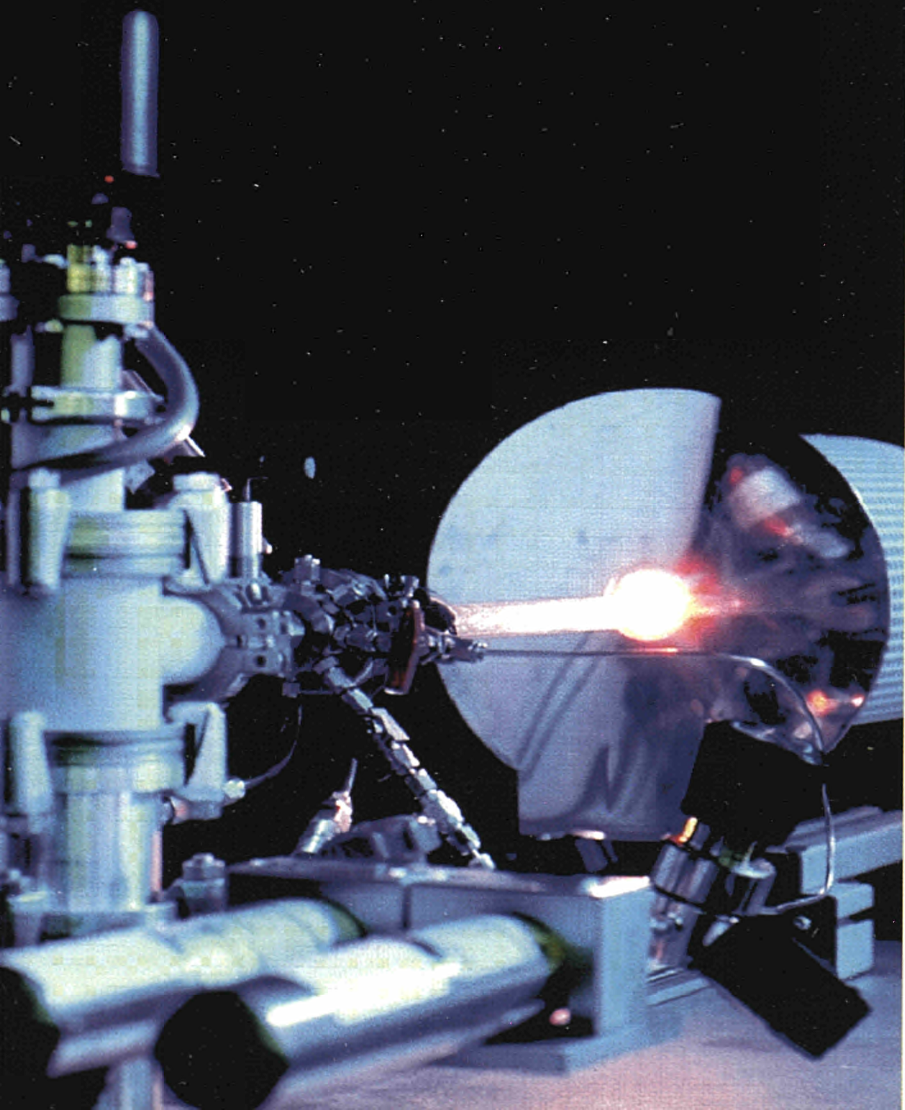
iam

Annual Report 96



Institute
for
Advanced
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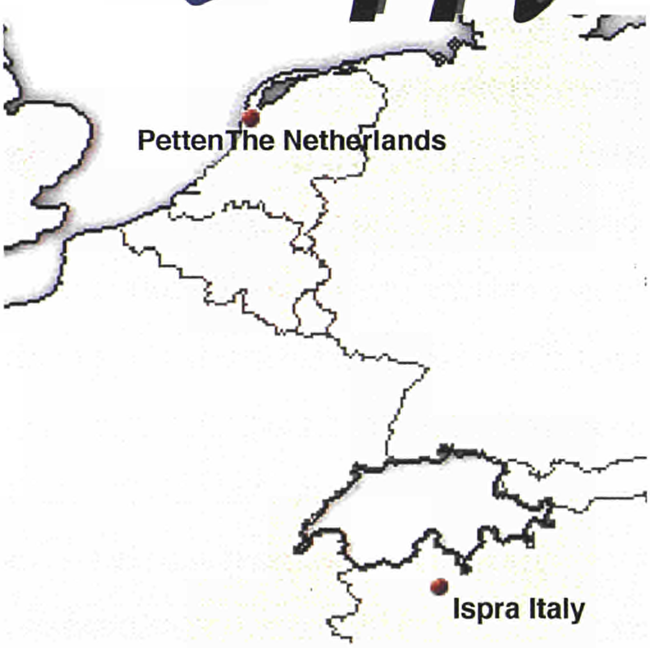

ANNUAL REPORT '96

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Institute for Advanced Materials

Foreword

During 1996 the change in the management culture in the Institute for Advanced Materials (IAM) was continued. New tools related to Total Quality Management were introduced. The organisation was made leaner; duplication of activities in Units and between Petten and Ispra was removed. Surface Engineering was concentrated in Ispra, Non-Destructive Testing in Petten. A major achievement was the Quality accreditation granted to major testing activities in the Energy Systems Testing Unit.

The participation in the Commission Programmes in Shared Cost Actions was a major success.The average success rate of those consortia, where IAM participated, was about 50 %, and the total volume for IAM came close to target levels. The achievements on other competitive activities, i.e. third party work and competitive support to the Commission, did not fully meet expectations. It is planned to correct this situation during the remaining two years of the 4th Framework Programme. To this end new marketing and selling tools, and Marketing Plan were established during the last part of 1996.

The work on the IAM Institutional Programme of IAM continued as planned. The results and deliverables are outlined in some detail in this Annual Report.

The Institute activities were evaluated by a Visiting Group of representatives from industry and academia. The outcome of the evaluation was favourable to IAM, noting in particular the new direction towards more customer-orientation and improved management, but at the same time maintaining the high scientific quality and European level added value in major activity areas. Recommendations for future improvements were also provided, in line with the management goals of the institute.

The year 1996 was the first year of the new EURATOM Supplementary Programme for the High Flux Reactor (HFR). It was a successful year; the new leaner organisation proved to be effective, and the programme targets were achieved. It is particularly pleasing to report that the competitive targets for the full four year programme period were secured. Details of HFR activities are presented in separate HFR Annual Report.

In conclusion, I would like to express my sincere thanks to the entire staff of the Institute for their successful efforts in 1996.

Kari Törrönen

JOINT RESEARCH CENTRE
EUROPEAN COMMISSION

Institute for Advanced Materials

INTRODUCING *iam* in 1996

Institute for Advanced Materials

Improved knowledge and understanding in the scope of advanced and conventional materials as well as advanced engineering applied to citizen welfare and European industry competitiveness.

MISSION

The mission of the Institute is to contribute to enhancing the technical and scientific infrastructure of Europe and thereby its industrial competitiveness and citizen welfare, mainly in the energy, transport, environment, life science, manufacturing and nuclear sectors, through knowledge and understanding on advanced and conventional materials as well as advanced engineering.

The Institute specifically supports European industry by providing state-of-art materials research and operating advanced facilities, and supplies neutral scientific support for the preparation of EU directives.

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The Work of the Institute

The work of the Institute can be defined as follows :

1. to conduct basic and technological research and development of advanced materials relevant to their development in industrial applications.
2. to conduct prenormative research in engineering materials which contributes to European industrial and safety standards,
3. to develop and validate non-destructive methods for checking the integrity of large critical industrial plant and components,
4. to develop and validate characterisation and processing of innovative materials in the field of photo-voltaic energy, energy savings in buildings and electricity storage.
5. to develop diagnostic and therapeutic techniques in the medical field, and orthopaedic devices
6. to promote integration and coherency in European research, resources, and scientific development by co-ordination and contribution to European networks in key materials-technology area
7. to improve the dissemination of information on materials, structural aspects, and advanced energy systems, in particular towards Small and Medium-sized Enterprises, by international networking.

The IAM performs basic fundamental and mechanistic research to the development and characterisation of industrial materials, components and testing methodologies.

Fields of activity include:

- High Temperature Materials Technology
- Surface Modification Technology
- Advanced Testing Methodologies for Materials and Components
- Next Generation Materials
- Rejuvenation of Aged Materials
- Materials Recycling and the Environment
- Thermonuclear Fusion-Related Materials Technology
- Photovoltaic Systems
- Rational Use of Energy

The results are competitive products and services delivered to the customers of the Institute: the European Commission for the Research and Technological Development Framework Programme, Various Directorates General of the European Commission, European Industrial Companies, research institutes and licensing authorities the "human capitol" of Europe's scientific community, and European citizens in general.

The Future

The Institute plays an essential role at the European Union level. The field of materials is a generic one, with implications common to most human activities, industrial as well as social. Rapidly changing demands on the industrial market as well as new demands for citizen welfare generate fresh requirements, which demand a steady stream of new materials, or novel conditions of application for existing ones.

Pre-normalisation, characterisation and the supply of reliable information, together with the interpretation of the scientific results and their translation into models usable by the engineer and manufacturer, requires collaboration, networking, and co-ordination of activities on an international scale. The IAM aims to fulfil this need in the EU context within its area of competence.

Organisation

IAM scientific activities are carried out by eight units, each focusing on different aspects of materials technologies:

- Materials Engineering
- Surface Engineering
- Structural Component Integrity
- Structural Materials and Tritium Technology
- Energy Systems Testing
- High Flux Reactor
- Cyclotron
- Testing, Analysis and Mechanical Engineering

There is also one Unit for Administration and a Directorate Support Group. This group includes Marketing and Information tasks.

Financing the Institute

The table below show the IAM expenditure on the main programme lines during 1996. It also indicates the number of scientific staff allocated to each of the research areas.

Programme Line: Institutional	**Expenditure, MECUs**	**Scientific and Technical Staff**
Industrial and Materials Technologies		
Advanced Materials, Standards for Applications	9,93	48
Surface Engineering	8,35	34
Recycling and Eco-technology	1,67	13
Non Destructive Testing	1,58	8
Sub-Total	*21,53*	*103*
Non Nuclear Energy		
Photovoltaics and Energy Storage	2,31	12
Materials for Clean Combustion	1,53	7
Sub-Total	*3,84*	*19*
Nuclear Safety		
Eur. Networks on Ageing and Inspection (research)	1,51	12
Eur. Networks on Ageing and Inspection (support)	1,08	9
Sub-Total:	*2,59*	*21*
Controlled Thermonuclear Fusion		
Fusion materials	1,85	11
Fluid Separation and Analysis	7,01	22
Sub-Total	*8,86*	*33*
Sub-Total Institutional	36,82	176
Programme Line: Competitive		
Materials areas competitive work	3,27	17
Programme Line: HFR		
Supplementary programme	10,4	18
Isotope production	5,92	5
Other competitive	0,66	1
Subtotal HFR	*16,98*	*24*
Institute Total	57.07	217

The following table gives the amount of signed contracts in 1996, (figures are in KECUs):

IAM (excl. HFR)	
SCA (100%)	2506
CSA	567
TPW	1144
Sub-Total IAM (excl. HFR)	***4217***
HFR	
SCA (100%)	812
TPW	6184
Sub-Total HFR	***6996***
Total IAM (incl. HFR)	***11213***

STAFFING OF THE INSTITUTE

At the end of 1996, the Institute had 234 statutory staff plus 43 temporary staff (PhDs, post-doctoral and industrial fellows, seconded experts) financed by the Institutional programme and the Competitive programme. In addition, 29 scientific, technical and administrative staff were supported by the HFR programmes which bring the total of the Institute to 306 people. Non scientific staff including administration, directorate support, and all clerical and secretarial staff in scientific units amounts to 46 staff members.

The breakdown of the IAM staff according to grade was as follows;

Statutory staff on Institutional and Competitive programmes	
Scientific/Professional staff (A grade)	71
Technical and administration support staff (B grade)	103
Skilled, clerical and secretarial staff (C/D grade)	61
Sub-Total	***234***
Visiting staff	
Post doctoral fellows	13
Doctoral student	14
Visiting Scientists	2
Short term staff	14
Sub-Total	***43***
Statutory staff on HFR programmes	
Scientific/Professional staff (A grade)	9
Technical and administration support staff (B grade)	9
Skilled, clerical and secretarial staff (C/D grade)	11
Sub-Total	***29***
Total	***306***

RADIOISOTOPE PRODUCTION

The intention of the Institute is to increase market share where it can demonstrate its competitive advantage. In particular, penetration for medical isotope production in the HFR is one of these markets. In view of the high market share already achieved in Europe only a limited gain can be targeted. On the world market less ambitious figures are indicated due to the strong local nature of the medical isotope business.

