on neutron irradiated materials				26,2
PSI	SiC-base and C-base materials characterization, post irradiation experiments				16,8

Objectives

Development of high purity, nanometric SiC particles powder and of high purity, dense, nanostructured, monolithic SiC. Characterization, irradiation and post-irradiation evaluation of C-based and SiC-based materials developed within the whole project.

Description of work

(1) Development of high purity, nanometric SiC particles powder for preparation of high quality, advanced SiC-based materials within SP1.
(2) Development of high purity, dense, nanostructured, monolithic SiC.
(3) Characterization of C-based and SiC-based materials: microstructure, physical and mechanical properties.
(4) Irradiations of C-based and SiC-based materials: ion irradiations, neutron irradiation, irradiation in SINQ.
(5) Post-irradiation evaluation (PIE) of the irradiated materials.
(6) Assessment of requirements for high temperature devices and components.

2.3.3.2 Results

During Phase 1 the WP 3.3 partners contributed to the preparation and evaluation of four Users Requirement Specifications (URSs) that were identified by the SP3 partners, which led to the preparation by the WP 3.3 partners of two Material Requirement Specifications (MRSs) for nanostructured, monolithic ceramic materials and carbon fibre reinforced ceramics, respectively. These documents were submitted for validation to the members of the Scientific Industrial Committee. In addition, all needed numerical and experimental tools for R&D activities during subsequent Phases have been assembled and tested by the five different partners. During Phase 2 Alternative Concepts (ACs) for further activities have been identified by the WP 3.3 partners. R&D activities were performed during Phases 3, 4 and 5.

Note that only high purity, nanometric SiC powder particles destined to the preparation of high quality, advanced SiC-based materials within SP1 and high purity, dense, nanostructured, monolithic SiC have been produced within this work package. Other C-based and SiC-based materials have been developed within SP1.

<u>Development of high purity, nanometric SiC powder particles:</u> Various powder batches have been produced by laser pyrolysis. A powder batch of SiC nanoparticles was delivered to INCAR (SP1 partner). Powder batches of SiC/B nanoparticles have been delivered to CEIT

and University of Alicante (SP1 partners). Powder batches of SiC/Ti nanoparticles were found to contain a too high level of oxygen (about 8 wt.%).

Development of high purity, dense, nanostructured, monolithic SiC: The optimal manufacturing route has been identified, i.e., sintering without additives. A nanostructured, monolithic SiC material with a density of about 75% has been produced. The microstructure is made of a dense skeleton of micrometric grains with large porosities, which contain agglomerated nanoparticles.

High heat flux testing: High heat flux experiments were performed on unirradiated and available irradiated specimens, in the aim to define testing parameters to be used for further high heat flux experiments on the specimens neutron-irradiated within ExtreMat. In particular, high heat flux testing of unirradiated and available irradiated specimens of various tungsten-base materials (pure tungsten, W-1.3%Mo, W-0.2%Re, W-1%La_2O_3) has been performed. Transient thermal events similar to ITER ELM events (t = 5 ms, up to 1.3 GW/m^2) led to severe cracking of all materials (except W-0.2%Re), characterized by primary cracks along the grain boundaries and secondary cracks located inside the grains. No significant irradiation-induced degradation of weight loss and particle emission was evidenced.

Neutron irradiation experiments: Various types of specimens have been prepared by a number of ExtreMat partners from the most promising ceramic materials developed within the project. Irradiation matrices have been defined, and the irradiation capsule has been designed. The irradiation rig has been then mounted. Due to the refurbishment of the high flux reactor (HFR) for operation with lower enriched uranium fuel and an unexpected lack of professional staff at NRG for a certain period of time, the two planned neutron irradiation campaigns were started with a few months delay in February and May 2008, respectively. Unfortunately, due to a subsequent corrosion-produced leakage problem, the neutron irradiations were stopped in summer 2008. The HFR was repaired and re-started in February 2009. The ExtreMat I irradiation (low-dose, low temperature neutron irradiation) ended on April 26, 2009. Specimens were irradiated at 300 and/or 550°C. The ExtreMat II irradiation (high-dose, high temperature neutron irradiation) ended on September 08, 2009. Specimens were irradiated at 600 and/or 900°C to about 4 dpa (in steels). Both irradiations included ceramic and metallic specimens. 493 specimens in total have been neutron-irradiated in the HFR.

Other irradiation experiments: SiC and SiC/SiC specimens have been irradiated in the Swiss Spallation Neutron Source (SINQ facility) with a mixed spectrum of high-energy protons and spallation neutrons. Testing of reference (unirradiated) specimens was performed by means of three-points bend tests, small ball punch tests and Extended X-ray Absorption Fine Spectroscopy (EXAFS). On the other hand, specimens of SiC were implanted with helium ions having an energy up to 24 MeV, to various doses and helium concentrations up to 0.147 dpa and 2451 appm He, respectively. Helium-implanted specimens were subsequently characterized using EXAFS. The Fourier transform of EXAFS spectra revealed a strong decrease of the coordination number of atoms in the second shell, corresponding to the next neighbour silicon atoms, with increasing irradiation dose. The EXAFS spectra of helium-implanted specimens were simulated by means of *ab initio* multiple scattering calculations, assuming that helium platelets have been produced by the implantation. The experimental and simulated pseudo-radial distribution functions showed similar tendencies as a function of irradiation dose.

Post-irradiation experiments: Post-irradiation experiments (PIEs) on ceramic materials that were neutron-irradiated in the HFR have been shared between NRG, the CEA and FZJ. The physical properties of neutron-irradiated ceramic specimens with a low to moderate dose rate have been measured at NRG. These included general integrity (photography), mass loss (micro balance), laser flash analysis (thermal diffusivity), CTE (Coefficient of Thermal Expansion), and dynamic Young's modulus (by velocity of sound). The neutron-irradiated ceramic specimens with a medium to high dose rate are in still in the cooling phase, i.e., they are still too radioactive to be tested. The transports from NRG to the CEA of transmission electron microscopy (TEM) specimens of nanostructured, monolithic SiC and from NRG to FZJ of high heat flux specimens of various ceramic materials, which were neutron-irradiated in the HFR, are being organized by the respective Nuclear Safety Authorities. The transports should take place by the end of 2010. Then, the activity of each specimen will be measured at the CEA and FZJ, and testing plans will be prepared accordingly.

Industrial evaluation: Industrial evaluation of the materials developed within SP3 and neutron-irradiated in the HFR was performed with respect to their potential applications. Data requirements for industrial application, R&D, manufacturing and design have been identified. In particular, the materials requirements as well as the candidate materials for Generation IV reactors, more especially for very high temperature reactors (VHTRS), have been reviewed. It seems that advanced SiC/SiC_f materials are not needed for current VHTR designs (10 dpa, < 950°C) and have not yet been proven to be gas tight enough for use as cladding materials for gas cooled fast reactors (GFRs).

2.3.3.3 Deviations from the work programme, and corrective actions taken

Due to some delays in the achievement of irradiation experiments, due to technical problems, the transports of neutron-irradiated TEM and high heat flux specimens from NRG to the CEA and FZJ are just being organized. Depending on their activity, the specimens should be characterized in 2011-2012. The rest of irradiated ceramic specimens will be tested at NRG when their dose rate will permit it.

2.3.3.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.3.4 WP 3.4: Applied studies and design rules

2.3.4.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 48	**Duration**	48 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
NNC	**Evaluation of results, data base, correlation to design rules (e.g. KTA-rules).**				28,0
EA	Evaluation of results, data base, correlation to design rules (e.g. KTA-rules).				11,0
AREVA-F	Evaluation of results, data base, correlation to design rules (e.g. KTA-rules).				8,8

Objectives

Industrial evaluation and assessment of database requirements for the materials to be investigated within the subproject SP3 with respect to their envisaged applications.

Description of work

(1) Review database requirements for the materials and applications chosen for investigation within SP3. Make outline proposals based on approaches used in Fission and Fusion industries. Provide industrial review and input to the experiments and research activities being undertaken.
(2) Develop needs for the short term to record important results and requirements from the ongoing experiments. Make proposals for the database specification. Provide ongoing industrial input to the experiments and research activities being undertaken.
(3) Formalise the database specification for the benefit of the longer-term industrialisation. Make recommendations with respect to future investigations for continued industrial development.
(4) Provide specialist input to modelling of composite materials through sub-contract with University of Manchester, UK.

2.3.4.2 Results

During Phase 1 the WP 3.4 partners contributed to the preparation and evaluation of four Users Requirement Specifications (URSs) that were identified by the SP3 partners, which led to the preparation by the SP3 partners of four Material Requirement Specifications (MRSs) for oxide dispersion strengthened (ODS) steels, tungsten-based alloys, nanostructured, monolithic ceramic materials and carbon fibre reinforced ceramics, respectively. These documents were submitted for validation to the members of the Scientific Industrial Committee. During Phase 2 pre- and post-irradiation needed information for C-based materials, SiC-based materials, nanostructured ODS steels, ODS ferritic steels for high temperature applications and W-based alloys, has been identified by the WP 3.4 partners. Corresponding results to be obtained have been also identified, as well as the selection criteria and the potential spin-offs. Alternative Concepts (ACs) for further WP 3.4 activities have been defined, and WP 3.4 partners also reviewed the ACs prepared within the other SP3 work packages. Activities by the WP 3.4 partners were pursued during Phases 3 and 4.

Industrial evaluation of the materials developed within SP3 with respect to applications chosen for investigation has been continuously performed. In particular, SP3 materials showing good industrial potential for fission and fusion applications have been reviewed. A technical report has been issued, which summarizes the future needs in terms of experiments for materials to be used for very high temperature reactor (VHTR) core structures and core components. This document has been submitted to ExtreMat partners for comments. The longer-term industrialisation of the materials developed within SP3 was also addressed. A review of ceramic materials developed within ExtreMat has been done with respect to their potential applications in VHTRs. It was pointed out that several types of C-based materials and coatings could be eventually used for the different core components of VHTRs because their functions will be different. In addition, a review of existing standards for experimental testing of non-metallic materials has been also performed. Manchester University contributed to modelling of composite materials to support the neutron irradiation experiments and evaluation.