Market Penetration for HFR Produced Medical Isotopes

	1995	1996
% European Market	65	75
% World Market	25	27
Total sale in KECUs	5100	5924

THE INSTITUTE NETWORKS

IAM is involved in international networks related to several of its institutional activities.

These networks are related to Nuclear activities, both Fission and Fusion, and to Renewable Energy.

In 1996, the IAM managed five international Networks which have grown from the earlier PISC Programme.

AMES: Ageing Materials Evaluation and Studies
NESC: Network for Evaluating Steel Components
EPERC: European Pressure Equipment Research Council
ENIQ: European Network for Inspection Qualification
ENAIS: European Network For Advanced Information System

For the Thermonuclear Fusion related activities, IAM was involved in two Networks:

The first deals with Advanced Materials for DEMO (DEMOnstration reactor), a network managed by IAM on the development and characterisation of low activation materials.

The second network, for which IAM has the leadership, has the main task to produce a Materials Property Handbook, based on the collection and evaluation of data produced by different EC labs. This constitutes an input to the International Data Bank for ITER (International Thermonuclear Experimental Reactor).

As a target for 1997, a new network will be organised with laboratories and industries involved in the Thermal Fatigue Testing of Primary Wall Mock-ups and Components.

In the Renewable Energy field, IAM was observer in two networks within the tasks of the International Energy Agency, namely:

Demand Side Management
Photovoltaic Systems

Within Europe, a Network of Renewable Energy Laboratories will be established in 1997, with the scope to form a structure for component tests leading to a quality label for decentralised photovoltaic systems.

A second target for 1997 will be to create a Network of European Energy Efficiency Centres, with the aim to exchange and harmonise information, results and data of European energy efficiency programmes.

STANDARDS, CODES AND INDUSTRIAL PRACTICES

In 1996, the IAM contribution to standards, codes, and industrial practices committees were the following

Contributions to Technical Committees in 1996:

- Membership in CEN BT Working Group 70 STAR (standards and Research Interaction) and CEN STAR Subgroup on Pre-normative Research
- Membership in IEC (International Electrotechnical Commission), Technical committee 82 (Photovoltaic Solar energy), Working Group 2 (Modules) and Working Group 3 (Systems).
- Membership in PEP (Photovoltaic Energy Project) of the Versailles Project on Technology, Growth and Employment.
- Membership of Executive Committee of PV-GAP (Photovoltaic Global Accreditation Programme), responsible for test standards.

CEN Technical Committees (TCs), contributions

- TC 54 Unfired Pressure Vessels
- TC 121 Welding
- TC 138 Non-destructive Testing
- TC 184 Advanced Technical Ceramics
- TC 262 Protection of Metallic Materials against Corrosion

ISO Technical Committee 164

Pre-normative Research Bodies, Contributions

- VAMAS, the Versailles Project on Advanced Materials and Standards
- ESIS, the European Structural Integrity Society
- MTMTC, the European High Temperature Materials Testing Committee
- CODATA, the Committee on Data for Science and Technology of the International Council of Scientific Unions

MAIN ACTIVITIES

CONTENTS

The Work programme of the Institute, is divided into the following major activity lines:

Advanced Materials, Standards for Applications

Surface Engineering for Improved Properties

Non -Destructive Examination and Inspection

Recycling and Eco-Technologies

Materials for Clean Energy Technologies

Reactor Safety Networks for Component Ageing and Inspection

Materials for Controlled Thermonuclear Fusion

Photovoltaic Energy and Energy Conservation

What follows is a summary of the objectives and achieved results in 1996

SPECIFIC RESEARCH THEME: INDUSTRIAL TECHNOLOGIES AND MATERIALS TECHNOLOGIES

Research Area: Advanced Materials, Standards for Application

Final User: Aerospace, Materials Manufacturing and Petro-Chemical Industries, Power Generation, Standards Organisations

Objective: to promote the more efficient use and improved performance of advanced materials by:

- assessment and evaluation of material behaviour
- catalysing breakthrough of novel materials
- pre-normative research and support to standardisation within CEN, ISO and related bodies

1996 Keywords and Goals:

Experimental assessment & modelling of mechanical performance:
- Model for predicting the rafting of precipitates in superalloys under uniaxial and triaxial stress loading

- **Result:** Task completed by establishment of a microstructurally informed model using among others Monte Carlo simulation. Work published.

Interface aspects in composites and joining:
Application of a novel joining route for the cost-effective fabrication of refractory ceramic joints (service temperature in excess of 500oC)

- **Result:** Development in the area of transient liquid phase brazing of Silicon Nitride. Four publications.

Advanced corrosion studies and corrosion protection:
- Installation and commissioning of corrosion test facility for chlorine-containing environments

Result: The test facility has been installed and started operation at the end of 1996. Commissioning tests extend into 1997.

Residual stress measurement, optimisation of CMC's processing:
- Extension to novel processing techniques for non-oxide 2D composites for next generation wear components

Result: Residual stresses and microstructure were investigated by neutron diffraction in two types of CMC's, i.e., C/Si3N4 and Si/SiC. It is evident that neutron diffraction studies are essential for the optimisation of CMC's processing as they provide valuable information concerning material homogeneity and isotropy in addition to residual stress levels.

Extended evaluation & modelling of brittle behaviour of bcc alloys:
- Determination of the temperature dependence of the fracture toughness of high-purity chromium

Result: Set-up of a facility for fracture toughness measurements of sintered high purity chromium in the range of 20°C to 300°C

SPECIFIC RESEARCH THEME: INDUSTRIAL TECHNOLOGIES AND MATERIALS TECHNOLOGIES

Research Area: Surface Engineering for Improved Properties

Final User: Aerospace, Mechanical and Petro-Chemical Industry

Objective:
- to stimulate industrial acceptance of rapidly evolving surface engineering technologies
- to facilitate introduction of surface engineering applications by qualification techniques and procedures
- to foster research co-operation on surface engineering

1996 Keywords and Goals:

Corrosion and Wear:
- Application of Thin Layer Activation methods for measuring wear on nitrided surfaces

Result: The use of Thin Layer Activation (TLA) method was successfully applied in a pin on disk wear testing of a ZrN coated material, in the frame of potential biomaterial application. The TLA method proved to be orders of magnitude more sensitive than conventional gravimetry, thus proving the advantages of the method in the development of very wear resistant materials or surface treatments.

Fatigue:
- Determination of the effectiveness of laser surface melting of stainless steel in improving fatigue resistance

Result: Studies on AISI 316L steel regarding improvement of the high cycle fatigue behaviour after laser surface remelting have been completed and fatigue damage parameters have been identified by X-ray diffraction measurements. A European Network for hardness measurements using photothermal methods has been set-up.

Synthesis and characterisation of advanced coatings:
- Evaluation of MCrAlY coating containing small percentage of silicon

Result: MCrAlY coatings containing silicon exhibited significantly improved corrosion resistance after an appropriate heat treatment and are particularly interesting for protection of components in contact with dirty fuels at high temperature.

Biomedical applications:

- Testing of oxygen and chromium implanted titanium

Result: Wear of Ultra High Molecular Weight polyethylene (UHMW-PE) specimens against oxygen and chromium implanted Ti_6Al_4V was reduced two and three orders of the magnitude, respectively, as compared with a wear against un-implanted Ti_6Al_4V.

Thermal barrier coatings:

- Sealing with SiO_2 of porosity of ZrO_2 coatings. A patent proposal on the use of SiO_2 in sealing of porosity of ZrO_2 coatings to be submitted.

Result: Oxidation in thermal barrier coatings (TBC) have been modified by the application of an exterior Metallo-Organic Chemical Vapor Deposited (MOCVD) SiO_2 layer and the resultant samples showed a reduced porosity of conventional YSZ based TBC. High temperature exposure of the modified coatings show a reduction in the oxidation rate in air although no improvement could be found when exposed to more aggressive $Ar/10\%O_2/0.3\%SO_2$ atmosphere (patent application filed).

Chemical sensors:

- Tests on sensors for urban pollutant gases CO and NOx performed in the city of Milano

Result: Sensors based on tin oxide semiconductor were tested and compared with spectroscopic methods in a controlling station in the city of Milan. The tests revealed that after the adjusting period of about 10 minutes, both in the case of CO and NO_2, the sensors in their continuous recording followed fairly well the spectroscopic testing based on discontinuous sampling of the atmosphere. After about 23 h the sensing surfaces became saturated. The possibility to regenerate the surface by heating is being investigated.

SPECIFIC RESEARCH THEME: INDUSTRIAL TECHNOLOGIES AND MATERIALS TECHNOLOGIES

Research Area: Recycling and Materials for Eco-Technology

Final User: Automotive Industry, Mechanical Industry, Consumers

Objective: Organisation of a focal activity for the development and operation of a European Materials Eco-Technology Network.

1996 Keywords and Goals:

Life Cycle Assessment:

- Comparison and establishment of practices for materials life cycle assessment. This activity is supported by a pilot study on Zn coated steel products

Result: Comparison of products/materials/processes have been established as effective methods of employing life cycle inventories. A pilot study of comparing traditional Zn-coated steel with PVD Zn-coated steel, for the automotive industry, has been carried out.

Database:

- Life Cycle assessment data base: to set up a data source networks and methodology for data exchange and data quality management

Result: Participation in development of methodology and data source network, for data exchange and data quality management. A proposal with European partners, for the Environment and Climate Program (DG XII) is developed.

Applications:

- Substitution of Zn in Zn coated steels by alternative coatings, especially in automobile sectors, thus decreasing environmental hazards

Result: A search in journals, patent files, in national and supra-national projects has been performed to identify candidate materials for hot dipped Zn coatings presently used in the automobile sector. First experiments with Zn-Al alloys were performed and show promising results.

Development of radiotracer methods:

- this is an inter-institute activity with the Environment Institute, for which IAM, by its capability to produce radio-tracers with the Cyclotron, addresses two aspects: -release of Pt and other heavy metals from gas catalyst, -release of heavy alloy constituents from technical components in food processing.

Result: In collaboration with the Environment Institute, radiotracers of Pt, Rh, and Pd were produced for toxicology studies, related to the use of gas catalysts. In the frame to study heavy metal contamination of nutrition products by corrosion of technical components in the food processing industry, an advanced facility has been developed and became operational at the end of 1996. This facility which combines (classical) electrochemical test methods with Thin Layer Activation method serves a wide range of applications.

SPECIFIC RESEARCH THEME: INDUSTRIAL TECHNOLOGIES AND MATERIALS TECHNOLOGIES

Research Area: Non Destructive Evaluation: Testing and Inspection of Structural Components.

Final User: Engineering Components Manufacturers

Objective: To enable effective use of NDE technique for pressure vessels and piping equipment, to develop and validate advanced NDE techniques for power generation components, to identify and assess NDE techniques for reliable operation and remanent life prediction.

1996 Keywords and Goals:

Prediction of NDE capability:
Further analysis of PISC results is still requested in the frame of steam generator tubes inspection in view of a difficult agreement to be found between all the EU and American partners on the non-effectiveness of inspection in this particular case and on the low cost methods proposed by JRC / IAM to qualify techniques of inspection .

Result: Further analysis of PISC data and report on heat exchangers inspection

Components and coatings characterisation:
Development and application of characterisation of ceramic and metallic coatings lead to results presently considered for more industrial application by the development of special ultrasonic transducers and procedures

Result: Report completed and published

Qualification:
The European Methodology for Inspection Qualification is applied in several countries and is the object of a pilot study at JRC, valid for pipe-line inspection. Proposed to CEN TC 135

Result: Document issued

Damage and degradation:
Hydrogen damage in steel containers is detected by neutron radiography techniques. The expected but the activity is now concentrating on the development of a patent for hydrogen storage. A patent was proposed for the design of hydrogen containers.

Network:

- Launching of EPERC network

Result: EPERC started in October 1995 and the general assembly of November 1996 was a success. It confirmed the options and directions chosen for technical co-operation as well as for the enhancement of the corporation of the pressure equipment manufacturers and users.

SPECIFIC RESEARCH THEME: NUCLEAR SAFETY AND SAFEGUARDS (INSTITUTIONAL RESEARCH).

Research Area: European Networks on Components Ageing: Inspection Techniques and Structural Integrity.

Final User: Nuclear Fission Industry, Safety Authorities, Standards Organisation

Objective:
- To contribute through research to the study of ageing mechanisms in critical structural components in Nuclear Power Plant (NPP), including management of a European collaborative network
- To gather the technical background necessary for the continuation of ENIQ
- To establish an international forum for agreement of methods of structural integrity assessment.

1996 Keywords and Goals:

LYRA rig
The LYRA rig is completed and the pre irradiation quality control are being done. The rig is fully booked for irradiation in 1996 and 1997 for the Shared Cost Action and AMES network members

Design rules for pressure vessels:

Result: NESC I is in its second phase (spinning in March 1997 in presence of authorities)
Forerunners of other NESC projects were developed and financed (BIMET, SINTAP,...)

Stress corrosion damage:
- Definition of ENIQ pilot study on austenitic piping

Result: The ENIQ pilot study is continuing and being evaluated by the operators and the authorities. All assemblies, inspection procedures, qualification procedure and documents were prepared in 1996 as well as the assembly of the industrial testing equipment.

SPECIFIC RESEARCH THEME: NUCLEAR SAFETY AND SAFEGUARDS (INSTITUTIONAL SUPPORT TO THE COMMISSION).

Research Area: European Networks on Components Ageing: Inspection Techniques and Structural Integrity.

Final User: DG XI, DG XVII

Objective: To develop and orient activities of the Networks AMES, ENIQ and NESC in support of EC Directorates in charge of reactor safety and energy policy.

1996 Keywords and Goals:

Reach a consensus on safety matter in agreement with DGXVII and DG XI.
- Harmonisation of codes and standards on industry practices in service inspection and safety assessment:

Result: The RSEM code, the Swedish ISI practice and IAEA guidelines are using the ENIQ work for the issues prepared or issued in 1996.

Working groups
- Support to DG XI

Result: Codes & Standards Working Group, Nuclear Reactor Working Group, and Reactor Safety Working Group.

- Support to DGXVII

Result: Co-operation with Eastern European Countries for DG XVII (see below).

- Development of support to DG I for DG XI and DG XVII.

Result: Experts of the SCI unit participated in several evaluations to support DG IA TACIS and PHARE actions.

Information transfer:
- Definition of the new network ENSIT for information transfer on Structural Integrity

Result: Network ENSIT proposal was detailed but financial means were not identified such as to be able to start the work as an independent activity.

Central and Eastern Europe:
SCI unit called as subcontractor by Belgatom, Tecnatom and Skoda to support and contribute to PHARE actions on Inspection Qualification in the Czech Republic.
- Performance of subcontracts for Phare activities towards Czech republic

Result: EPERC has developed Task Group 5 under the leadership of the JRC / IAM.

SPECIFIC RESEARCH THEME: NON NUCLEAR ENERGY

Research Area: Materials for Clean Technologies

Final User: Car Manufacturing Industry

Objective: To develop improved materials for reducing automotive exhaust emissions, with a longer operating lifetime of the catalytic converter.

1996 Keywords and Goals:

Automotive exhaust emissions, catalyst carrier:
- To develop a method for making a nanoscale dispersion of catalytically active noble metal within the novel catalyst carrier

Result: A method was developed to distribute nanoscale dispersions of noble metals in the improved alumina catalyst carrier material developed by IAM in 1995.

- To develop a method for producing thin film coating of the new material on existing cordierite catalytic converter

Result: A method was developed to coat cordierite honeycomb with thin and uniform layers of the newly developed catalyst system described above.

- To investigate and characterise environmental degradation mechanisms of current and newly developed ceramic catalyst support carriers

Result: The study of environmentally induced degradation mechanisms of current ceramic catalyst support systems was completed. The main conclusion was that the degradation of catalytic behaviour during the lifetime of an automobile catalytic converter is not necessarily only a result of catalyst poisoning or spallation caused by temperature excursions but may also result from thermochemical cycling.

Ceramic:

- To investigate alternative support materials and catalyst promoter systems in models and simulated automobile exhaust environments.

Result: Studies of newly developed and alternative systems was suspended pending the replacement of personnel as a result of internal mobility and the delivery of new materials.

Thermomechanical Behaviour of SiCf / SiC Composites

- The development and characterisation of ceramic composites as materials for environmentally friendly energy applications was continued.

Result: Bending tests at 800 (C on 2-D SiCf/SiC composites, produced by Chemical Vapour Infiltration (CVI), were carried out for samples annealed in vacuum and in He-atmospheres. Temperatures of 800°C were considered for the isothermal ageing cycles, for 100 hour periods. Helium atmospheres contained residual amounts of oxygen and hydrogen, which could be detrimental for the integrity of the fibre/matrix interface. The results indicate that thermal ageing effects at this temperature on mechanical properties are negligible.

SPECIFIC RESEARCH THEME: NON NUCLEAR ENERGY

Research Area: Photovoltaic Energy and Electricity Storage

Final User: European Photovoltaic industry, accumulator industry, standards making bodies.

Objective:

- Development and operation of diagnostic testing and monitoring techniques for Photovoltaic components and installation for electricity storage.
- Research on methodologies and procedures to be standardised on a European or International level

1996 Keywords and Goals:

PV Component testing:

- Implementation of automatic correction procedures at ESTI's flash-simulators

Result: Programme WinLappss takes into account actual irradiance measurements.

Performance evaluation:

- Design parameters for new encapsulation material for spherical solar cells

Result: Parameters for a new encapsulant ready forecasting 17% efficiency increase, with non significant cost increase. Contacts established with Ontario Hydropower (CND) for possible incorporation within production line. Five publications have been issued on the subject until now.

- Capacitance effects in high-efficiency solar cells

Result: Patent filed in April 1997 concerning On-chip tuneable capacitor.

Diagnostic:

- Diagnostics of Photovoltaic facades

Result: Thermal imaging on Ispra facade executed. No failures after, 12 months of monitoring data analysed and presented at conference, and only one slight degradation observable (<5%). Conversion into a Hybrid PV-Thermal system is a scope of a proposed Shared Cost Action for the Joule III call for proposals.

System design and Performance:
- Low-cost intelligent Solar radiation sensor: concept

Result: Microcontroller system developed and optimised for low-consumption. On-board storage of data possible for 6 months of monitoring. Independent power-supply presently not feasible. Prototype circuit board design.

Standards:
- Participation in IEC-Committee (Thin film qualification, Product Quality Assessment)

Result: Participation in IEC TC 82 WG2 / WG3. Thin-film qualification test specification of JRC modified and finalised. Standard accepted. JRC proposed New Work Item on Tracability of Reference Cells and Type Approval of PV stand-alone systems. As executive committee member of GAP (Global Accreditation Programme), JRC proposed affiliation with IEC Quality Assessment System. A memorandum of Understanding was drafted.

SPECIFIC RESEARCH THEME: FUSION

Research Area: Fluid Separation & Structural Analysis

Final User: European Fusion Programme and related industries

Objective:
- research on tritiated gas treatment for the fusion fuel cycle
- to develop management methods for waste

1996 Keywords and Goals:

Measurements on irradiated Be samples:
- Set up and testing of the de-tritiation process plant and measurement loop

Result: Report to SEAL 1.4.1: "Apparatus for in-situ oxidation of Be samples and for measuring its effect on tritium out-gassing: construction and calibration." June 1996.

Recycling and disposal:
- Assessment of radioactivity limits for recycling and disposal of activated and tritiated wastes

Result: Reported in SEAL/R-10.1, Rev:2(96) "Definition of limits for Recycling and Clearance and Application to Fusion Materials", and published J. Nucl. Mater., 233-237 (1996) 1500-1504.

Waste monitoring:
- Development of a new method for tritium assaying in low level soft waste

Result: Patent applied for. Report sent to EFTP after filing patent Feb. 1997

Real time analysis, inventories:
- New high level tritium monitor for controlling tritium processes: commissioning and calibration

Result: Patent application made, before publication Sept. 1996 of paper on new tritium monitor based on gas scintillation

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SPECIFIC RESEARCH THEME: CONTROLLED THERMONUCLEAR FUSION

Research Area: Fusion Materials

Final User: European Fusion Programme and related industries

Objective:
- to develop and characterise low activation materials for long term technology
- to support ITER's technological needs for material selection

1996 Keywords and Goals:

Thermal fatigue testing on small mock ups:
- Completion of thermal fatigue testing of an explosive welded copper-stainless steel mock up
- Upgrade of the test facility for testing First wall mock-ups specimens with Be-coating

Result: No severe damage observed after 13 000 thermal cycles under ITER relevant conditions

ITER material properties handbook:
- Loading in the ITER Material Properties Handbook of final data on 316 LN (IG) S.S. and Cu-Cr-Zr alloys

Result: Input of fracture toughness through data of irradiated and un-irradiated AISI 316 LN (IG) stainless steel and Input of thermomechanical data for precipitation hardened Cu-Cr-Zr alloys

Non ferrous low activation alloys:
- Post irradiation bending tests of SiC/SiC specimens irradiated in HFR up to 5 dpa.

Result: Post irradiation bending tests of neutron irradiated material show significant material degradation after fluences > 2dpa.

Fusion materials irradiations:
- Data of the low temperature irradiation creep rate in 316 LN (IG) and welds

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SPECIFIC RESEARCH THEME: CONTROLLED THERMONUCLEAR FUSION

Research Area: Tritium Materials Interaction

Final User: European Fusion Programme and related industries

Objective: To provide basic data for the accurate design of next step fusion reactor with regard to tritium interaction with materials

1996 Keywords and Goals:

Tritium permeation reduction by advanced coatings:
- Development and characterisation of TaO_x coating on martensitic steel

Result: Deposit of TaO_x coating on martensitic steel have been performed by CVD. The permeation reduction factor (PRF) obtained has been quite small PRF=10. This kind of layer has been evaluated not suitable as permeation barrier and a different material has been chosen.

Tritium recycling parameters:
- Completion of absorption and recombination constants measurements on MANET

Result: Experimental runs in order to obtain data to compute the values of the recombination and absorption constraints of MANET has been performed. Preliminary data has been obtained. Further development of a model for data processing is however needed and is under way.

Transport parameters of tritium in different candidate materials:
- Tritium permeation rates data on F82H modified steel

Result: All the tritium transport parameters (diffusivity, solubility, permeability, surface rate constant and trapping characteristics) in F82H modified martensitic steel have been measured. The results have been published in the open literature. As expected, the values of diffusivity, solubility, and permeation rate are not very different from those for MANET steel.

MANAGEMENT GOALS AND ACHIEVEMENTS IN 1996

- Strategy, Organisation and Evaluation:

- During 1996, the Institute Strategy Framework was defined and presented to top management of the JRC.
- Linked to this definition of a new Strategy, a new Organisation was designed and implemented in September 1996.
- Improvement on procedure on Project review and Programme review were introduced in 1996 and will be applied in February 1997.

- Towards Total Quality Management:

- As a major achievement for the past year, the ESTI laboratory was accredited in September 1996.
- The Institute Quality Policy and Quality Strategy were issued respectively in July 1996 and in September 1996 (as first draft).
- The Institute Quality Structure is in place since November 1996. Selection of Quality Officers was accomplished and training of the Quality Officers is ongoing.
- Definition of the various main processes is ongoing since November 1996. The definition of the list of required IAM Corporate Procedures was done according to ISO 9001 and Guidelines were issued in December 1996.

– Marketing:

- The design of new IAM Marketing Plan First draft was completed in December 1996.
- The First Customer Satisfaction Assessment process was started targeted to Customers representatives selected by market sectors. The questionnaires were defined in November 1996 and the first assessment is planned for May 1997.

MAIN FACILITIES

LIST OF FACILITIES

CONTENTS

DESCRIPTION OF FACILITIES

45 MW High Flux Reactor (Petten, the Netherlands)

The High Flux Reactor (HFR) is one of the most powerful multi-purpose research and test reactors in Europe. Activities include:

- nuclear fission and fusion energy research
- providing neutron beams for analytical applications (e.g., neutron activation analysis, neutron radiography) and further research (solid state physics, materials science, medical therapy)
- producing radioisotopes for scientific, industrial, medical and agricultural purposes

40 MeV Variable Energy Cyclotron (Ispra, Italy)

The Cyclotron is used in the IAM's research programme into Thin Layer Activation, a powerful analytical technique where a high energy particle beam (produced by the cyclotron) strikes the sample, creating radionuclides in its surface layer. This 'activated thin layer' them emits gamma radiation, which can be analysed to provide information on the material's behaviour under working conditions.

The Cyclotron is also used to produce radioisotopes, particularly radiotracers for environmental monitoring.

European Tritium Handling Experimental Laboratory, (Ispra, Italy)

The European Tritium Handling Experimental Laboratory (ETHEL), was created to perform research into the hydrogen isotope tritium – a vital component of nuclear fusion fuel.

European Solar Test Installation,(Ispra, Italy)

ESTI is a reference laboratory for photovoltaic (PV) technology which recently received its accreditation. Apart from characterising and testing PV systems for European industry, ESTI also carries out research into new testing techniques and develops the necessary software.

Non Destructive Evaluation Laboratory (Petten, the Netherlands)

NDE Laboratory (Petten, the Netherlands). Two major techniques are available in this laboratory, namely ultra-sonic testing and X-ray radiography. This latest is located in a dedicated bunker including a 2MeV Linear Accelerator, 3 X-ray machines respectively from 200, 300 and 450 kV, one micro focuses x-ray device of 200 kV for research and industrial radiography. The ultra-sonic equipment is composed of an extensive range of sensors and analysis apparatus.