The work also addressed activities related to the development of a database to store the test results from the neutron irradiation programme carried out in the high flux reactor (HFR) and to share these amongst the ExtreMat partners. The database stores/will store all aspects of the two neutron irradiation campaigns, materials, specimens, test methods and results, and is/will be available to the ExtreMat partners via the internet and BSCW system. Extensive coordinated action has been put on this activity so as to fulfil the goals, which included formalisation of a reference datasheet for each specific research area, the organisation and arrangement of a database structure, and implementation of the database. Database requirements and functional specifications have been reviewed. Excel templates for recording results of pre- and post-irradiation examinations have been prepared and are/will be available on the BSCW site. Database usage has been presented to the ExtreMat partners, with an on-line demonstration via internet connection. Access rights have been defined. Future maintenance of the database has been extensively discussed.

The following coordination meetings of the EU-funded FP6 projects related to SP3 have been attended: RAPHAEL, GCFR, EUROTRANS, ELSY and EISOFAR. The project ELSY, from which new materials requests for liquid metal cooled reactors (LMRs) have been obtained, was found of special interest.

2.3.4.3 Deviations from the work programme, and corrective actions taken

none

2.3.4.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.4 Subproject 4

SP4 is divided into ***4 workpackages*** (WP) and ***14 project partners*** contribute to this SP.

Partners	**WP4.1: Materials Systems Engineering**	**WP 4.2: Integrated Diffusion Barriers**	**WP 4.3: Bonding of Heterogeneous Materials**	**WP4.4: Environmental Tests for Industrial Applications**
AEN (SP coord)	**X**	**X**	**X**	
ATL		**X**		
CEA	**X**		**X**	
DEMOKRITOS			**X**	
FZJ				**X**
IMSAS	**X**		**X**	
INASMET			**X**	
IPP	**X**	**X**		**X**
IPP-CZ			**X**	
NNC	**X**			
PLANSEE	**X**			
POLITO			**X**	
SIEMENS	**X**		**X**	**X**
TUW			**X**	

X: WP partners **X** **:WP** leader

2.4.1 WP 4.1: Materials Systems Engineering

2.4.1.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 10	**Duration**	10 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
SIEMENS	Systems requirement Co-ordination				3,0
AEN	System requirements for joining (fusion applications)				2,7
CEA	System requirements for joining (electronic applications)				2,0
IMSAS	Thermal management aspects of compounds				2,0
IPP	System requirements for fusion application				2,0
NNC	Nuclear systems requirements				1,2
PLANSEE	System requirements for thermal management in electronic applications				1,3

Objectives

The purpose of WP4.1 is technical and scientific supervision of other WPs in the whole project, so that needs arising from joining, integrated diffusion barriers deposition and, in general, from practical 'industrial exploitability', are taken into account during new materials development. Moreover it will improve information flow and harmonize RTD activities among all the different WPs in the project. Due to its special nature WP4.1, will require a particular commitment from industrial participants.

The role of WP4.1, i.e. its objectives, are particularly important during the first phases of the project (good integration of the project has to be created from the beginning, otherwise time and efforts will be wasted).

Phase 1: Definition of "system requirements" (i.e. needs specially arising from the fact that new materials will have to be coated and/or joined in order to be practically exploited) for all the WPs.

Phase 2: Ensuring that "system requirements" are duly taken into consideration during different concepts evaluation and the selection of proven ones.

Phase 3: Continuous technical and scientific follow-up of all WPs concerning their compliance with the "system requirements".

Description of work

Close interaction with WPs 1.1, 1.2, 1.3, 2.1, 2.2, 3.2, 3.3, 4.2, 4.3, 4.4.

Phase 1: The "system needs" of the users of joined compounds / components and of diffusion barriers will be collected, producing first "system requirements" to be given as input to other WPs. The system requirements will be supplemented with other relevant scientific information and validated by the users (Scientific Industrial Committee).

Input to other WPs as regards system requirements will start as soon as possible too.

Phase 2: Participation to the alternative concepts evaluation performed by other WPs, providing input concerning the system requirements aspects. Elaboration, together with other WPs, of the methods and procedures to be followed during R&D phase (phase 3) for monitoring of fulfilment of system requirements.

2.4.1.2 Results

Specific contribution of WP4.1 was preparation of the User Requirements Specifications in phase 1 and Alternative Concepts verification from system requirements point of view in phase 2. No specific contribution was expected during the remaining part of the project, because the continuous follow-up from the point of view of system requirements shows a large overlapping with monitoring and validation activities performed by Scientific Industrial Committee members. As a consequence WP4.1 has been kept in stand-by conditions during the remaining part of the project.

2.4.1.3 Deviations from the work programme, and corrective actions taken

No significant deviations from the work programme happened. The fact that this WP has been kept in stand-by after phase 2 should not be considered as a real deviation from its work programme: in fact continuous monitoring of the project as regards system requirements was planned and it has been performed, through Scientific Industrial Committee activities.

2.4.1.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.4.2 WP4.2: Integrated Diffusion Barriers

2.4.2.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
IPP	Development of thin film interfaces which prevent the transport of hydrogen through the interface (up to 650°C) and/or which prevent the diffusion of detrimental species to the bonded interface and promote adhesion (up to 1050 °C during the joining process).				72,0
AEN	Evaluation of diffusion barriers and adhesion promoter films in joining for plasma facing compounds and gas turbine high temperature components (spin-off).				58,3
ATL	Evaluation of the possibility for industrial up-scaling of ACs and supply of sample coatings for investigation by other participants.				32,6

Objectives

Main: The **overall objective** is to develop, in a phased approach, buried thin film interfaces, which prevent the transport of hydrogen through the interface (up to 650°C) and/or which prevent the diffusion of detrimental species to the bonded interface (up to 1050 °C during the joining process), and/or which improve wetting by brazing alloys and/or which withstand residual stresses instead of fragile protective materials.

Objective 1: Diffusion barriers, as buried thin films, able to prevent hydrogen diffusion in plasma facing compounds for nuclear fusion reactors application (in order to avoid tritium contamination).

Objective 2: Coatings, as buried thin films, able to improve wetting (therefore also adhesion) by brazing alloys and/or able to prevent diffusion of detrimental species from the materials to be joined to the joint zone. Their evaluation through the production and thermal shock testing of PFC compounds.

Objective 3: Thick layers (up to 2 mm), deposited as CVD coatings on C-based protective materials or on CuCrZr tubes, with the purpose of withstanding residual stresses produced during joining to heat sinks for the formation of plasma facing compounds. Their evaluation through the production and thermal shock testing of PFC compounds.

Objective 4: Development towards industrialization and production at mock-up scale of Erbia films as Tritium diffusion barriers and of Si_3N_4 and TiC films as wetter promoters in brazing of plasma facing compounds (delivered to WP4.3 and WP4.4).

Objective 5: Development towards industrialization and production at mock-up scale of coatings on diamond particles, for improved thermal exchange with metal matrix in heat sinks (delivered to SP2).

Objective 6: Production of active cooled plasma facing compound mock-ups including wetter promoters and/or thick CVD W interlayers, for testing in conditions relevant for nuclear fusion applications; on the basis of the achieved results, the industrial applicability of wetter promoters and/or thick CVD W interlayers will be evaluated.

Objective 7: Design (& implementation where appropriate) of increased size CVD furnaces and sputter deposition equipments (industrial scaling) for developed processes.

Description of work

The barrier interfaces to be developed are a vital element to the required functionality of components for any application in hydrogen/tritium environment and for the high heat flux application of bonded protection/heat sink systems. The development covers barrier films, buried at the protection/heat sink interface. Film materials are metal, metal nitrides and carbides and rare earth oxides and tailored refractory alloys with controlled nanostructure.

Activity 1: Development of deposition techniques for diffusion barriers against tritium permeation, including the deposition of W overlayers and the delivery of samples for environmental characterisation. Deposition of CVD and sputtered hydrogen diffusion barriers and their characterisation. Development and production for testing of erbia diffusion barriers on steel substrate, including duplex W/erbia layers.

Activity 2: Development of deposition techniques for thin films able to prevent diffusion of detrimental species and/or to improve wetting by brazing alloys, including brazing trials and characterisation of relating compounds. Deposition of CVD Si_3N_4 films and of sputtered TiC films on C-based tiles, to be delivered for brazing tests and for compounds and active cooled PFC mock-ups production.

Activity 3: Development of deposition techniques for thick interlayers for improved joints strength, including brazing trials and characterisation of relating compounds.

Activity 4: Deposition of thick W layers both on C-based materials and on CuCrZr heat sinks and their characterisation, including brazing tests.

Activity 5: Production, examination and testing of compounds with thick and thin films, including active cooled plasma facing mock-ups.

Activity 6: Development and application of CVD W and SiC coatings to diamonds for improvement of interface performance in heat sinks. Produce coated material for incorporation in concept mock-up parts

Activity 7: Contribution to final evaluation of the industrial applicability of the successful concepts; design of industrial upscaling of deposition facilities and processes for the chosen concepts.

Activity 8: Industrialization of CVD Silicon Nitride coatings for the silicon processing industry.

2.4.2.2 Results

The summary of activities and results achieved in particular by **IPP** is the following:

In three different growth rate ranges, the control of the stoichiometry of TiC coatings was achieved. Compressive stress was tentatively identified as a problem. Introducing the argon gas flux as a deposition parameter of the magnetron sputter deposition device, layer stress measurements have been carried out to solve that problem. Nearly stress-free TiC coatings could be achieved with increased argon gas flux and discharge power ranges.
Within the collaboration of IPP Garching with TUW, first TiN films were analysed withrespect to their composition by Rutherford Backscattering.
In order to improve the wettability of pure copper on stoichiometric TiC it was figured out that the titanium content has to be increased. On the other side a copper-titanium alloy has wetted both the uncoated and the TiC coated graphite substrate, but obtaining a low contact angle occurred faster with a TiC layer.

Wetting experiments of Copper-ABA® on TiCx layers have been performed. An improved wettability at increased titanium content (optimum: pure Ti) have been adopted. The wetting droplet surface was compact and dense at TiC0.7 to TiC0.2. Therefore, the wetting optimum is $TiC_{0.2}$. Furthermore first shear strength tests with brazed compounds have been carried out with graphite substrates. The failure occurred in the fine grain graphite (too brittle) and not in the joint. Thereupon a comparison of different TiCx interlayers was impossible.
New wetting experiments of Copper-ABA® on TiC_x layers have been performed includingstoichiometric TiC coatings and by increasing the test amount per sample. An improved wettability was measured at the composition range close to stoichiometric TiC and close to pure titanium. Nevertheless, there are still some problems indicated by widely spread contact angle curves. The results of the shear strength experiments with brazed Cu-CFC-Cu samples using different TiC_x interlayers were unsatisfactory. A distinction of the effect of the various coating stoichiometries is not possible.
Wetting experiments of Copper-ABA® on TiC_x layers have been performed using a newself-constructed contact angle measurement device. Now, reproducibility of wetting results could be obtained by improving the wetting experiment conditions. The slight trend of a good wetting behaviour at both, stoichiometric TiC and Ti coatings, was confirmed. Additionally, shear strength experiments with brazed Cu-graphite-Cu samples using different TiC_x coatings have been performed. Former tests with CFC instead of graphite revealed that a comparison between TiC_x films is not possible. But values measured at TiC_x-coated graphite – copper interfaces showed a trend which are in correlation to the wetting test results.
New shear strength experiments with oxygen plasma treated and brazed Cu-CFC-Cu samples using different TiC_x coatings have been performed. Former tests with CFC without treatment revealed that the shear strength values of all TiC_x films are at the same level of the uncoated case, except CVD-TiC. Now, the oxygen plasma treatment, which induced physical and chemical erosion and resulted in a roughening of the CFC surface before coating and brazing, show an increase of shear strength by a factor of 2. Tensile tests with oxygen plasma treated and TiC_x coated CFC/Cu compounds have been performed.
For the following high heat flux tests with mock-ups using TiC_x coatings as wetting promoters, temperature calculations have been performed.
Comparative permeation measurements of vacuum-arc-deposited erbia and alumina coatings on Eurofer yielded indications that the permeation reduction factor of erbia is of similar magnitude as that of alumina. The basic applicability of alumina as integrated hydrogen diffusion barrier in a thin tungsten/alumina/Eurofer compound was demonstrated by sputter-depositing 1 µm of tungsten on top of an alumina layer. Millimeter-range CVD tungsten coatings on Eurofer with micrometer copper and silica interlayers did not show sufficient adhesion. XRD analysis of first CVD-erbia coatings (delivered by ATL) shows the crystal structure of body-centered cubic Er_2O_3. Columnar structures could probably inhibit good permeation test results.

The summary of activities and results achieved in particular by **AEN** is the following:
Ansaldo's main activities in WP4.2 during the whole project duration have been focused on evaluation of the suitability of wetter promoters and thick CVD W layers for the production of plasma facing components through industrial joining processes, in particular high temperature vacuum brazing to heat sinks with the use of low CTE interlayers.
In particular Ansaldo performed:

- Evaluation through brazing, in the monoblock configuration, of thick CVD W layers, planned to act as low CTE stress bearing interlayers; both W layers deposited inside graphite and CFC monoblock tiles, as well as layers deposited on the external surface of CuCrZr tubes, have been evaluated. Even though some progress with respect to initial results has been achieved, these concepts didn't show industrial applicability in plasma facing components.

- Diffusion heat treatment of Re films deposited on nickel based alloy has been performed and a CrRe layer has been produced, potentially able to perform as diffusion barrier for NiCoCrAlY+M coatings.
- Brazing tests on TiC_x, TiN_x and Si_3N_4 wetter promoters, contributing to their development until the choice of their best compositions and thickness.
- Production and thermal shock tests of compounds with graphite and CFC tiles coated with the chosen wetter promoters.
- Production of active cooled PFC compounds with CFC tiles coated with the chosen wetter promoters.

Ansaldo's main contribution to ExtreMat project as regards WP4.2 activities has been the evaluation of suitability of wetter promoters and stress bearing thick interlayers by CVD for production of plasma facing components through industrial joining processes, in particular brazing.

In particular, in the last phase of the project, devoted to demonstration of industrial applicability of research results, the following results have been reached:

The evaluation of the CVD thick W layers deposited inside the bore of C-based tiles has been completed; a 3 tiles mock-up has been brazed, in mono-block configuration. Good adhesion has been achieved only in limited regions, whereas much larger zones show detachments. As a consequence CVD thick W as stress bearing interlayer for brazing in mono-block configuration has been discarded.

The evaluation of TiN_x sputtered films as wetter promoters has been concluded; compounds with CuCrZr blocks and 2 mm thick Mo interlayers have been produced and successfully tested for thermal shock resistance. $TiN_{0.3}$ sputtered films are accepted as wetter promoters, with thickness $\geq$ 100 nm.

The evaluation of CVD Si_3N_4 films as wetter promoters has been concluded too; compounds with CuCrZr blocks and 2 mm thick Mo interlayers have been produced and successfully tested for thermal shock resistance. CVD Si_3N_4 films are accepted as wetter promoters, with thickness about 3 μm.

Compounds with CFC and graphite tiles, coated on 2 surfaces by sputtered TiC_x and brazed in between copper blocks, have been produced, for mechanical characterization performed by IPP. Different geometries have been brazed, including large cubic blocks (joint surface 50 x 50 mm).

On the basis of above mentioned results achieved on compounds, the preparation of active cooled mock-ups started.

5 active cooled mock-ups, with 5 different TiC_x wetter promoters and geometry suitable for high heat flux testing in GLADIS facility, have been brazed using materials supplied by IPP.

Preparation of 3 active cooled flat tile mock-ups for HHF tests at FZJ has been completed: first one included $TiN_{0.3}$, TiC and Si_3N_4 coated tiles, one tile per each wetter promoter; the second one included the same choice of wetter promoters, but uses W_f/Cu MMC as low CTE interlayer instead of Mo; the third one included a Ti-coated tile instead of the TiC-coated one. Just the second one was suitable for HHF tests, because the first and the third ones failed during minor machining immediately after brazing; in any case HHF behaviour has been quite poor and failure happened in the CFC.

The summary of activities and results achieved in particular by **ATL** is the following:

ATL was originally involved in 2 of the 4 subprojects and this led to discussions and collaborations with a large number of companies, institutions and universities, many of whom it has had no contact with before. This was in fact so productive that through this discussion ATL took on an additional contribution in SP2 once SP1 activities were complete.
On the technical and commercial front there have been many benefits. Some of these are listed below:

- Of the 10 coatings that have researched, the results from eight of these have been used in some way within commercial projects outside ExtreMat.
- Two of the four coatings that were completely new to ATL have been taken to a development level that has given us confidence to offer them as CVD coatings on our products list.
- Five of the previously existing ATL coating processes have been used in new applications or have been applied to previously unused substrates. This has extended our knowledge base with our current coating materials and made them easier to sell into a wider variety of applications.
- One coating has been successfully tested for thousands of hours in ongoing corrosion tests with a major industrial company. It looks likely that this will lead to some level of ongoing production.

2.4.2.3 Deviations from the work programme, and corrective actions taken

No significant deviations from the work programme happened. Some delays in the preparation and environmental tests of compounds have been recovered during the first 6 months of the fifth reporting period.

2.4.2.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.4.3 WP4.3: Bonding of Heterogeneous Materials

2.4.3.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 48	**Duration**	48 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
AEN	Improvement and/or development of joining techniques based on high temperature brazing for plasma facing compounds in fusion applications				86,6
CEA	Development of isolation layers for electronic compounds, based on the bonding of a ceramic layer on a metallic or composite substrates				47,7
DEMOKRITOS	Development of joining between ceramics (C/SiC, SiC/SiC) with metals (PM 1000, PLANSEE) combining sputtering (DEMOKRITOS) and diffusion bonding (INASMET) techniques for aerospace applications				46,2
IMSAS	Bonding of Cu-W high temperature heat sink with W based plasma facing compound using functionally graded interlayers and hot isostatic pressing				35,5
INASMET	Development of joining between ceramics (C/SiC, SiC/SiC) with metals (PM 1000, PLANSEE) combining sputtering (DEMOKRITOS) and diffusion bonding (INASMET) techniques for aerospace applications				36,9
IPP-CZ	Development of plasma sprayed W-based layers and W+Cu bonding interlayers with high thermal shock resistance and high thermal conductivity for plasma facing compounds. Development of plasma sprayed electrically insulating interlayers for electronic compounds				40,7
POLITO	Definition and/or development of mechanical tests for Joints on ExtreMat Compounds. Development of mechanical and sealant joints for SiC/SiC materials				33,0
SIEMENS	Development of alternative electronic compounds for power electronics and optoelectronics with a high reliability caused by adapted CTE values and high thermal conductivity of the basic materials and the bonding interfaces				14,0
TUW	Wettability study of wetting promoting thin films for Plasma Facing Compounds with C-based protective material and further enhancement of joining of this C-material to the heat sink				31,3

Objectives

The **general aim** of this WP is the development of joining techniques able to form compounds/components of the materials developed within the other WPs of this IP. Joining of other heterogeneous materials is expected as a spin-off. Resistance of joints in the extreme conditions foreseen for the applications envisaged (high heat flux, high temperature, repeated and fast thermal cycling) will be the measure of the successful achievement of the overall WP objective. The concepts that will demonstrate to be able of complying with Materials Requirements Specifications will be developed until demonstration of practical applicability (i.e. possibility of upscaling for industrial production).

Objective 1: Joining techniques (including interface tailoring) for the formation of compounds with carbon-based and W-based protective materials and heat sinks, able to withstand the environmental conditions expected for plasma facing components in nuclear fusion reactors. The compounds can include integrated hydrogen diffusion barriers and will be produced also with new materials from SP1 and SP2.

Objective 2: Joining techniques for the fabrication of structural parts of SiC/SiC CMC, able to withstand the expected operating conditions in nuclear fusion reactors. Production and test of mock-ups for SiC/SiC mechanical joint and relating sealing. Delivery of compounds for neutron irradiation tests and relating PIE.

Objective 3: Joining techniques (including interface tailoring) for the formation of compounds of C/Cf, SiC/C_f and SiC/SiC Ceramic Matrix Composites and high temperature resistant alloys, able to withstand the environmental conditions expected for the heat shields of space re-entry vehicles. Integration of the different processes developed for production of compounds for space applications and demonstration of their capability for industrial application

Objective 4: Techniques for the deposition of electrically insulating interlayers, with high thermal conductivity, on heat sinks, suitable to be joined to electronic components. Complete electronic compounds and optimization of the manufacturing processes towards possible industrial scale-up.

Objective 5: Active cooled PFCs mock-ups, in both monoblock and flat tile configuration, mainly using C-based materials from SP1, low CTE and high conductivity interlayers and heat sinks from SP2, wetter promoters and interlayers from WP4.2 and graded W/Cu plasma sprayed interlayers from WP4.3.