Environmental Testing Laboratory, (Petten, the Netherlands)

The ETL is used for testing materials under a wide range of environmental stresses. Aero-engine manufacturers, for example, have used the ETL to place new turbine blade materials and coatings under the simulated stress of thousands of hours in the combustion chamber of an aero-engine. Microstructural analyses then pinpoints how – and, eventually, why not – the material dealt with these stresses.

Surface Engineering Laboratory, (Ispra, Italy)

The major activities of the Laboratory are centered around the implementation and qualification of advanced coatings and surface modification methods in European industry, especially manufacturing, and to develop new coatings on specific request. Amongst the major installations are a High current ion implanter, a Low Pressure Plasma Spraying (LPPS) unit, a Distributed Electron Cyclotron Resonance (DECR) Plasma Assisted Chemical Vapour Deposition (PACVD) unit, a Inductively Radio Frequency Plasma Assisted Chemical Vapour Deposition (rf – PACVD) unit and a Inductively Coupled Plasma Assisted Chemical Vapour Deposition unit

UTILISATION OF CAPITAL FACILITIES

Figures are appended for the utilisation of three major facilities in the IAM, the HFR and the ETL at Petten and the Cyclotron at Ispra. The objective here is to maximise the availability of the facilities for the customer projects whilst remaining within the limits of safe operation and at the same time to increase the throughput of work.

HFR Utilisation	1995	1996	
		target	achieved
Operation days per year	288	285	291
% Occupation of irradiation positions	75	88	85

ETL Utilisation	1995	1996	
		target	achieved
Operation days per year	180*	350	351
% occupation predicted (active positions)	82	50	65

* major repair period included

Cyclotron Utilisation	1995	1996	
		target	achieved
Operation days	150	150	150
micro-amps.* hours	18000	18000	20000
availability for isotope production (in %)	95	98	100

PARTNERS

& Customers

IN BRIEF

The Institute for Advanced Materials collaborates with a wide range of companies and research institutes across the many sectors where advanced materials technologies are valued.

It receives a significant amount of funding from industrial companies in the form of Third Party Work, and is involved in 27 new Shared Cost Actions with companies and research institutes around Europe.

It is also the Operating Agency of a number of European networks involved in non-destructive examination techniques for the nuclear and non-nuclear sectors, in Fusion technology, and in renewable energy field.

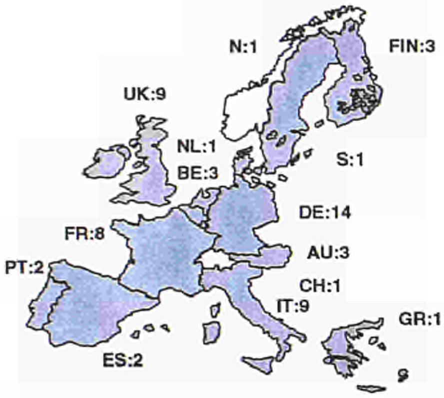
CONTENTS

SHARED COST ACTIONS

During 1996, IAM was involved in 55 proposals for Shared Cost Actions following calls for tender for programmes such as Brite Euram, Standard Measurements and Testing, or Nuclear Fission Safety. From those proposals, 27 were accepted for funding.

These SCAs involve several research institutes and major companies from around Europe. As an illustration, the following figure gives the partnership distribution for the successful proposals in the Brite Euram programme.

IAM partners in 1996 Brite Euram successful proposals in 1996



The following table gives the ranking of those proposals, were it should be noted that A, A1, and A2 proposals were retained for funding.

	A/A1	A2	B	C	Total
Brite-Euram	4	6	11	3	24
Biomedicine and Health				1	1
ESPRIT				1	1
JOULE	8		9		17
Nuclear Fission	4	2		1	7
SMT	3		2		5
Total	19	8	22	6	55

COLLABORATION WITH INDUSTRY

The IAM supplies many European industrial companies with expertise in advanced materials technologies through Third Party Work.

To begin with, the Institute's High Flux Reactor dominates the Europe market in radioisotopes (70% market share) and has made serious inroads internationally (27% global market share).

Moreover the IAM has clients, past and present, in various sectors such as electricity utilities, including nuclear operators, aeronautics and space systems manufacturers, petrochemical industries, engineering manufacturing industry, consumer electronics and appliance producers, inspection companies, licensing authorities, and more. The IAM is also marketing its services to sectors such as the marine and off-shore industry, car manufacturers, environment and pharmaceutical agencies, biomedical industries and pulp/paper manufacturers.

COMPETITIVE SUPPORT TO EUROPEAN COMMISSION SERVICES

The Institute's Competitive Support contracts won in 1996 are mainly in the technology transfer and validation activities for the Innovation Programme (DG XIII/D):

Out of 12 proposals, the following 6 were selected:

- Coatings for High Temperature Protection of Carbon-containing Ceramic Matrix Composites
- Innovative Long-carbon Fiber Reinforced Ceramic Matrix Composite Materials for Industrial Wear Applications
- Conversion Software with graphical User Interface for MRI and CT Image Data

- Corrosion Test Manager CTM

- New Concept of Pressure Vessel Design in Order to Avoid Hydrogen Attack
- Prototyping of an Automated Video Image Capturing system (PAVICS)

OTHER COMPETITIVE ACTIVITIES

The IAM was also active during 1996 with the TACIS and PHARE programmes.

Institute for Advanced Materials

IAM contribution in 1996 to Research and Technological Development

INDUSTRIAL TECHNOLOGY: ADVANCED MATERIALS-STANDARDS FOR APPLICATION

MECHANICAL PERFORMANCE OF ADVANCED MATERIALS

Introduction

Hot path components of aero gas turbines are subjected to extreme demands for high temperatures structural strength and corrosion resistance. The continuing demand for economy of operation of the turbine calls for higher thermal efficiency, longer lifetimes and more reliable components. This project contributes to achieving these goals by evaluating the mechanical performance of advanced materials for hot path components, and by formulating and validating models to predict stresses and lifetimes. Two classes of materials are currently investigated.

a) coated single crystal nickel based superalloys, which are applied in the high and intermediate pressure stage blades of aero gas turbines, and which are on the verge of introduction in land based turbines.
b) monolithic ceramics and continuous fibre reinforced ceramic matrix materials (CMCs), which have potential for future application in static aero-engine components.

A key aspect of the evaluation of the performance of materials at high temperatures is the availability of advanced measurement and testing techniques, which eventually must be standardised in order to enable verified material data bases to be established and free trade to be promoted. In this context novel test and measurement instrumentation and techniques are continuously developed within the framework of this project. Finally, project staff are also involved in pre-standardisation research and in the drafting of standards within the framework of CEN, ISO and ASTM.

Mechanical Performance of Alloys

Research is focused on single crystal nickel-based superalloys for gas turbine blades. Coatings are applied to protect the blades from oxidation/corrosion. In view of their effect on the in-service performance of the blades, the choice and the optimisation of the coating towards maximising the lifetime are issues of major concern. The major cause of failure in current aero engine blades is thermal fatigue. In the laboratory the in-service behaviour of coated blades is closely simulated by means of thermo-mechanical fatigue (TMF) tests, which are designed to reproduce the temperature and strain cycles experienced by critical volume elements of the blade in service. (Fig. 1) is a close-up of a test specimen in a TMF testing rig using high-frequency induction for heating the specimen through the desired temperature cycle.

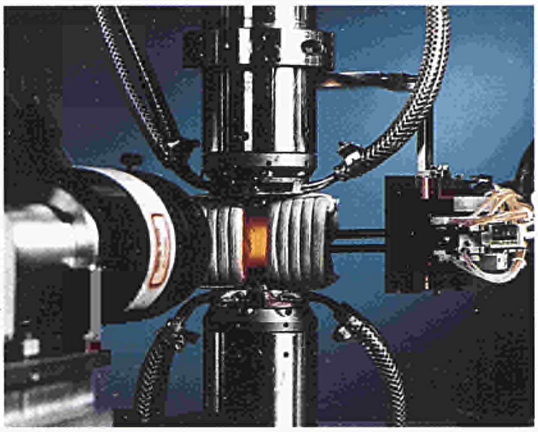


Fig. 1: TMF test set-up showing the test specimen heated by means of the high-frequency induction coil.

An evaluation of the effect of different diffusion and MCrAlY-type overlay coatings on the thermal-mechanical performance of various single crystal superalloys under TMF conditions, was completed in 1995. The effect of the TMF test parameters on the stress response and on the numbers of cycles to crack initiation and to failure was analysed. Work in 1996 focused on the analysis of the associated damage and failure processes in the coating. The aim is to derive guidelines for the microstructural tailoring of the coating towards improving coating life and to provide a mechanism-based input towards life prediction. Damage of coated blades in service and during TMF testing occurs in the form of micro-cracks. Optimisation of the in-service behaviour of coatings on blades requires an understanding of the damage mechanisms in control of the lower bound and of the average life of the coating. Hence the analysis must be structured so as to yield statistically significant data concerning the damage mechanism and its microstructural causes. To achieve this goal quantitative microstructural analysis techniques are applied to surfaces and volumes which are very large on the scale of the dimension of the microstructure. The evaluation of the ageing of the coating microstructure with time at temperature under TMF loading is part of this analysis. A microstructural study of the degradation of a NiAl coating on two different single crystal nickel-based alloys has been completed. The complexity of the coating microstructure is illustrated in Fig. 2, which also illustrates the changes occurring in the coating microstructure as the result of TMF loading. Various thermal histories were investigated, and the associated coating degradation mechanisms identified. Large differences in the microstructure evolution and damage are observed upon TMF cycling as opposed to LCF loading, providing evidence for the fact that TMF cycling does mimic the in-service blade behaviour whist LCF loading does not. Another major aspect of the analysis is concerned with generating data bases of crack statistics and with correlating the crack data with the microstructural in order to identify the microstructural features that cause crack initiation and that present paths for easy crack growth. To this end use is made of high magnification video images of the coating surface, collected in-situ during the TMF test, and of image scans collected at different depths of the coating / base material system following incremental polishing of the post test specimen. An example of the crack statistics of two specimens which have been TMF tested at small strain ranges and minimum-to-maximum strain ratios R=0 (A) and R= ∞ (B) is shown in Fig. 3. Peak values in the crack size and crack spacing distribution at the coating surface and in the outermost coating layers are related to the average B_2-NiAl grain size of the coating for both specimens A and B, pointing to a creep / oxidation controlled failure of grain boundaries. For intermediate depths (corresponding to the main coating to subcoating transition) the peak in the crack spacing distribution corresponds to the dendrite secondary arm spacing of the substrate alloy. Such a spatial periodicity suggests that the long which lead to failure of the specimen are generated in the subcoating and are associated with and influenced by the solidification structure of the single crystal substrate material.

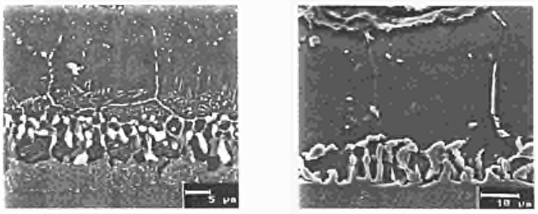


Fig. 2: Back scattered electron SEM image of an NiAl coating on single crystal RR2000 and phase identification. Upper images show the coating in the as received state, the lower images describe the situation after TMF cycling.

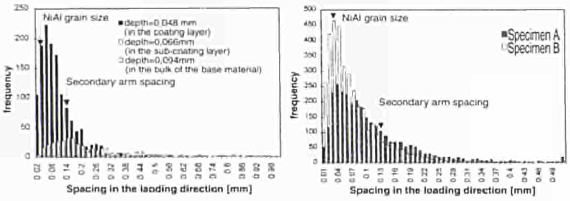


Fig. 3: Crack spacings along the loading direction of TMF tested specimens A (-135°lag, R=0) and B (-135°lag, R= -∞) at the coating surface (right) and at various depths in the coating / base material system (left, specimen A).

The in-depth evaluation of the mechanical performance of advanced materials at high temperatures frequently requires equipment and testing techniques which are not commercially available. Hence in-house design and development is required. In-situ video image acquisition at high optical magnification during mechanical testing and dedicated image analysis are techniques of major importance to our research projects. The capability of these techniques is extended on a continuous basis as part of our research programme. In 1995 a dedicated image acquisition system was developed for the quantitative analysis of microcracking at the surface and below-surface layers of TMF tested, coated specimens, and for the correlation with the local microstructure. The system software was expanded in 1996 with a view to improving specimen alignment and cutting down on measurement time. With respect to the in-situ microcrack monitoring for high temperature experiments, the design and construction of the prototype of a computer controlled imaging system has started. Commercial imaging systems have proved not to meet the demanding specification of high optical resolution (2mm or less), over large areas of material (up to 10x10 mm2) collected in-situ, which our testing programme requires. The system consists of a high-resolution digital video camera interfaced to a computer, and positioned on a three axis, high precision computer controlled motorized stage, permitting the video imaging of the surface sector by sector or in a single shot. The specimen to be examined is illuminated with a synchronized strobe lamp, which, along with the camera, is triggered when the sample is under maximum tensile stress. An interface mechanism to allow communication between the testing machine and the camera system has been built. A schematic diagram of the system is shown in Fig. 4. Work is well underway on the algorithms and the detailed specification of the software.