Objective 6: Development towards industrialization and production at mock-up scale of sputtered TiNx films as wetter promoters on C-based protective materials.

Description of work

The importance of WP4.3, and its activity, are determined by the fact that the practical exploitation in extreme environments of new materials is often dependent on their joining together or to other materials. Also exploiting thin films developed in WP4.2, in order to improve "wettability" by brazing alloys and in order to avoid diffusion of undesired elements, joining techniques based on brazing (high or low temperature) and on "HIPing" have to be developed. Part of the R&D activities have to be devoted to tailoring the interface (surface geometry modification, graded layers, compliant layers). Choice and/or specification of new brazing alloys, together with parameters adjustment and characterisation will allow production of compounds/components for environmental testing (WP4.4).

Activity 1: Development of brazing techniques, including interface tailoring with thin and or thick interlayers and use of graded sprayed and HIPped layers produced in WP4.3, between copper alloy heat sinks (also from WP2.1) and C-based and W-based protective materials (both commercial and new ones from SP1 or coated in WP4.2). Plasma Facing Compounds samples will be produced and delivered for characterisation and environmental testing (WP4.4). Production of PFCs mock-ups, with the chosen concepts, their delivery to WP4.4 for High Heat Flux tests and demonstration of the reproducibility and industrial up-scaling capabilities of the chosen processes.

Activity 2: Development of plasma spray technique (including post-deposition modifications) for direct production of plasma facing compounds, with W protective material, on both commercial and new heat sinks from WP2.1.

Activity 3: Development of joints (including sealing coatings) between parts of SiC/SiC CMC, for structural applications in nuclear fusion reactors, including the delivery of samples for neutron irradiation tests in SP3.

Activity 4: Development of joining techniques (mainly based on diffusion bonding), including the interface tailoring through multi-layers deposition, for the production of heat shields to be used on space vehicles. CMC-metal compounds will be produced and characterised, including delivery of samples for testing by other participants. Tests of space compounds in industrially relevant conditions and evaluation/design of industrial upscaling of developed processes.

Activity 5:	Development of deposition techniques for electrically insulating layers on heat sinks (both commercial and new ones from WP2.2). Thermal spray, brazing, electrophoresis and screen printing will be researched and compounds will be delivered for characterisation and testing. Optimization of processes, design of their scale-up to industrial applications and production of electronic compounds suitable for tests in industrial representative conditions.
Activity 6:	Characterization of compounds (mechanical strength, thermal shock, metallographic examinations, wetting angle measurements and other tests relevant for the planned applications).

2.4.3.2 Results

The summary of activities and results achieved in particular by **AEN** is the following:
Ansaldo's main activities in WP4.3 during the whole project duration have been focused on the development of joining concepts for plasma facing applications, up to the evaluation of their suitability for industrial applications through the production of active cooled mock-ups to be tested in relevant conditions.

In particular Ansaldo performed:

- Development, up to production of mock-ups for testing in relevant conditions, of the concept entailing the use of low CTE interlayers for the joining of heterogeneous materials in nuclear fusion plasma facing applications; it included FEM calculations (supporting the expected stress reduction), brazing test and thermal shock test of compounds and successful production of active cooled mock-ups with different protective materials (also from SP1) and different interlayers (also from SP2).
- Development of the direct brazing between W tiles and CuCrZr for relatively less demanding applications, that included the production of small active cooled mock-ups to be tested for HHF also after neutron irradiation.
- Contribution to development of monoblock PFCs, through brazing of mock-ups with new MMC tubes and with plasma spray coated CuCrZR ones.

Ansaldo's main contribution to ExtreMat project, as regards WP4.3 activities, has been the evaluation of suitability of the low CTE interlayer concept for the production of plasma facing components through industrial processes, in particular brazing; this includes development of the joining concept and process as well as its test and application with interlayers and heat sink materials from SP2 partners. Final visible result has been the delivery of active cooled mock-ups for high heat flux tests in relevant conditions for the nuclear fusion application.

The summary of activities and results achieved in particular by **CEA** is the following:
Extended work has been done within phase 3 of the project concerning the development of insulation layers for electronic applications, both through air plasma spray technique and through electrophoresis; nevertheless it did not result in a compound with higher performances and lower cost than the already existing solution.
However, the electrophoretic concept developed was estimated interesting for other applications (e.g. nuclear applications), which would not necessarily involve the same materials than electronic applications.
Therefore, other materials were tried, which could be more suitable for a processing by electrophoresis. For instance, TiC layers turned out to be easily obtained on Ti-based substrates by electrophoresis followed by a rather low temperature reactive sintering, taking advantage of the high formation enthalpy of TiC from Ti and C.

Furthermore, not only the deposition of layers is possible by electrophoresis, but also the infiltration of porous or fibrous performs. The only conditions for this are to have a conductive preform or to use a conductive electrode behind it, and to have powders that are fine enough to find their way through the pores or interstices. Near the end of the project, the processing of Carbon woven fibres reinforced SiC matrix composites was successfully experimented at CEA. Carbon fibres were chosen for their high electrical conductivity and for their low cost compared to SiC fibres (which is the finally most interesting material for fibres in nuclear applications). The nanosized SiC powders produced at CEA (in SP3) were used for infiltration in the Carbon mats, and their density turned out to be surprisingly high, so that sintering can be easily envisaged.

As regards demonstration of industrial applicability of electrophoretic concept, an analysis of the electrophoretic process for identification of the critical issues has been performed, then resulted in the design of a pilot unit for the demonstration at pre-industrial scale.

The summary of activities and results achieved in particular by **DEMOKRITOS** is the following:

- Successful brazing of ceramic composite materials (C_f/C, C_f/SiC) and graphite to Nimonic alloys for aerospace applications.
- Accomplishment of joining strength higher than that of the joined materials: the ceramic materials fail before the joining.
- Tailored formation of a metallic layer on ceramic composites by magnetron sputtering deposition of Cr layers and subsequent heat-treatments under vacuum. The modification of the composite surface facilitates the joining.
- Successful brazing of silicon to PM1000 alloy.
- Database on metal/ceramics interface interactions.

The summary of activities and results achieved in particular by **IMSAS** is the following:

Activities of IMSAS were during the full project duration directed towards preparation of materials for ExtreMat compounds, particularly divertor mock-ups. Plasma sprayed Cu-W functionally graded coatings and Cu-W metal matrix composites reinforced with W wires were the subject fields of interest within WP 4.3.4.
Plasma sprayed Cu-W coatings with various volume fractions of constituents were prepared and HIP-ed in as-sprayed condition and as-encapsulated in flat Cu cans.
Structural analysis and observations of fracture surfaces revealed that HIP had reduced the porosity of as-sprayed coatings by at least 50 % and the thermal diffusivity of HIP-ed coatings had been improved by 500 – 700 %, particularly for low Cu contents. The effect of HIP was more efficient for encapsulated samples.
Plasma sprayed Cu-W coatings exhibited good resistance towards thermal cycling loadings. The general behaviour reflected the coating composition. Higher values of CTE followed the increasing copper content. No detrimental effect on the thermal diffusivity was observed. There are good preconditions to combine Cu-W plasma sprayed coatings with copper matrix composite material reinforced with tungsten wires in order to get a compound with improved thermal stability.
Structural observations and thermal expansion measurements in x, y and z directions showed, that the deformation of Cu-W coatings with prevailing W volume contents is not homogeneous. It is larger in the z direction than in the x, y directions.
Finally Cu-W gradient coatings with full range of compositions between pure Cu and W were plasma sprayed on CuCrZr tubes. These were additionally consolidated by hot isostatic

pressing. Samples for brazing tests and heat flux tests were prepared. The feasibility of preparation of samples with required technical dimensions was confirmed.
However, modelling of the performance of plasma sprayed Cu-W coatings in SP2 showed that very little if any contribution is to be expected from this gradient concept. The input data for this work were taken from the vacuum plasma sprayed Cu-W coatings. As the current work was performed with the air plasma sprayed coatings, no better properties than those used in the modelling work could have been expected. Therefore this alternative concept had been revised and the efforts and research capacities were directed towards the preparation of divertor mock-ups with wire reinforced Cu-W heat sinks.
Cu-W composite materials reinforced with W cloth and W wires with cross ply arrangements were developed in cooperation with SP2 by diffusion bonding and gas pressure infiltration. Samples with dimensions required for divertor mock-ups were obtained. Fibre volume fractions of 10 % and 50 % were experienced. Thermal expansion measurements of Cu-W composites were performed and properties of composites were obtained. Both concepts appeared to be promising for manufacturing of divertor mock-ups with composite heat sink tubes. Divertor segments based on W monoblock design were prepared by gas pressure infiltration technique. Cu-W composite with ~ 60 % and gradient volume fractions were used as heat sink material.
Finally three divertor W monoblock mock-ups with different wire volume fractions and orientations were prepared via gas pressure infiltration and submitted for heat flux testing at FZJ. Cu-W composite with W wire cloth has been used as reinforcement in design I; combination of wire cloth and circumferentially wound wire in design II and lonely W wire circumferentially wound in design III. The results of heat flux testing revealed that design II and design III survived the most severe testing conditions. The analysis of tested mock-ups revealed some manufacturing and material weaknesses that required some further development within activities performed in SP2.
New PC controlled winding machine has been built to prepare more sophisticated W wire arrangements.

The summary of activities and results achieved in particular by **INASMET** is the following:

- Successful brazing of ceramic composite materials: C/C, C/SiC, SiC and graphite to Nimonic Alloys, grade 105, 95 and 90 for aerospace applications.
- Mechanical characterization of joints that show a joining strength higher than that of the composite material.
- Successful brazing of Silicon to PM 1000 alloy.
- Successful Brazing of C/SiC materials to Nimonic alloys by means of the filler metal modification.
- Successful mechanical results of joints performed with the filler metal modified (modification of the CTE of the filler metal TiCuSil by means of adding Carbon in different forms. In all cases the fracture appears in the composite material, not in the joint itself.
- Good knowledge of the effect of different coatings on the brazing process.

The summary of activities and results achieved in particular by **IPP-CZ** is the following:
<u>As regards fusion applications, main activities and achievements have been the following:</u>
The main alternative concept was plasma sprayed W+Cu composites and FGMs, intended as a stress-relieving layer for plasma facing components in fusion devices. Two configuration options are possible for this application: a) fully sprayed coating up to the tungsten plasma

facing surface on copper-based cooling system, b) plasma sprayed interlayer between bulk tungsten armour and bulk copper-based cooling system. For these two materials, several stages of optimizing the spraying conditions were conducted prior to the production of the composites. While an improvement in the properties (the main being thermal conductivity) was achieved, the processing technique appears to be close to its limit and the thermal conductivity of the sprayed coatings is still significantly lower than for bulk materials. Therefore, a variety of alternative post-processing concepts was applied and examined.

Copper infiltration into plasma sprayed W coating achieved a significant improvement in conductivity. However, the application to real plasma facing components would be difficult. A possible application route would be to produce W+Cu interlayer this way and then join it between the cooling system and the W armour, either bulk tiles or plasma sprayed. Similarly, HIPping of the plasma sprayed coatings achieved significantly higher conductivity, but the need of high temperature treatment complicates direct manufacturing of plasma facing components. Laser remelting has the advantage that it can treat the 100% W surface and that the associated heat treatment is very localized. Promising results were achieved, i.e. layer of significant thickness was remelted, porosity reduced and conductivity increased.

As an alternative to plasma spraying, HVOF spraying was also tested. Due to lower temperature and higher velocity, the amount of oxides and pores are markedly reduced. Consequently, increased conductivity was observed. However, only low tungsten content was achieved.

Besides W+Cu, W+SS composites and FGMs were also prepared by plasma spraying.

In conclusion, plasma spraying of tungsten-based coatings, with or without HIPping, and laser remelting still remain a promising alternative concept. The main advantages of this technique are the ability to coat larger area, including non-planar shapes, possibility of FGM formation, repairs, and elimination of one joining step. The potential application would likely be components with moderate heat flux – as the results from mock-up testing indicate, the performance limit in the actively cooled configuration is around 5 MW/m^2 for coatings 3 to 5 mm thick. This is not adequate for the components operating in highest heat flux conditions, such as ITER divertor, but is sufficient for the first wall of a fusion reactor, where heat fluxes of the order of 1 MW/m^2 are expected. As this technology is rather simple and mature, the industrialization could be relatively straightforward, should there be an interest from the fusion community.

<u>As regards electronic applications, main activities and achievements have been the following:</u>

Plasma sprayed alumina coating as an electrically insulating layer on either copper or AlSiC substrate was the main alternative concept. First coatings showed good performance in partial discharge tests, however, too high thermal resistance. Thinner coatings with low thermal resistance, on the other hand, did not perform satisfactorily in the PD tests. Varied spraying conditions for pure alumina and alumina+steatite did not yield a significant improvement, although the coatings obtained showed very small, closed porosity and no through-thickness cracking. The exact reason was not elucidated; a suspected cause is impurities in the powder.

HVOF spraying was recommended as alternative technique yielding very dense coatings, and promising results were obtained.

The summary of activities and results achieved in particular by **POLITO** is the following:

- POLITO was directly contacted by partners interested in having their compounds tested: tests and sample size were agreed between partner and POLITO
- POLITO tested joined samples as agreed and results were sent to partners (FZJ, IPP-CZ, ATL and IPP).

- Design of the mechanical joint for SiC/SiC was performed in collaboration with MT Aerospace AG.
- Design of the sealant material and of the mechanical and neutron irradiation tests were carried out in collaboration with AEN and NRG-Petten.
- Joined specimens were prepared and mechanical tested.
- Joined specimens were sent to NRG-Petten for neutron irradiation tests.

Main RTD achievements have been the following:
A low-activation silica–alumina–yttria glass to join SiC/SiC composites was designed, developed and tested. The wettability and adhesion of the glass were very good on the SiC/SiC substrate. The joining process at 1375 °C leads to a glass–ceramic joining material, which is thermomechanically compatible with the composites to be joined. Three different kinds of joined samples have been manufactured to couple the reliability of a machined joint with sealant properties of the glass–ceramic joining material. The bending strength results are very promising: higher bending strength than 120 MPa at room temperature were obtained. Future developments will be addressed towards the irradiation tests on joining material and joined samples.
The results described above were achieved in strict collaboration with MT Aerospace AG, which produced machined SiC/SiC, and NRG-Petten, which will perform neutron irradiation tests.

The summary of activities and results achieved in particular by **SIEMENS** is the following:
SIEMENS contributed to WP4.3 mainly as regards characterization of ceramic layers deposited by CEA and IPC, but they also supplied much information about the required properties and they strongly contributed to the formulation of concepts to be evaluated and developed:
To decrease process steps and costs of packaging technologies, alternative substrate technologies to DCB Al_2O_3 ceramics have been tested. As a possible approach, it was suggested to replace a DCB or AMB substrate by a plasma sprayed isolation layer on a baseplate. Furthermore, a new composite material, developed within SP2 and having a high thermal conductivity and appropriate coefficient of thermal expansion, can be used as a baseplate material.
To determine the optimal thickness of an isolation layer, the thermal resistance of Alumina layers was measured and compared to the thermal resistance of the 0.32 mm Alumina DCB including the 80 µm Sn-Ag solder. It was found, that the thermal resistance of plasma sprayed Alumina layers is approximately six times larger than that of the DCB substrate with solder.
Regarding the dielectric strength of the isolation layer, partial discharge measurements of different samples were performed: plasma sprayed Alumina layers from IPP-CZ, plasma sprayed graded layers from CEA, the Al_2O_3 with $MgSiO_3$ layers with different concentration of the oxides from IPC, HVOF sprayed Alumina layers from IPP-CZ and $PbOB_2O_3$ / AlN layers by electrophoresis+sintering from CEA.
The highest dielectric strength among all investigated samples was obtained for the HVOF Al_2O_3 sprayed coatings on AlSiC substrates with the Al metallization deposited by Siemens using electron beam evaporation technique. Their dielectric strength is higher than 25 kV/mm, since the partial discharge values were lower as 10 pC for all measurements.
Other samples delivered by IPP-CZ were with pure Al_2O_3 or Al_2O_3 with $MgSiO_3$ layers with different concentration of the oxides on Cu or AlSiC substrates. The highest dielectric strength of 9.9 kV/mm was observed for the pure Alumina coatings, whereas for the alumina with steatite this value was 6.6 kV/mm (for 75% Alumina layer on the Cu substrate).

Two types of samples were delivered by CEA: plasma sprayed Alumina / Alumina with Al / Al graded layers, and electrophoretically deposited PbO-B_2O_3/AlN layers. For the plasma sprayed layers the highest dielectric strength of 12.5 kV/mm was measured for the sample with a very small metallization area. The electrophoretically deposited layer was six times thicker as required (max. 900 µm) and the thickness varied by about 50% along the surface of samples. According to the performed partial discharge measurements, the dielectric strength of these thick layers is lower than 13 kV/mm.
The soldering process has been simulated on the sample with the HVOF Al_2O_3 coatings on AlSiC substrates with the Al metallization. It was heated to 300°C for 15 minutes. This temperature was found to be too high for the sample, since the partial discharge measurement performed after the heating has led to electrical breakdown.
3 three types of substrates with single ceramic layer were investigated:
AMB Si_3N_4, DCB Al_2O_3 with Zr, and DCB AlN. The isolation strength of the DCB Al_2O_3 with Zr substrate was found to be 3 times lower in comparison with tested AMB Si_3N_4 and DCB AlN substrates, which have the same dielectric strength of > 19 kV/mm. Furthermore, AMB Si_3N_4 substrates, 0.32 mm Si_3N_4 ceramics with 0.5 mm and 0.3 mm Cu metallization, have been tested in view to thermal cycling in a two chamber oven with parameters: temperatures -55°C / 150°C, dwell time of 15 min, and 3600 thermal cycles. After optical control first cracks at the edge of the metallization have been found after 3600 thermal cycles.
The AMB Si_3N_4 substrates with double layers of 320 and 640 µm have been investigated. The double layer substrate has failed already at a very low voltage of 0.5 kV. The reason for this high partial discharge was the air inclusions in the double layer system.

The summary of activities and results achieved in particular by **TUW** is the following:
TUW contributed the technology of the reproducible production of substoichiometric TiN as well as the means for the static and dynamic measurement of the contact angle of several brazing alloys with pre treated and coated surfaces. From these activities several publications and presentations at international meetings emerged. Within WP4.3 one PhD thesis and one master thesis were finished and the students active within ExtreMat obtained high technological, scientific and social skills, which enabled them to pursue careers in technology oriented fields right after finishing their respective works.
TUW mainly worked about TiN development, a part from designing and setting up a new facility for high temperature wetting angle measurements in vacuum (High Temperature Sessile Drop Device, HTSDD). The characterization of the wetting behaviour of liquid metals on solid substrates is done by contact angle measurement of the liquid and the time evolution of the contact angle. The unique feature of this device is the removable and heatable substrate holder, which can additionally be used in a PVD chamber through a similar load-lock system. This gives the possibility of depositing wetting promoting thin films and the transport of them under vacuum conditions to the contact angle measurements. The heating of the substrate holder is done by a resistance heated Ta foil, onto which the sample for the experiment is clamped. The device allows reaching temperatures of about 1100°C. The contact angle is recorded optically by a commercial webcam connected to a PC.
The investigations on a wetting promoting interlayer for CFC have been mainly focused on substoichiometric TiN_x films. Also some work was done with Mo interlayers, which also showed a good wetting by the CuABA® brazing alloy.
The deposition of the TiN_x coatings is done be reactive magnetron sputtering with a mixture of Ar and N_2 gases. TiN_x films with a tuneable x can be produced. The composition of these substoichiometric TiN_x was analyzed by means of Rutherford Back Scattering (RBS) at IPP,

which allows checking the composition through the thickness of the coating. This led to a linear correlation between nitrogen content in the coating and nitrogen flow.
A time dependent contact angle study of CuABA® on different substoichiometric TiN_x was performed with the result that the reduction of nitrogen in the coating to a content of x = 0,15 – 0,30 leads to the fastest wetting. This means that the time for reaching the final contact angle (which is the same for all samples, approx. 10°) was decreased by decreasing the nitrogen content in the coating. Therefore these films were concluded to be the best wetting promoting layers.
The type of reactive wetting can be identified by plotting the contact angle vs. the droplet radius. A straight line in this plot is significant for the reaction controlled reactive wetting type, a power-law curve would suggest diffusion controlled reactive wetting. The samples with CuABA® on Ti, TiN and C were analyzed in this way, and all of them showed reaction controlled reactive wetting.
After finding the best wetting promoting thin film ($TiN_{0,3}$) in trial experiments, these films were deposited onto graphite and CFC substrates provided by Ansaldo for first brazing trials. Two thicknesses and two stoichiometries were chosen and deposited.
Concerning possible up-scaling the present TiN coating is well within the stable region of reactive sputtering and therefore there seem to be no major obstacles for up-scaling this process even to the extent of coating large areas.