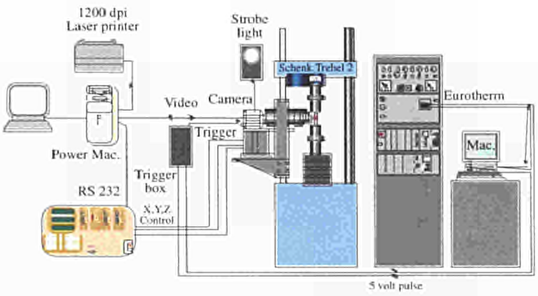


Fig. 4: Schematic of the automated, high resolution video imaging system for the in-situ monitoring of the specimen surface during mechanical testing.

Work in 1996 also focused on the evaluation and implementation of an appropriate heating system for the thermo-mechanical fatigue testing of thermal barrier coated (TBC) specimens. The thermal barrier coating in question is $ZrO_2.7Y_2O_3$. Two routes for heating were selected i.e. high frequency induction and infrared lamp radiation. The high frequency induction system was implemented on a TMF testing rig and the geometry of the induction coil was optimized to minimize the temperature gradient over the cross section and gauge length of the specimen. Despite the small negative temperature gradient of about 5°C over the TBC layer, which is inherent to the use of induction heating (which only couples to the metal bond coat and base materials), the system is very flexible and convenient in terms of strain control and accessibility of the test specimen. Whether the test setup correctly simulates the spalling of the TBC as the result of bond coat-TBC interface oxidation will be tested in 1997. Infrared lamp radiation for heating of the TBC coated TMF specimens presented two major problems i. insufficient absorption of the radiation by the test specimen as the result of the thermal properties of $ZrO_2.7Y_2O_3$ and ii. temperature measurement and control problems caused by the unknown emissivity coefficient which moreover changes during the TMF test. Following various modifications to the system, heating up to 1100°C at a rate of up to 10°C /s proved feasible.

Research results were disseminated by means of seven publications in scientific journals and at conferences. Staff members edited, respectively co-edited the proceedings of the International Symposia "Fatigue under Thermal and Mechanical Loading" held at Petten in 1995 and "Local Strain and Temperature Measurements" held at Berlin in 1996. The expertise accumulated in the context of the institutional research projects on gas turbine materials is often called for by industrial partners, inviting IAM to perform third party contract work or participate in Shared Cost Actions. In 1996 contracts were obtained from the VALUE programme and from several industries.

Mechanical Performance of Ceramics and CMCs

Advanced non-oxide monolithic ceramics and ceramic matrix composites reinforced by continuous ceramic fibres are being considered for load bearing applications at high temperatures. This subproject aims at the generation of reliable design data for these classes of materials, in order to enable their use in industrial high temperature applications.

Among the monolithic ceramics, silicon nitride and silicon carbide represent the largest potential, particularly for power generating applications. Mechanical tests on a hot pressed silicon nitride (HPSN) exposed to a simulated coal gasification environment at 1200 and 1300°C indicate that the material fails either through subcritical crack growth or through creep, which are both affected by cavity formation. The coalescence of cavities near the surface induces surface cracking. Below a threshold stress, the crack propagation rate equals that of the corrosion front, implying that there is no mechanical component to the crack growth rate. This threshold stress is a very important design parameter. The fact that the crack growth rate does not depend on the environment (see Fig. 5) indicates that a corrosion reaction at the crack tip is not the rate controlling mechanism for subcritical crack growth. The activation area lies between 7 x 10^{-22} and 1 x 10^{-21} m^2 in all situations. Also, a value of around 24 kT is obtained for the free energy ΔF. These figures agree nicely with the predictions from Schoeck's lattice trapping theory, of less than 1 x 10^{-20} m^2 and 20 kT, respectively, and suggest that subcritical crack growth is most likely controlled by thermal activation, independently of the surrounding gaseous environment.

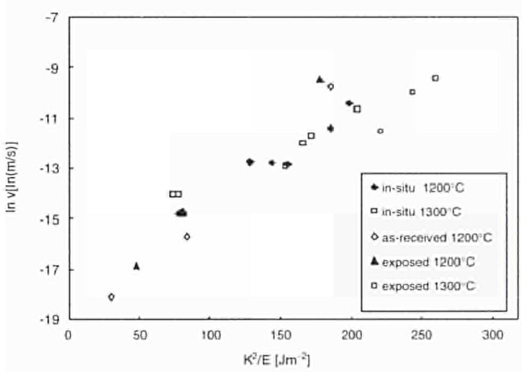


Fig. 5: Crack growth rate of HPSN under various experimental conditions plotted versus K^2/E according to the lattice trapping model

Surprisingly, the specimens showed evidence of active corrosion during the tests. This can only be caused by an extremely low partial oxygen pressure in the immediate vicinity of the surface. However, when the partial oxygen pressure is increased by increasing the humidity of the test gas, the corrosion behaviour remains unaffected. The insensitivity of the local partial oxygen pressure to the composition of the test gas clearly indicates that a local phenomenon plays a part. It was found that the electric potential difference between the surface of the (partially conducting) HPSN sample and the graphite susceptor used to heat it leads to an ionisation of the test gas and hence to a sharp decrease in the O2 activity near the specimen surface. This explanation is confirmed by the observation of active oxidation during air testing of a silicon carbide at 1400 °C heated by induction through a susceptor, whereas passive oxidation prevails when resistance heating is used.

During the year further evidence has been gathered showing that the existence of a positive fatigue effect in monolithic ceramics at high temperatures critically depends on the presence of amorphous intergranular phases. The damping capacity of these phases results in a hysteretic stress-strain behaviour. The amount of hysteresis depends on the stress amplitude, on the temperature and on the frequency. These dependencies lead to the conclusion that the fatigue behaviour at high temperatures is most probably controlled by viscous grain boundary creep. The quantification of the damping capacity of the amorphous intergranular phases through measurements of the area of the hysteresis loops has been compared to results obtained by internal friction. Taking into account the large difference in stress amplitude between the two loading modes, the results are consistent. The loss of positive fatigue effect with increasing number of cycles has been attributed to progressive devitrification caused by the heat treatment associated with time at temperature. This devitrification has been substantiated by XRD measurements.
The quantitative evaluation of hysteresis loops corresponding to unloading-reloading cycles in short-term tensile tests on ceramic matrix composites to determine the axial residual stresses in the fibres and in the matrix has been continued. The residual stresses and their evolution with temperature have been quantified for composites with opposite thermal expansion mismatch between the fibres and the matrix. The results agree nicely with literature data obtained on microcomposites (model composite consisting of a single fibre and the surrounding matrix mantle) and with those from experimental investigations using neutron diffraction. When the variation of the axial residual stresses between test specimens is taken into account, the scatter in the tensile behaviour (first matrix cracking stress, initial elastic modulus, fibre failure stress,...) dramatically decreases and the temperature dependence of these properties vanishes. Individual tensile curves obtained on a number of specimens over a range of temperatures collapse on a single master curve which uniquely characterises the short term tensile behaviour. An example of such a master curve for a C(f)/SiC(m) composite is shown in Fig. 6 where the different colours correspond to tests at different temperatures.

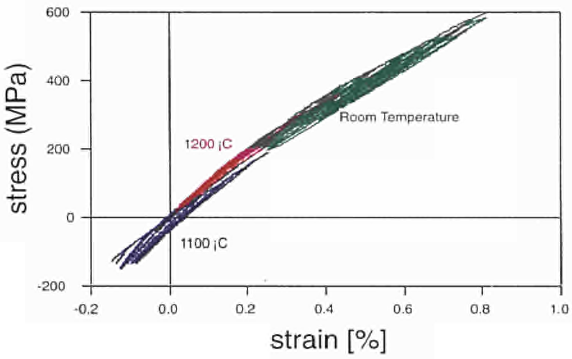


Fig. 6: Master curve for C/SiC composite

The results from this subproject have been disseminated in sixteen publications in open literature and as invited presentations at conferences. Two patents have been applied for. Competitive income was generated from third party work and from shared cost actions.

Modelling of the Mechanical Performance of Advanced Materials

This project is aimed at using the microstructural mechanisms identified as controlling the mechanical behaviour of bulk materials to model their macroscopic behaviour. It is generally accepted that to reproduce accurately the mechanical response of components, it is necessary to use a modelling approach which incorporates as much information about the damage mechanisms as possible. As a consequence of this, the modelling studies carried out are strongly linked to the experimental studies carried out within the sector on ceramic matrix composites and coated

single crystal nickel-base superalloys.
A criterion which has been developed in-house to predict the coalescence of precipitates within superalloys subjected to multiaxial loading conditions has been incorporated in a computer model. This has been successfully applied to the problem of reproducing the effect that the stresses induced by the casting have on the coalescence of the particles. The simulations have shown marked differences between the behaviour of the precipitates in different regions within a sample (Fig. 7), in line with the experimental evidence, enabling the verification of our calculations of the magnitude of the stresses within the material.

The development of a model for simulating creep in superalloys based on the approach of direct incorporation of microstructural features into the system has continued, allowing a more realistic representation of the material, and producing improved creep curves. The model is based solely on the movement of individual dislocations within the matrix of the alloy and their interaction with the strengthening precipitates

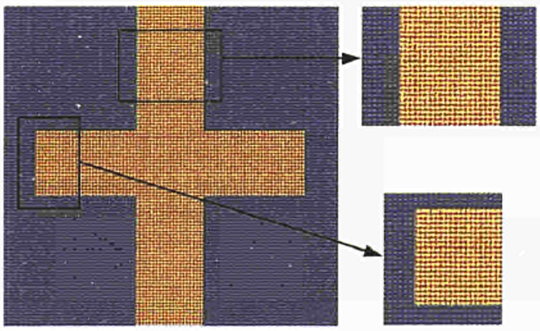


Fig. 7: Simulation of coalescence of precipitates showing details within the dendrites (yellow). 200MPa stress is applied along the longest dendrite direction in a superalloy tested at 850°C.

In the area of modelling of the mechanical behaviour of continuous fibre reinforced ceramic matrix composites the recently developed extended one-dimensional shear lag model has been adapted to bidirectionally reinforced composites. Calibration of the model parameters via the results of tensile tests at the level of the longitudinal bundles with their surrounding interbundle matrix, and at the level of the individual longitudinal fibres within the intrabundle matrix, allows to derive values of the interfacial friction coefficient at the two levels, as well as of the in-situ fibre and matrix strength and their Weibull modulus. Also, it is shown that the presence of transverse bundles gives rise to a radial tensile strength of the interface between the bundles and the interbundle matrix and between the fibres and the intrabundle matrix.

A simplified version of the shear lag model has been coupled to a chain-of-bundles model, which directly accounts for the probabilistic nature of the constituent properties of matrix and fibres. This model successfully predicts the complete stress-strain curve of unidirectionally reinforced CMCs.

The results of this subproject have been published in 5 contributions to journals and conference proceedings. Moreover, an International Workshop on Modelling of the Mechanical Behaviour of Continuous Fibre-reinforced Ceramic Matrix Composites, attended by renowned experts from Europe, Japan and USA, was successfully organised.

CORROSION PROTECTION

The principal objective of the project is to establish an in-depth understanding of the mechanisms and kinetics governing the high temperature corrosive degradation of advanced materials in complex environments and from this to derive ways by which resistance to attack can be improved. The interactive influence of solid ash deposits, molten salts or acidic condensates with aggressive gaseous atmospheres is an important aspect of these studies which, in addition, seek to establish the applicability of laboratory studies to industrial situations.
Several major thrusts during the year have focused on;

- increasing the severity of reducing-sulphidising gaseous environments by introducing Cl-containing ash deposits and down-time periods in aqueous condensates,
- studying the effectiveness of coatings on 12%Cr ferritic steels in improving resistance to attack,
- establishing the oxidation behaviour of Si3N4 and mullite- coated SiC,
- evaluating the performance of coated CMC's exposed to oxidising atmospheres,
- designing corrosion test guidelines, in collaboration with partners, for submission to CEN and other standards-making bodies,
- installing and commissioning a high-temperature thermal cycling oxidation/corrosion rig.