2.4.3.3 Deviations from the work programme, and corrective actions taken

No major deviations from the work programme happened during the project.

2.4.3.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

2.4.4 WP4.4: Environmental tests for industrial applications

2.4.4.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 54	**Duration**	54 months
Participants	**Responsibility in the WP (identify in bold WP leader)**				**Effort (ppm)**
FZJ	Screening tests and thermal fatigue tests of plasma facing compounds with fusion relevant heat loads (main application) and other compounds.				**26,5**
IPP	Deuterium diffusion tests and thermal stability tests on tritium diffusion barriers				26,6
PLANSEE	Environmental tests of electronic compounds prepared by other WPs (CTE, TC, chemical stability, corrosion resistance, thermal cycle stability and shock resistance)				3,4
SIEMENS	Environmental tests of electronic compounds prepared by other WPs (high temperature storage, thermal shock tests, humidity tests)				5,0

Objectives

Main: The general aim of this WP is the performance of tests, in relevant conditions for industrial applications, on diffusion barriers and on compounds/components produced by joining of new materials, developed by other WPs. In order to better comply with the above mentioned aim, this WP aims also at the integration of characterisation techniques available within the consortium. In the last part of the project the tests will continue on mock-ups devoted to demonstrate the industrial applicability of the materials/concepts/technologies developed.

Objective 1: Understanding of the failure mechanisms of actively cooled nuclear fusion components through screening, thermal fatigue testing and post-tests analyses, including interaction with component modelling (WP2.3) for pointing out possibilities of improvement.

Objective 2: Investigate the feasibility of ceramic CVD coatings as tritium diffusion barriers by measuring the deuterium diffusion through such coatings.

Objective 3: Verification/demonstration of the reliability of electronic compounds and relating materials.

Objective 4: Determination of the behaviour of compounds close to industrial applications and of their failure mechanisms when exposed to environmental conditions representative of the real ones.

Objective 5: Contribution to the final evaluation of the industrial applicability of ExtreMat compounds.

Description of work

Integration of most advanced testing capabilities available within the consortium will allow to test both integrated diffusion barriers and compounds/components produced by joining of heterogeneous materials in conditions very similar to the real ones, expected for components in industrial applications. Synergistic effects due to different loadings acting simultaneously will be investigated too. Industrial expertise from the application field will be at the basis of final evaluation of the achieved results. Testing in relevant conditions for industrial applications of materials and compounds will be extensively performed. Tests will allow the verification of the materials and compounds capability of complying with the Materials Requirement Specifications.

Activity 1: Actively cooled nuclear fusion components will be tested in screening and thermal fatigue tests with fusion relevant heat loads up to 20 MW/m^2 during up to 100 cycles. The thermal performance of the components will be monitored and they will be accompanied by microstructural/metallographical post-mortem analyses. Through interaction with modelling activity better understanding of failure mechanism will be achieved and possibilities for improvements will be determined.

Activity 2: Permeation measurements through alumina and erbia coatings on Eurofer substrates and through alumina and/or erbia coatings on Eourofer with a CVD tungsten top layer.
Activity 3: Environmental tests of electronic materials and compounds representative of industrial ones.
Activity 4: Reliability testing of IGBT modules with diamond composite baseplates.

2.4.4.2 Results

The summary of activities and results achieved in particular by **FZJ** is the following:
FZJ contribution, a part from some testing of electronic modules, was mainly dedicated to nuclear fusion related materials and compounds characterisation. All delivered actively cooled test components representing the different designs, i.e. flat tile and monoblock, and comprising a combination of standard and ExtreMat materials were exposed to high heat fluxes in the electron beam facility JUDITH 1.
This comprised firstly the application of steady state heat loads and cyclic thermal loads on conventional CFC flat tile mock-ups. These were produced using different production routes starting from Cu-casting on CFC with subsequent brazing to the CuCrZr heat sink, ending up in a single brazing step process aiming at the reduction of the production time and cost, always achieving a steady improvement of the life time performance.
Secondly, a qualification of four W/Cu flat tile test modules, twice with and twice without pure Cu-interlayer, of which two will be irradiated in the campaign organised in the frame of the ExtreMat project was performed. For this purpose a new fixture to attach the component to the cooling system was manufactured to meet the requirements of compound dimension and manipulator operation, necessary for the handling of neutron irradiated materials.
The compounds survived screening loads up to 22.5 MW/m^2 without any visible degradation of their thermal performance.
In addition to the application of steady state heat loads on compounds, material testing, in particular of coatings, was performed by thermal shock tests. Therein CVD-W/Re coatings as well as plasma sprayed W-coatings were exposed to disruption like loads up to 0.8 GW/m^2 for 4 ms, which is about the melting threshold for pure tungsten. All tested coatings showed heavy melting, due to a low thermal conductivity, as well as cracking and erosion, already at power densities well below the melting threshold for pure W. Since thermal conductivity among others plays a major role in the performance of materials under thermal shock loads, laser flash measurements of plasma sprayed free standing samples were done to qualify the materials and to identify the most promising parameter set up for the production process.
In screening and thermal cycling tests the thermal fatigue behaviour of the components, i.e. the reliability of the joining technique and the materials themselves, were investigated. Depending on the design and the manufacturing technology, the components survived different loading conditions up to 100 cycles at 20 MW/m^2.
By infra-red imaging to monitor the cooling down behaviour of the components to identify hot spots, water calorimetry to determined the real absorbed power density and pyrometer measurements the behaviour during the thermal loading was investigated. In post mortem analyses the specific failure modes for the various components were identified, which are mainly resulting from the thermal mismatch of the materials causing crack formation at the material's interfaces, from too low thermal conductivities of the plasma facing materials, resulting in an increased erosion during heat loading, and from insufficient interfacial strength at the joint, which mainly occurs for the plasma sprayed components.

Various compounds with graphite (UALI), CFC (INCAR, AEN) and W (IPP) flat tile structure were exposed to steady state and cyclic heat loads. In these tests large differences in components performance was observed. Therein the module comprising a 3D CFC from INCAR as plasma facing material showed the highest performance. Testing (cycling at 20 MW/m^2) of this compound was only stopped due to a malfunction of the clamping mechanism to the cooling structure. The resulting slight ingress of water vapour caused a breakdown of the vacuum.
At the PFMC-12 workshop in Jülich which took place from 11.-14. May 2009 the results of former investigations on CFC flat tile modules from AEN, POLITO and FZJ as well as W-monoblock mock-ups from IMSAS were presented.

The summary of activities and results achieved in particular by **IPP** is the following:
During the project, IPP contributed to the development of erbia coatings on steel substrates produced by ATL. The characterization work comprised XRD and SEM analysis, FIB and conventional sample preparation. A gas permeation setup was used to determine the barrier property of the coating for hydrogen permeation.
Tungsten, alumina and erbia coatings have been produced by PVD and CVD deposition techniques and subsequently tested by IPP's deuterium permeation device. In order to compare results of deuterium permeation through CVD tungsten, reference measurements of bulk tungsten have been performed in a first step. Due to oxidation behaviour of tungsten at high temperatures,a vacuum leakage has occurred during the permeation test. Therefore with the currently installed setup no further tungsten permeation measurements have been possible. Comparative permeation measurements of vacuum-arc-deposited erbia and alumina coatings on Eurofer yielded indications that the permeation reduction factor of erbia is of similar magnitude as that of alumina. The basic applicability of alumina as integrated hydrogen diffusion barrier in a thin tungsten/alumina/Eurofer compound was demonstrated by sputter-depositing 1 μm of tungsten on top of an alumina layer.
XRD analysis of first CVD-erbia coatings on Eurofer substrates (delivered by ATL) has shown the crystal structure of body-centered cubic Er_2O_3. An estimated permeation reduction factor of 10^3 to 10^4 has been achieved. These coatings, however, display a partly unsuitable microstructure.
The first erbia coatings on Eurofer prepared by ATL showed a good permeation reduction (10^3-10^4) but the microstructure was not optimal. After a dense erbia layer, coarse grain growth appeared. New depositions showed an improved microstructure, but conventional steel was used as substrate material. Similar depositions on Eurofer (EO40) showed cracks and voids. Therefore, such coatings showed no permea-tion reduction for hydrogen. These results indicate a strong influence of the substrate material on the microstructure and therefore on the barrier property. Further improvement is needed for Eurofer substrate, e.g. an additional interlayer.

The summary of activities and results achieved in particular by **PLANSEE** is the following:
PLANSEE main contribution has been the performance of thermal conductivity measurements on new heat sinks from SP2:

1. The thermal diffusivity of the silver and aluminium diamond composites remains the same before and after thermal cycling, indicating a stable interface.
2. In case of copper-diamond composites, with additions of Si, thermal diffusivity degrades substantially after thermal cycling.

3. Pressure cooker test indicates no visible damage to the parts after subjecting them to 100% relative humidity and high temperatures. These results were especially surprising in the case of Al-diamond composites, as aluminium carbide is known to be a hygroscopic material capable of damage in humidity.

Taking into account that PLANSEE was in fact performing characterization only for SP2 materials and that their main activity was just performed in SP2 (development of high thermal conductivity materials), their contribution has been completely moved to SP2 since May 2006.

The summary of activities and results achieved in particular by **SIEMENS** is the following:
The reliability testing was performed by SIEMENS for ExtreMat partners from WP 2.2.
During this time more than 630 samples were received. The thermal cycling test is a standard test (Reference: JESD22 A-104) used for the components in power electronic modules, and it was performed with a two-chamber oven, type "Vötsch" at following parameters: -55°C / +150°C, dwell time at each temperature of 20 min, transfer time of 2 s and thermal cycles from 100 to 1000. During the pressure cooker test (Reference: JESD22 A-102) the specimens were held at 121°C and 2 bar for 168 hours.
SIEMENS has investigated the influence of the environmental test on the properties of Al-diamond from EMPA (Al/CD/60p) and PLANSEE (AlSi/CD/60p) and bimodal AlSiC from EMPA (Al/SiC/70p) and compared with industrial AlSiC. The most stable material against the environmental test was found to be the bimodal AlSiC without Si from EMPA, which thermal conductivity of 315 W/(mK) was much higher than for industrial AlSiC with Si (200 W/(mK)). In contrast to the Al-diamond from EMPA, the Al-diamond from PLANSEE has higher thermal conductivity and, at the same time, lower coefficient of thermal expansion, both before and after thermal cycling. The flexural strength and Young's modulus were characterised by four-point bending test. In case of AlSiC from EMPA it was done before and after the thermal cycling. Industrial AlSiC with Si in the matrix, as a reference, has the highest characteristic flexural strength of 345 MPa. At the same time the stiffest material was AlSiC from EMPA even after the thermal cycling with 1000 cycles, since it's Young's modulus was 237 GPa. Industrial AlSiC and Al-diamond composites from EMPA and PLANSEE had about 200 GPa.
3.3 kV test IGBT modules were manufactured with diamond composite baseplates from PLANSEE and AIT. Prior to soldering of DCB substrates with chips, the baseplates were nickel plated. The surface roughness and the presence of open pores in some baseplates have worsened the quality of nickel plating and, as a consequence, the quality of soldering of DCB substrate. To investigate the influence of the passive cycling on the system solder lifetime, thermal cycling was performed. In the case of Cu-diamond test module, warpage of the baseplate increased significantly and that resulted in changing of cooling conditions during the thermal resistance measurements.

2.4.4.3 Deviations from the work programme, and corrective actions taken

No major deviations from the work programme happened during the project.

2.4.4.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

3. Section 3 – Consortium Management

3.1 Consortium management tasks

Subproject/Workpackage 5 is divided into ***3 work packages*** (WP) and ***2 project partners*** contribute to this SP/WP.

Partners	**WP5.1: Project Co-ordination**	**WP 5.2: Project Management Office**	**WP 5.3: Knowledge Dissemination / Training Activities**
IPP	**X**	**X**	**X**
BI			**X**

X: WP partners

3.1.1 WP 5.1: Project Co-ordination

3.1.1.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants					**Effort (ppm)**
IPP					70,0

Objectives

Main: Management and control of the entire ExtreMat IP

Objective 1: Focal interface to the European Commission

Objective 2: Maintaining of a well-functioning Consortium

Description of work

Main: Maintaining efficient external working relations to the Commission and internal to the Consortium participants and all organisational units

Activity 1: Maintaining of the entire Project organisation for the entire project life-cycle

Activity 2: Monitoring and control of the Project execution in accordance with the Contract and the Council instructions

Activity 3: Monitoring science and society issues, related to the research activities conducted within the project

Activity 4: Council, PCC, SIC, Industrial User Group

Activity 5: Monitoring the promotion of gender equality in the project

Activity 6: Administration of the Community Contributions

3.1.1.2 Main work carried out

- Preparation and Co-chair of the Kick-Off Meeting as well as of all Council Meetings
- Preparation of all Technical Reviews
- Issuing and maintenance of the Consortium Agreement (CA)
- Chair of all RTD Steering Committe Meetings as well as of all Project Coordination Committee (PCC) Meetings
- Revisions of Annex I and Consortium Agreement
- Organisation of the Neutron Irradiation Coordination Meetings in cooperation with partner NRG
- Follow up of the scientific and technological evolutions

3.1.1.3 Problems encountered

There are no problems encountered.

3.1.1.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

3.1.2 WP 5.2: Project Management Office

3.1.2.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants	**Responsibility in the WP (identify in bold: lead-contractor)**				**Effort (ppm)**
IPP					58,0
RAMS-CON					16,6

Objectives

Main: Management and control of the entire ExtreMat IP in accordance with the Contract, the Council, PCC and Coordinator instructions

Objective 1: Management and control of the ExtreMat IP Consortium

Objective 2: Support to the preparation of reports to the European Commission

Objective 3: Providing management support to the Subproject Coordinators to enable them to concentrate on the scientific coordination and progress of the research activities

Description of work

Main: Operation of the Project Management Office

Activity 1: Overall legal, contractual, financial and administrative management **of the consortium** (Consortium Operation), including
- maintaining of the Consortium Agreement
- generation of the necessary consortium communication
- monitoring of management procedures and regulations

Activity 2: Overall technical, legal, contractual, financial and administrative management **of the IP** including
- financial, technical and configuration management, and schedule related monitoring
- monitoring of the preparation of deliverables
- operation of project level communication system (BSCW)
- organisation of progress meetings(PCC)

Activity 3: Preparation and delivery of status / progress reports and planning documents to the Coordinator, the Council and for the Commission

Activity 4: Management of all Project Reviews; support of SIC / Council activities

3.1.2.2 Main work carried out

- Organisation of the Kick-Off Meeting as well as of all Council Meetings
- Organisation of the all Technical Reviews
- Revisions / updates of Annex I according to the outcome of the Technical Review
- Handling of contractual aspects reagrding change of partners legal forms
- Preparation and execution of all written Council decisons
- Issuing and maintenance of the Consortium Agreement (CA)
- Definition, establishing and implementation of all Project Management Regulations
- Distribution of the Community contribution
- Organisation of all RTD Steering Committe Meetings as well as of all Project Coordination Committee (PCC) Meetings
- Collection and revision of all deliverables (afterwards: uploading on the BSCW)
- Preparation of the Periodic Reportings as well as the entire Final Reporting

3.1.2.3 Problems encountered

Major corrective actions had been necessary with regard to the project management. In the course of the first 6 months of the project it had been realized by the project partners that the project management activities were not adequate to the needs of an integrated R&D project.

On July 18, 2005 the partner RAMS-CON (responsible for project management so far) declared the withdrawal from the project which was unanimously accepted by the consortium. The withdrawal became effective on September 6, 2005. After the withdrawal, the consortium agreed on changes of the governance and management structure and transferred the project management tasks to the Coordinator (IPP).

IPP activated the necessary resources by employing a project manager and former staff member of the Federal Ministry of Education and Research, Germany. The problems regarding project management were resolved before the end of the first project year.

3.1.2.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

3.1.3 WP 5.3: Knowledge Dissemination

3.1.3.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 70	**Duration**	70 months
Participants					**Effort (ppm)**
BI					18,4

Objectives

Main: Management of the Use and Dissemination of Knowledge generated in ExtreMat

Objective 1: Creating awareness in industry, especially at SMEs, for the research topics and results of ExtreMat.

Objective 2: Disseminating knowledge to the industrial and scientific community wherever protection and use is not adversely affected.

Objective 3: Documenting and reporting towards the CEC about the exploitable knowledge generated in ExtreMat, its current or potential scientific and industrial use and envisaged routes of its exploitation (confidential).

Description of work

Main: The framework of a specific "Plan for Using and Disseminating Knowledge" has been developed in Phase 1 and 2 of the project. During phase 3 this plan was successfully implemented, dissemination activities towards industry started and the plan was further detailed with respect to tangible results. Phase 4 was mainly devoted to dissemination and publication of those results, with a special highlight at the world largest industrial fair in Hanover in April 2008 and a specific ExtreMat conference in san Sebastian in June 2008. In the original contract, Month 48 was planned as the end date for the knowledge dissemination activities. Most ExtreMat project partners finished their industrialisation work of Phase 4 on schedule, so that the dissemination activities also could be completed at this time to a large extend. However, non-predictable technical problems at the test reactor at Petten delayed some parts of the ExtreMat project beyond the planned end date. The ExtreMat consortium decided that no additional dissemination activities should be planned for this prolongation period, but a smooth phasing-out of the dissemination should be performed on a strongly reduced level.

Activity 1: Update and maintenance of the ExtreMat project website *www.extremat.eu*, dissemination of ExtreMat related news, events and scientific publications through this website

Activity 2: Dissemination towards industry: Presentation of the ExtreMat project work and results towards the industry at various fairs (Transfac and Materialica 2006, JEC and Hanover Fair 2007), with the highlight at the Hanover Industry Fair 2008 as the major ExtreMat dissemination event

Activity 3: Press releases and intensive public relation work on ExtreMat

Activity 4: Update of confidential information on exploitable knowledge and its use by the ExtreMat participants

Activity 5: Update of the Plan for Using and Disseminating the Knowledge

3.1.3.2 Main work carried out

The highlight of the dissemination activities towards industry clearly was the presentation of the ExtreMat project at the world's largest industrial fair Hannover Messe 2008. ExtreMat organised a stand with 200 square meters in the hall "research and technology", participated in the "Night of Innovation" programme, presented selected results at the

"Gateway2Innovation" forum and published numerous articles in newspapers and magazines about ExtreMat and its presentation in Hanover. During the five days at the Hannover Messe, 39 ExtreMat researchers were present at the exhibition stand. The ExtreMat stand was visited by more than 1,500 professionals for technical discussions, by representatives from politics and media who wanted to learn more about the EU-funded integrated project, by young engineers interested in career opportunities in materials science, and even by a primary school class exploring the world of innovation. In total, participation in the Hannover Messe was a great success for the ExtreMat project.

Concerning dissemination activities towards the scientific world, the highlight was the ExtreMat Scientific Conference in San Sebastian 2008. 150 scientists from all over the world attended this conference and discussed latest developments on new materials and the corresponding crosscutting processing technologies, mainly generated within ExtreMat. In addition, ExtreMat organised specificthe topics „Materials for Extreme Environments" on Euromat conferences 2005, 2007 and 2009, and presented ExtreMat results at many other international scientific conferences. Finally, more than 100 scientific journal papers were already published about ExtreMat research results at the deadline of this report.

3.1.3.3 Problems encountered

No serious problems occurred during the project duration with respect to knowledge dissemination. In the course of the project several minor adjustments of dates and resources had to be performed, but this can be considered as normal business in research projects. In the end, all planned major activities were delivered successfully with avctive support of all ExtreMat project partners.