Continuing collaboration with EPRI (the Electric Power Research Institute in the USA) has enabled the corrosion behaviour of a ferritic 12%Cr steel (HCM 12) and a series of "model" alloys containing varying amounts of Si to be studied in the CO-rich dry-feed entrained slagging coal gasification atmosphere, shown in Table I. During 2000 hours exposure, it is evident that Si plays an important role in reducing sulphidation attack of the alloy substrate. The radical level of improvement is illustrated in Fig. 8 which combines the results of mass-gain measurements with cross-sectional examinations of corroded specimens after 2000 hours exposure. The relationship between metal-loss due to sulphidation attack and Si-level in the alloy is shown in Fig. 9.

	Composition (volume)				
gas	CO	CO2	H2S	H2	H2O
"dry" (A)	64	3.8	0.8	31.4	-
"wet" (B)	62	3.7	0.8	30.5	3

Table I: Composition of Test Gas Atmospheres

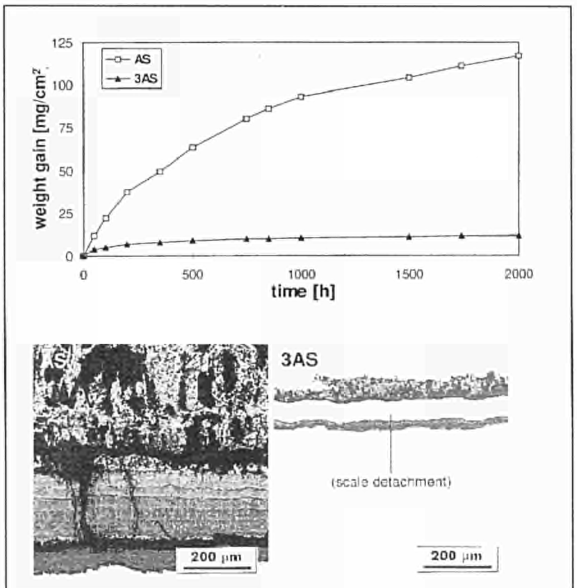


Fig. 8: Comparison of gravimetric data and cross-sectional appearance of the 12%Cr commercial and the 12%Cr-4%Si "model" alloy, exposed for 2000 to the "dry"gas

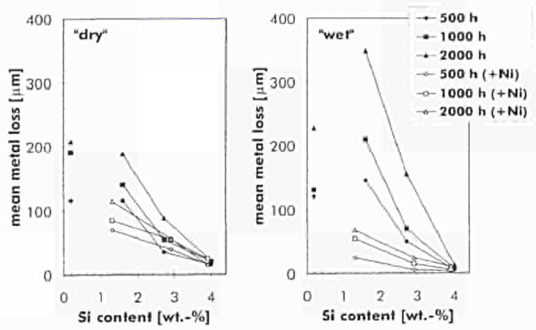


Fig. 9: Influence of Si content on the mean metal loss for the 12%Cr commercial and "mode" alloys and the 12%Cr-12%Ni "model" alloys in the "dry" and "wet" gas atmosphere

In industrial situations, however, coal conversion plants are also subjected to shutdown periods during which metallic components may cool to temperatures below the dew-point of the process gas. In such situations, the combination of aqueous/acidic condensates plus the presence of chloride-containing ashes deposited during the gasification/combustion of the coal, can lead to a much more severe form of attack, known as "down-time corrosion" (DTC). Studies have thus been carried out on selected Si-containing, 12%Cr alloys coated with an 80/10/10 blend of real ash, carbon and a 50-50 mixture of $FeCl_3$ and NaCl and exposed to the CO-rich gas for up to 1000 hours at 400°C with periods of 25 hours at 30°C every 200 hours. The effect of this on specimens exposed for 3, 200 hour DT cycles, is shown in Fig. 10. The multi-layered nature of the severe attack experienced by the sample subjected to moist DT is evident and seems clearly to be related to the number of cycles. The alternate light and darker-coloured layers vary in Fe, Cr and Si content, as well as in S and O.

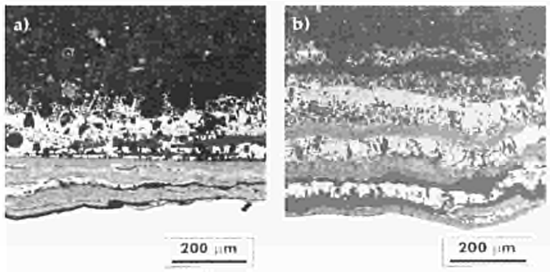


*Fig. 10: Cross-sectional examination of Fe-12%Cr-1.3%Si "model" alloy exposed 64%CO-31.4%H_2-3.8%CO_2-0.8%H_2S for 3X200 hours at 400°C in mixed ash deposit with **a)** normal dessication (dry DTC), and **b)** intermittent exposure in moist air at 30°C for 25 hours (moist DTC)*

Additional studies have been conducted to evaluate the corrosion behaviour of a range of alloys for heat exchanger applications at higher temperatures, in the region of 600°C, in this CO-rich sulphidising gas. The project started by monitoring the corrosion behaviour of high alloy steels, viz. HR 160, MA 956 and Sanicro 28, having compositions shown in Table II. Although these alloys showed good corrosion resistance, they are relatively expensive and hence there is a need to develop cheaper materials for these sorts of applications. The approach has been to upgrade alloys, originally designed to operate at lower temperatures, by alloy modification (as mentioned earlier), or by applying surface coatings to less-expensive alloys.

Elements	HR 160	MA 956	Sanicro 28	HCM12	Model Alloys					
					1AS	2AS	3AS	4AS	5AS	6AS
Fe	1.7	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.
Cr	28	20	27	12	12	12	12	12	12	12
Ni	36		31					12	12	12
Co	30									
Si	2.6	0.1	0.7	0.2	1.6	2.7	3.9	1.3	2.9	3.9
Ti	0.5	0.4			0.1	0.1	0.1	0.1	0.1	0.2
Cu			1							
Mo			3.5	1						

Table II: Nominal Chemical Composition of the Alloys Used in the Investigations

Two types of coatings are being studied, vacuum and air plasma sprayed, the main advantage of the latter being the ability to coat large components, plus the potential to affect repairs under field conditions. The air plasma sprayed overlay coating was applied to both the HCM 12 and Sanicro 28, using a powder containing 50Ni-49Cr-1Si (wt%). The vacuum plasma sprayed aluminide coating applied only to the Sanicro 28, was produced by spraying a powder with 89Al-11Si (wt%) followed by a diffusion heat treatment. After corrosion testing in the CO-rich gas at 600°C, the corrosion behaviour of the coated alloys was shown to be similar to the more expensive, highly alloyed materials (see Fig. 11). It should be pointed out that the mass gain of the uncoated HCM 12 alloy after only 100 hours of test was 28 mg.cm^2.

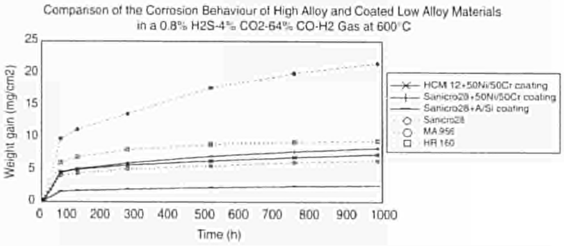


Fig. 11: Comparison of the behaviour of high alloy and coated low alloy materials in a 0.8%H_2S-4%CO_2-64%CO-H_2 at 600°C

The oxidation behaviour of monolithic Si_3N_4 is strongly influenced by the presence of sintering additives; while pure Si_3N_4 oxidises at a very low rate due to slow diffusion of oxygen through a surface layer of SiO_2, the participation of sintering additives in the surface reaction leads to an increase in the rate of diffusion which consequently produces a comparatively high rate of oxidation. In general, the higher the volume fraction of intergranular material, the higher the rate of oxidation.

Studies have been carried out on two batches of Si_3N_4 (with Y_2O_3 and Al_2O_3) of the same powder composition, but having received different sintering treatments. These have shown that the level of crystallinity of the intergranular material also influences oxidation behaviour (see Fig. 12). Oxidation at 1000°C in dry air in a thermobalance showed that the batch of Si_3N_4 with crystalline intergranular material oxidised faster than that containing an amorphous intergranular phase. At 1300°C, the trend was reversed.

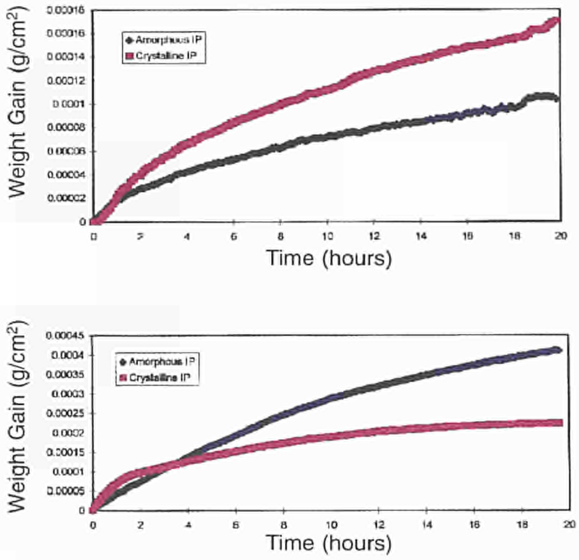


Fig. 12: Influence of level of crystallinity of the intergranular material on the oxidation behaviour

Detailed investigations to explain this behaviour are continuing, but it is apparent that at 1300°C, the crystalline state of the intergranular phase inhibits outward diffusion of Y and Al, thereby reducing the rate of oxidation compared to that of test pieces with the amorphous intergranular phase. At 1000°C, a crystalline surface SiO_2 is formed on Si_3N_4 having amorphous grain boundary phase, whereas an amorphous surface layer is formed on material with a crystalline grain boundary material. The crystalline surface layer is expected to, and indeed grows more slowly, but the reason for the difference in morphology has yet to be elucidated.

SiC fibre reinforced SiC is a candidate material for high temperature heat exchanger (HTHE) applications due to its enhanced fracture toughness compared with monolithic SiC. However, since the fibre-matrix interface provides an easy diffusion path for gaseous species (leading to a decrease in fracture toughness), corrosion resistant coatings are necessary.

One possibility studied has been an overlay coating of mullite applied by either atmospheric plasma spraying (APS) or high-velocity oxygen-flame spraying (HVOFS). 6mm SiC cubes, coated with mullite powders of different grain-size distributions using APS and HVOFS, have been thermally cycled in laboratory air at atmospheric pressure between 1300°C and room temperature. The results show that the best oxidation resistance was observed on the HVOF sprayed coatings, compared with those applied by APS which offered little protection (see Fig. 13 and Fig. 14). Further work is currently looking at the thermal stability of calcium hexa-aluminate ($CaO.6Al_2O_3$), since its thermal expansion coefficient is close to that of SiC and it also has a better resistance to environments having a high alkalinity.

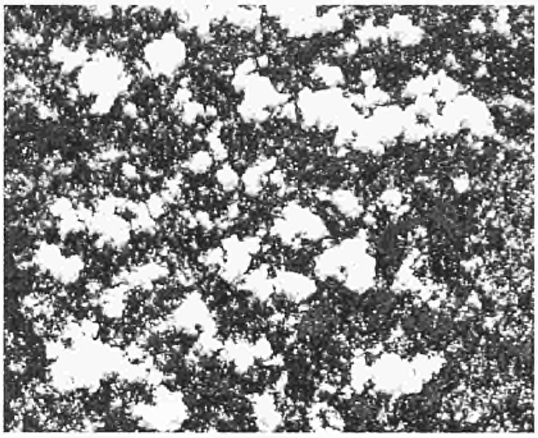


Fig. 13: APS mullite-SiC

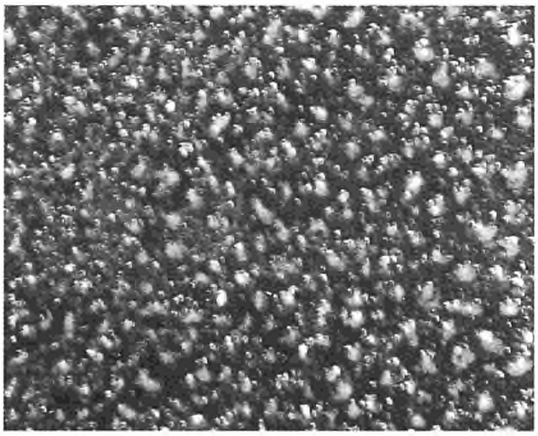


Fig. 14: HVOF mullite-SiC

In a complementary project, the oxidation behaviour of a Si_3N_4 matrix reinforced with Hi-Nicalon fibres pre-coated with a 0.4µm thick carbon interphase has been studied in dry air. At low temperature (T=700°C), only oxidation of the carbon interphase occurs resulting in a mass loss and creation of annular pores around the fibres. At higher temperatures, there is a competition between the oxidation of the interphase and the oxidation of both matrix and fibres with the formation of $Y_2Si_2O_7$ and SiO_2. Above 1300°C, oxide formation leads to the closure of the annular pores. However, the time needed to seal the gap between the fibre and the matrix is still too high to prevent the oxidation of the interphase to a significant depth. Consumption of the interphase can be avoided by the application of an external coating. A new coating protection (Patent applied for) has been investigated and has shown its potential efficiency for the protection of the carbon interphase.