3.1.3.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

3.1.4 WP 5.3: Training Activities

3.1.4.1 WP overview on objectives, involved partners, planned work and manpower

Start date	Month N° 1	**End date**	Month N° 48	**Duration**	48 months
Participants					**Effort (ppm)**
BI					4,7
IPP					0,7
INASMET					1,0

Objectives

Main: Training activities shall facilitate

- the immediate pick-up of materials design and processing know-how in the materials industry and a quick innovation cycle of market-ready new materials
- the rapid integration of the new technologies into new products and systems

Objective 1: Transfer of knowledge gained from basic research to the materials industry
Objective 2: Transfer of knowledge on newly developed materials and their compounds into the product and systems industry

Description of work

Main: Development, planning, marketing and organization of the training activities

Activity 1: Identification, selection and development of suitable training activities together with ExtreMat partners
Activity 2: Marketing and awareness activities to generate interest of potential participants in relevant industries, e.g. by internet, flyer, mailing, direct marketing with ExtreMat partners, presentation at JEC Composites Fair etc
Activity 3: Production of handouts and documents for the participants
Activity 4: Organization of the training activities at the different locations in Europe
Activity 5: Evaluation of the training activities and communication to the partners and participants
Activity 6: Implementation of key-learnings for subsequent trainings

3.1.4.2 Main work carried out

During the ExtreMat project, 10 targeted training activities with more than 300 participants in total were organised:

1. Tutorial Course “C&SiC Composites for Nuclear Applications”,
   20 September 2006, Petten, The Netherlands (main organizer NRG)
2. Tutorial Course “Plasma-Facing Materials and Components for Fusion Applications”,
   9 October 2006, Greifswald, Germany (main organizer IPP)
3. Conference “High-performance diamond-based composites: Innovations in superabrasives and thermal management”,
   10 November 2006 Dübendorf, Swizzerland (main organizer EMPA/PLANSEE)
4. ExtreMat Training “Thermophysical Workshop”,
   22-23 January 2007, Jülich, Germany (main organizer FZJ).

5. ExtreMat Training “Space Materials for high temperature applications”,
   26-27 June 2007, Torino, Italy (main organizer BI / POLITO)
6. ExtreMat Training “Novel heat sink materials for power electronics applications”,
   10 October 2007, Nuremberg, Germany (main organizer BI)
7. Workshop on “Protecting and Exploiting Intellectual Property generated in ExtreMat”,
   3-4 April 2008, Munich, Germany (main organizer BI)
8. Session “ExtreMat – New materials for Extreme Environments”
   within the public “Forum Tech Transfer – The Direct Route to the Market” at the Hanover Industry Fair,
   22 April 2008, Hanover, Germany (main organizer IPP)
9. Tutorial Course on “ExtreMat materials R&D” at the 1st Conference on New Materials for Extreme Environments,
   2 June 2008, San Sebastian, Spain (main organizer INASMET / IPP)
10. Evening Seminar for Professionals on “Materials for Extreme Environments”,
    16 October 2008, Garching, Germany (main organizer BI).

3.1.4.3 Problems encountered

There are no problems encountered.

3.1.4.4 List of deliverables and milestones

See Appendix 2 (List of deliverables)
See Appendix 3 (List of milestones)

3.2 Contractors

All contractors engaged the necessary resources to reach the scientific-technical objectives of the project.

For a large part of the contractors, the results obtained exceed the expectations. The contractors participated to the technical meetings on subproject level and at several working meetings on topics related to the integrative activities of the project (meetings to detail inter-partner cooperation, definition of neutron irradiations). The technical reporting provided by the partners during the meetings was of very high quality and all written reports required as deliverables have been received. The internal steering by the *Scientific Industrial Committee* was very efficient and the (mainly industry) partners involved in this committee provided valuable comments, steering the project activities towards their industrialization potential.

3.3 Project timetable and status

The status of the project is visible in the timetable in table 3.3.1.

Nr.	Task Name	Dauer	Anfang	Ende
1	*Milestone 1: Project start*	0 Tage	Mi 01.12.04	Mi 01.12.04
2		0 Monate	Mi 01.12.04	Mi 01.12.04
3	Phase 1 project months 1-4	84 Tage	Mi 01.12.04	Do 31.03.05
4		4 Monate	Mi 01.12.04	Do 31.03.05
5	SP 1: Self-passivating protection materials	84 Tage	Mi 01.12.04	Do 31.03.05
10	SP 2: Heat sink materials	84 Tage	Mi 01.12.04	Do 31.03.05
14	SP 3: Radiation-resistant materials	84 Tage	Mi 01.12.04	Do 31.03.05
19	SP 4: ExtreMat compounds	84 Tage	Mi 01.12.04	Do 31.03.05
24	SP 5: Project co-ordination & management	84 Tage	Mi 01.12.04	Do 31.03.05
28				
29	*Milestone 2: Requirement validation*	0 Tage	Do 31.03.05	Do 31.03.05
30		0 Tage	Do 31.03.05	Do 31.03.05
31	Phase 2 project months 5-10	126 Tage	Fr 01.04.05	Fr 30.09.05
32		6 Monate	Fr 01.04.05	Fr 30.09.05
33	SP 1: Self-passivating protection materials	126 Tage	Fr 01.04.05	Fr 30.09.05
38	SP 2: Heat sink materials	126 Tage	Fr 01.04.05	Fr 30.09.05
42	SP 3: Radiation-resistant materials	126 Tage	Fr 01.04.05	Fr 30.09.05
47	SP 4: ExtreMat compounds	126 Tage	Fr 01.04.05	Fr 30.09.05
52	SP 5: Project co-ordination & management	126 Tage	Fr 01.04.05	Fr 30.09.05
56	*Milestone 3: Concept review*	0 Tage	Fr 30.09.05	Fr 30.09.05
57		0 Tage	Fr 30.09.05	Fr 30.09.05
58	Phase 3 project months 11-36	546 Tage	Mo 03.10.05	Mo 03.12.07
59		26 Monate	Mo 03.10.05	Mo 03.12.07
60	SP 1: Self-passivating protection materials	546 Tage	Mo 03.10.05	Mo 03.12.07
68	SP 2: Heat sink materials	546 Tage	Mo 03.10.05	Mo 03.12.07
75	SP 3: Radiation-resistant materials	546 Tage	Mo 03.10.05	Mo 03.12.07
83	SP 4: ExtreMat compounds	546 Tage	Mo 03.10.05	Mo 03.12.07
91	SP 5: Project co-ordination & management	546 Tage	Mo 03.10.05	Mo 03.12.07
95	*Milestone 4: Development review*	0 Tage	Mo 03.12.07	Mo 03.12.07
96		0 Tage	Mo 03.12.07	Mo 03.12.07
97				
98	Phase 4 project months 37-54	378 Tage	Mo 03.12.07	Di 02.06.09
99		18 Monate	Mo 03.12.07	Di 02.06.09
100	SP 1: Self-passivating protection materials	378 Tage	Mo 03.12.07	Di 02.06.09
105	SP 2: Heat sink materials	378 Tage	Mo 03.12.07	Di 02.06.09
109	SP 3: Radiation-resistant materials	378 Tage	Mo 03.12.07	Di 02.06.09
114	SP 4: ExtreMat compounds	378 Tage	Mo 03.12.07	Di 02.06.09
119	SP 5: Project co-ordination & management	378 Tage	Mo 03.12.07	Di 02.06.09
123	*Milestone 5: Material validation*	0 Tage	Di 02.06.09	Di 02.06.09
124		0 Tage	Di 02.06.09	Di 02.06.09
125				
126	Phase 5 project months 55-72	378 Tage	Di 02.06.09	Do 02.12.10
127		18 Monate	Di 02.06.09	Do 02.12.10
128	SP 3: Radiation-resistant materials	378 Tage	Di 02.06.09	Do 02.12.10
130	*Milestone 6: Test review*	0 Tage	Do 02.12.10	Do 02.12.10
131		0 Tage	Do 02.12.10	Do 02.12.10
132				
133	Important Dates	1260 Tage	Mi 01.12.04	Di 01.12.09

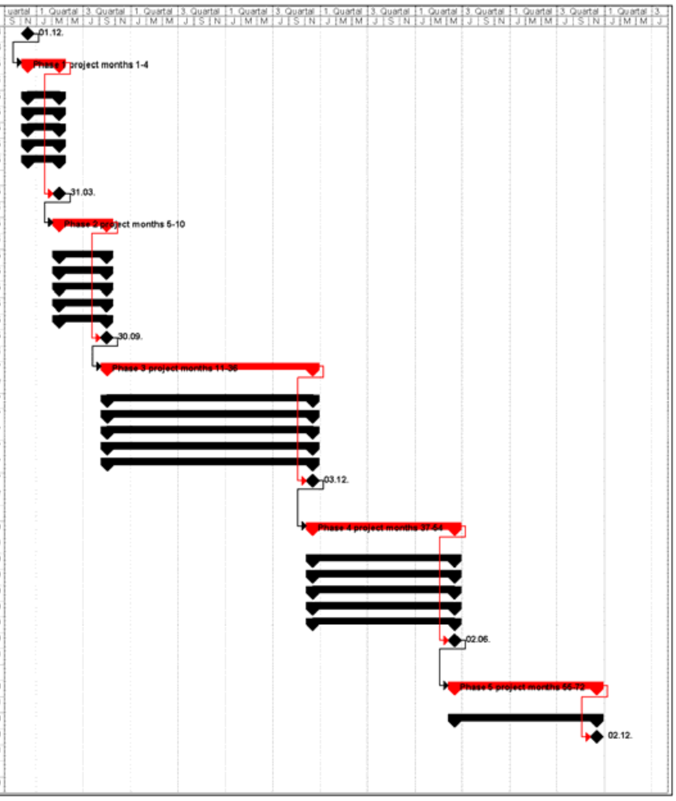

Table 3.3.1: Project timetable and status

4 References

The activity report is based on the deliverables of the project provided to the Commission together with all previous Periodic activity reports.

No further references are made.

5 Appendices

5.1 Appendix 1: "Plan for using and disseminating the knowledge"

The plan has been summarized and/or revised by partner Bayern Innovativ (BI), using the input of all partners.

The document also can be found on the "ExtreMat – Reporting-CD" within the folder "Final Activity Report".

5.2 Appendix 2: "List of all deliverables"

The list has been summarized by the PMO.

The documents also can be found on the "ExtreMat – Reporting-CD" within the folder "Final Activity Report".

Furthermore this document can be found on the BCSW within the folder "Commission Reports" (http://bscw.rzg.mpg.de/bscw/bscw.cgi/211236) and allows a "direct linking" to the respective deliverables within the BSCW.

5.3 Appendix 3: "List of Milestones"

The list has been summarized by the PMO.

The documents also can be found on the "ExtreMat – Reporting-CD" within the folder "Final Activity Report".