Active collaboration and interaction with partners on a project funded by the "Standard Measurements and Testing Programme" (SMT) has resulted in the issue of a 1st Draft Code of Practice for "Discontinuous Corrosion Testing in High Temperature Gaseous Atmospheres. One of the studies being implemented within the Institute, in support of this, is starting to look at the importance of experimental variables such as gas flow rate and test specimen containment. The results of this will assist in revising the draft code of practice prior to its use as a guideline for a round-robin investigation involving many European test laboratories.

Within CEN Technical Committee 184, Working Group 3 (Monolithic Ceramics), attention has been focused on 2 documents with respect to environmental test methods. A final draft of a "General Practice for Undertaking Corrosion Tests" has been produced and sent to CEN for the voting stage of the standardisation process. This method is very general and attempts only to guide the tester in the approach to conducting a valid test and does not prescribe actual test conditions. In order to develop what is intended to be a more prescriptive method for the oxidation testing of non-oxide ceramics, a second SMT project has been initiated (in a similar manner to that described earlier), to carry out research to support and validate specific aspects of the test procedure. This project continues until the final draft of the test method is presented to CEN next year.

During 1996, a new thermal cycling rig, designed to work with oxidising and corrosive atmospheres at temperatures up to 1500°C has been commissioned . To date, the rig has been used to expose monolithic ceramic materials in an oxidising environment in order to study their resistance to spallation during cyclic conditions between 1300°C and room temperature.

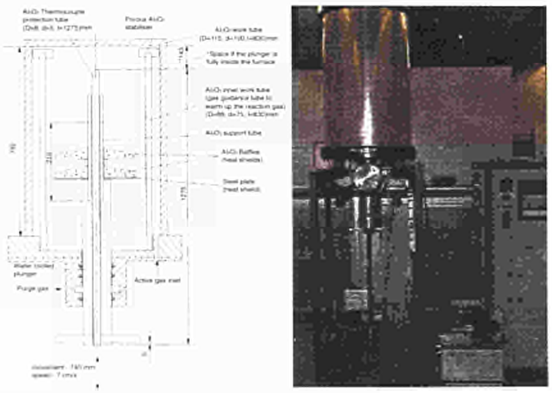


Fig. 15: Thermal cycling rig

A computerised software system has been developed and is now installed for user assessment. It provides the dual function of acting as a daily laboratory log-book during the planning and execution of corrosion experiments as well as an archive for all kinetic data and post-exposural microstructural observations. The Corrosion Test Manager (CTM) system provides the tools necessary to manage raw corrosion mass-change data from multi-specimen autoclaves and single-specimen thermobalances as well as the thermal cycling and burner rigs. Easy data manipulation and inter-test comparisons of the thousands of data points thereby facilitate a wider application of the data generated.

Highlights of achievements:

1. Improvements conferred by the addition of Si to 12%Cr steels have been quantified and the relationship between metal loss due to sulphidation and Si level established.

2. Selected alloys have been exposed to conditions more closely simulating those occurring in industrial situations where heat exchanger tubes become coated with ash and attack is exacerbated by acidic condensates which form during periods of plant shut-down.

3. The application of vacuum and air-plasma sprayed coatings has been shown to raise the resistance of these lower-alloy materials in reducing-sulphidising atmospheres to levels associated with more expensive higher-alloy materials; longer-term testing is, of course, still needed.

4. In Si_3N_4 sintered with Y_2O_3 and Al_2O_3, a higher level of crystallinity in the intergranular phase has been shown to reduce oxidation resistance at 1000°C whilst at 1300°C, the reverse is true.

5. Pre-normative collaborative studies have enabled a Draft Code of Practice for "Discontinuous Corrosion Testing in High Temperature Gaseous Atmospheres" to be prepared and also a Final Draft of a "General practice for Undertaking Corrosion Tests" has been submitted to CEN.

6. A new high temperature thermal cycling rig has been commissioned and used to determine the influence of thermal cycling upon the oxidation behaviour of hot-pressed Si_3N_4.

CORROSION-DEFORMATION INTERACTIONS

Investigations have been concentrated on the assessment of the mechanical integrity of oxide scales under creep deformation for the alloy MA 956 (20 Cr, 4.5 Al, 0.5 Y_2O_3, bal. Fe in mass %) and Ti48Al2Mn2Nb (numbers denote the composition in at. %). MA 956, an ODS-type Al_2O_3 former, is of high technical interest for application in oxidizing atmospheres up to 1200°C. Moreover, the high corrosion resistance in sulphidizing/oxidizing atmospheres of MA 956 has to be mentioned. TiAl-based alloys are considered as future alloys to be used for instance for aircraft turbines because of their high stress/density ratio. Since serious limitations can result from insufficient oxidation resistance, a better knowledge of the oxidation behaviour, in particular under service relevant conditions, is of high interest in order to characterize the technical potentials of these alloys.

The investigations on MA 956 have been conducted at 1100°C for up to about 3.000 h. Difficulties for the testing procedure arose from the fact that 2 mm thin sheet samples had to be used according to collaborative studies and modification of the testing equipment became necessary. Moreover the material exhibited a very low creep ductility of only 1.5% and a high sensitivity of creep rate on stress, both of which rendered testing more difficult. Microstructural investigations on MA 956 revealed the formation of a nearly pure Al_2O_3 scale which exhibited excellent adherence under stress application. Cracking and some limited spallation occurred only for ruptured samples. However local spallation of the scale occurred when a rough surface treatment was applied although restoration of the Al_2O_3 scale became possible. The scale exhibited a compact morphology but a small number of pores were found in the scale interior and at the scale-alloy interface. The number and size of these defects increased with the scale thickness. According to Fig. 16. In Fig. 17, it is demonstrated that interfacial pores acted as crack sources during mechanical exposure.

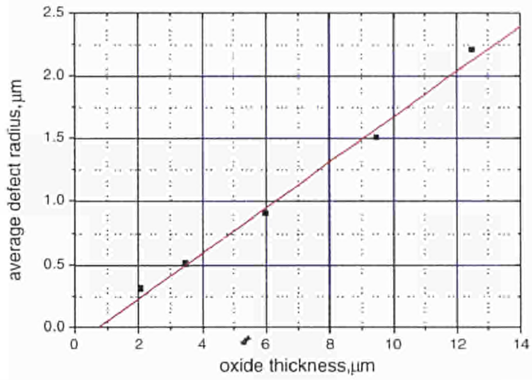


Fig. 16: Defect size versus scale thickness for MA 956 at 1100 °C

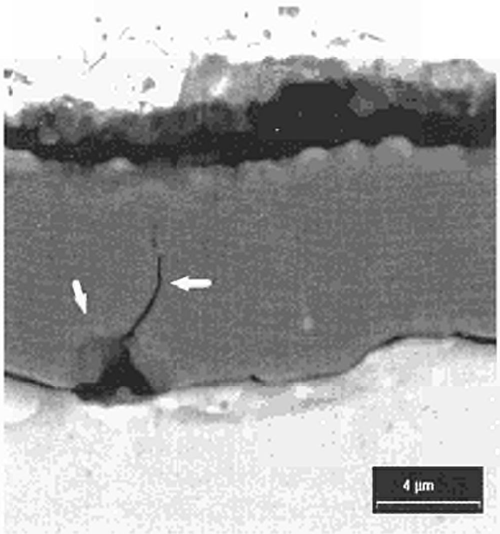


Fig. 17: Crack initiation and propagation in the Al_2O_3 scale on MA 956.1100 °C/1100 h

As a technical conclusion it emerged that:

- superimposing a stress on the isothermal exposure of MA 956 at 1100°C up to 3.000 hours does not deteriorate the effectiveness of scale protection
- the influence of a rough surface treatment has to be realized

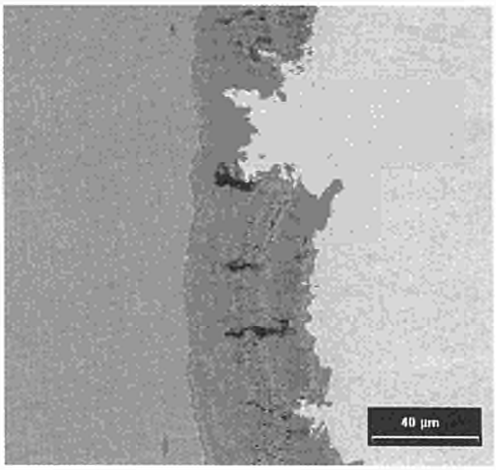


Fig. 18: Scale cracking in Ti48Al2Mn2Nb after creep at 8285 h at 800°C

Investigations on Ti48Al2Mn2Nb have shown a surprisingly good oxidation resistance of this alloy which was probably due to the Nb content. Moreover oxidation under stress did not cause serious problems. At 700°C, even after 13.000 h exposure, which lead to an internal oxide of about 7 mm, full scale adherence was observed. Oxide cracking happened close to the sample fracture surface and at a few other locations where high strains were acting. Fig. 18 documents the good scale adherence under stress. Testing at 800°C revealed deterioration of the scale behaviour after about 300 h exposure. Local spallation occurred but oxide remnants which remained attached to the sample surface, acted in a protective way. An example of this feature is given in Fig. 19.

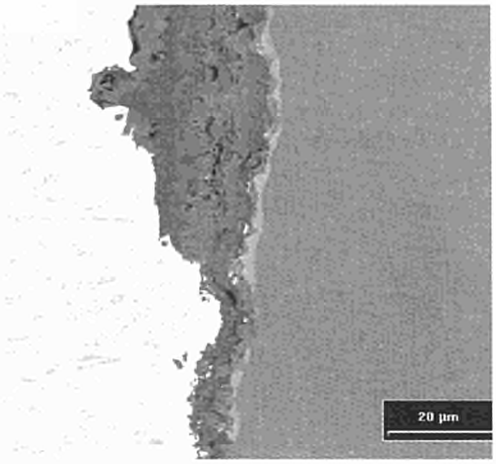


Fig. 19: Partial spallation in Ti48Al2Mn2Nb after 8285 h at 800°C

The technical outcome of the investigations on the TiAl alloy are:

- an understanding of the oxidation behaviour for long term exposure,
- creep does not promote scale spallation,
- strains of about 2-3% could be subjected without scale cracking

COMPONENT INTEGRITY TESTING AND EVALUATION

During the year, the output from this institutional project has had to be restricted to the research on advanced steels for conventional power generation, awaiting the recruitment of new staff and students for the work on materials for very high temperature heat exchangers and for thermal fatigue/creep interactions in stainless steel components. All the contributions in the area of power plant steels is unavoidably linked to some contracts for Third Parties and Shared Cost Actions which are running in parallel. The main actions have been related to developing, firstly, special test methods for studying the integrity of welded components in preparation for test methodology standardisation and, secondly, analytical tools and models for enabling improved assessment of design and remanent lives in plant components.

Creep and creep crack growth test methods for either tubular components or specimens obtained from welded components are as yet not standardised, wherea3 the data emanating from such tests could urgently be used by both the designer and operator of power plant if it could be considered as derived from a valid test method. Four different test methods are under study, two for creep and two for creep crack growth. It is shown on Fig. 20

how the different test-pieces are extracted from a plant steam pipe. The CT specimen is oriented in such a way that the notch is located in the heat affected zone (HAZ) of the weld in order that the most prevalent type of crack growth observed in plant, Type IV cracking, can be realised. Similarly, the notch in the notched tube is located so that part of the crack growth will take place in this vulnerable HAZ. Both the tubular and uniaxial cross weld creep test-pieces contain the weld/parent metal interface at its original as welded angle.

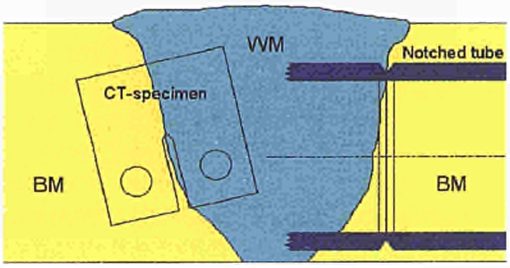


Fig. 20: Novel test-pieces for testing creep crack growth of welds in steam pipes

The results from the CT and tubular component crack growth tests are under evaluation in the frame of the SCA and TPW respectively, although a major improvement in the CT test method itself has been realised with the introduction of a new lightweight design for the extensometry which has increased sensitivity and repeatability over older designs employed by the partner organisations. For the creep tests on welded test-pieces an interesting observation has been made concerning the location of the failure in that failure in the base metal, close to the so called intercritical zone (Fig. 20), is always observed for the cross weld tensile specimens loaded uniaxially whereas the internally pressurised tubes always fail in the weldment. The weldment should be the weaker of the two materials as shown from the creep results on base only and weld only specimens and an explanation is sought which will include an assessment of geometry, stress multiaxiality and stress level. For the tubular components, the influence of stress multiaxiality will be further elucidated through tests under combined tensile and internal pressure loading which are specifically designed to represent the conditions in real plant components which may lead to Type IV cracking.

In the activities centred on modelling for lifetime prediction, most of the effort was expended towards uniaxial and multiaxial creep deformation modelling for base materials of CMV, $2^1/_4$Cr1Mo, and P91 (9Cr1Mo) steels under the frame of the TPW contract. Both Theta Projection and Continuum Damage Mechanics models have been successfully applied to predict longer term creep behaviour under multiaxial loading from short term uniaxial tests. The spin-off for the institutional project has been the preparation of the various computer codes ready for application to the welded components. As an example of the success of the methodology developed, the application of the CDM model to uniaxial creep curves obtained from the weldment only is shown in Fig. 21. The three higher stress curves were first fitted to the model and, from the parameters evaluated, predictions could be made for these curves for interpolation but also for extrapolation to a longer test carried out at a lower stress of 130 Mpa. As can be seen a very accurate prediction indeed was made when compared with the actual test result. When sufficient data is available for parent metal and for the HAZ (obtained using heat treatment simulation) the models will be combined to enable predictions to be made for the uniaxial cross weld and multiaxial welded tubular component tests.

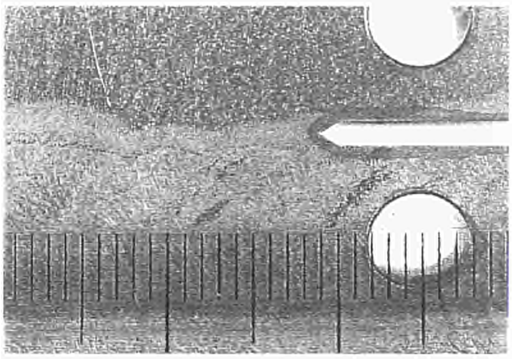


Fig. 21: Close-up of the notch location in the heat affected zone of a weldment

NEUTRON SCATTERING

Introduction

Residual stresses can be introduced into components and materials by a variety of forging, welding, heat treatment and surface treatment processes. These stresses can affect strength and performance. Neutron diffraction has the potential to measure these stresses non-destructively. Neutron diffraction is superior to x-ray diffraction because it allows for internal strain measurements at material interfaces and well below the specimen free surface.

Objective

To develop and validate advanced experimental techniques based on neutron diffraction for the investigation of residual stresses in monolithic and composite structural components of advanced energy production and propulsion systems and in composite materials under development.
To develop and validate state of the art computational methods for the prediction of internal stresses in

a) monolithic structural components, including welds, subjected to severe industrial environments and loading combinations in order to facilitate the assessment of their structural integrity
b) advanced materials under development, such as fibre reinforced CMCs and thermal barrier coatings.

Methodology

Development of state of the art stress diffractometers based on the HB4&5 /HFR facilities and the installation of a new position sensitive 32-wire neutron multi-detector, new adequate shielding of the diffracted neutron beam, large X-Y Table and small crane for the support of large structural components, a stress rig for elastic constants investigations, and efficient motor control and data acquisition system based on LABVIEW.

Development of state of the art numerical models based on advanced FEM techniques with moving boundary and automatic re-meshing options for the prediction of internal stresses due to welding and coating deposition.

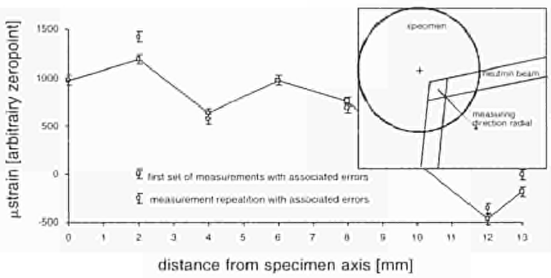
Targeted industries

Vendors of process and power plant structural components; operators of process and power plants; developers of composite materials including coatings and joints.

Results

On HB4 a new position sensitive 32-wire-detector was installed. Several tests were run showing the performance of the new detector. It proved to cut down measurement times by a factor of 10 to 30. Additionally the setup of the secondary beam path was upgraded in accordance with the requirements of the new system. Successful tests were performed on the more flexible use of the double monochromator using iron powder and a silicon nitride based CMC.

A Si/SiC Composite was investigated on uniformity of strain distribution and on the amount of silicon still present in the material. A three directional stress measurement campaign was performed on two specimens of a low alloyed steel cast truck motor part. Strains were measured in two orthonormal directions in a shock quenched austenitic steel cylinder. Two Si_3N_4/C CMC were investigated in three orthonormal directions. This campaign included extensive powder studies for the determination of the unstrained lattice parameters.



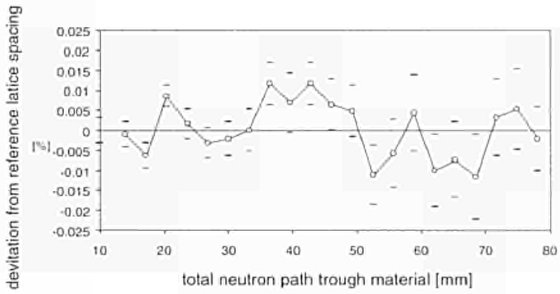


Fig. 22: Strains in radial direction in an austenitic steel cylinder

Fig. 23: Diffraction performance on thick specimens.

Deliverables

Reference measurement method for the calibration and performance demonstration of field residual stress measurement techniques. Full, through thickness, residual stress tensor mapping in structural components of energy, petrochemical, heavy machinery and transportation systems for a credible assessment of their structural integrity. Evaluation of the performance of residual stress relief techniques such as PWHT. Texture studies in high performance steel and aluminium alloys and welds. Mapping of residual stresses across dissimilar material interfaces as in joints and coatings for the assessment of manufacturing processes. Phase distribution and average values of residual stresses in multi-phase materials, such as ceramic matrix composites, for their efficient design.

Industrial relevance

Reliable estimates for internal stresses in structural components are indispensable for both their efficient design and remaining life assessment.

Reliable estimates of residual stresses across dissimilar material interfaces are indispensable for the development of advanced composite materials and their implementation in industrial systems.

Achievements – spin off

- Significant performance enhancement based on a new neutron multi-detector at HB4/HFR
- Feasibility of testing thick piping steel welds – Measurements performed at CRNL (CAN) and HFR.
- Standards for residual stress measurement – VAMAS TWA 20 Project started in January 1996 and is coordinated by Imperial College (UK).
- Calibration of volume residual stress measurements – INTAS Project started in January 1996 and is coordinated by Fraunhofer Institute (DE).
- "BIMET": Structural Integrity of Bi-Metallic Components (NFS) project starting in February 1997 – coordinated by EdF (FR).
- "VORSAC": Variation of residual stresses in aged components (NFS) project started in January 1997 – coordinated by TWI (UK).
- "C/SiC-Tubes for UHTHE": (BRITE-EURAM) project started in February 1997 – coordinated by SCHUNK (DE).
- "RESTAND": Residual stress standard using neutron diffraction (SMT) project expected to start in September 1997 – coordinated by JRC.

HTM-DB AND COR-DB

The 1995 activities in upgrading and commercialisation of the HTM-DB and COR-DB continued and a new activity was created: electronic data sheets.

1. Network Activities of the High Temperature Data Bank (databank for mechanical & physical properties of alloys)
   - data management and data evaluation within European Materials Programs
   - data exchange with European Research Centres
   - commercialisation of the overall databank system
     - database structure
     - released data content
     - User-Interface
     - evaluation programs
   - adaptation to individual needs of the customers

2. Development of a Corrosion Data Bank with a direct link to data evaluation routines (cooperation with the German 'Forschungszentrum Jülich', KFA)

3. Implementation of electronic data sheets (Werkstoffblätter) together with computerised inter- & extrapolation routines for KWU/Siemens

HTM-DB

The High Temperature Materials Databank (HTM-DB) *User-Interface* which is running on PC's under ***Microsoft Windows*** and the uniaxial creep evaluation programs were finished in 1995. It is now available as client/server and stand-alone PC versions. The figure shows the Replication stand-alone PC version of the HTM-DB. Data that are entered by the users into the shadow database of their PC have to be transferred into the databank of Petten were they are checked for their data quality. Then they are loaded into the production database of the Petten server and redistributed to the users. That has the advantage that the data flow within European projects can be organised centrally and all participants get back as well as their own data and the data of the whole project.
The HTM-DB participated in a European FISA project for reactor safety. Materials data for core melting studies of PWR's reactor vessels were entered into the databank and redistributed together with the User-Interface to the European partners.

HTM-DB applications together with uniaxial creep evaluation programs are purchased by several institutions such as MPA Stuttgart, Swedish Institute of Metals, ENEL, ABB, KEMA and MTU. The response of the users was very positive. A lot of other companies such as GEC Alsthom, FRAMATOME, EDF, CEA Saclay have already shown their interest.

COR-DB

The Corrosion Databank (COR-DB) and the corresponding *User-Interface* which is running on PC's under ***Microsoft Windows*** were finished in 1995 and linked to the data acquisition system of our institute's corrosion group. We have started to commercialise this databank, too. ENEL, ABB and the Swedish Institute of Metals got it for a Beta test period.

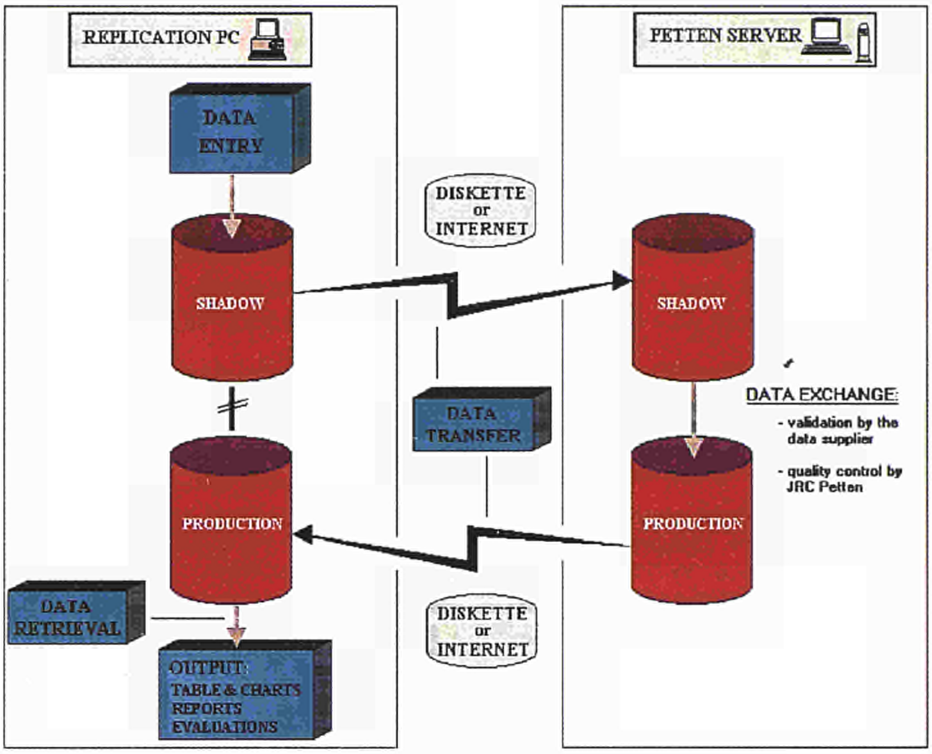


Fig. 24: HTM-DB Replication stand-alone PC version

BRITTLE MATERIALS

Intermetallics and refractory metals and alloys often have unique properties regarding high temperature and corrosion resistance. However, safety issues may arise using them as structural materials due to the transition from a brittle low temperature range to a ductile high temperature range. This phenomenon is well-known for many b.c.c. metals and has been intensively investigated in the past without reaching a complete understanding of the microstructural processes that are involved. It is the aim of this project to improve the knowledge about the ductile to brittle transition (DBT) and its relevance to safety and quality issues. With this the project provides basic contributions to other running projects at the institute (Fusion Materials, AMES, Surface Performance Standards).

Characterization of Chromium and Chromium Alloys

The properties of high purity chromium (DUCROPUR, 99,7% Cr) and chromium alloys (Cr 44Fe 5Al 0.3Ti 0.5Y_2O_3 and Cr 5Fe 1Y_2O_3 and) were investigated by means of tensile and fracture mechanical tests, optical and scanning electron microscopy with X-ray analysis. Emphasis was laid on the determination of the fracture toughness and the ductile to brittle transition of pure chromium which was produced by a sintering process (Metallwerke Plansee). Special difficulties in precracking were encountered in the brittle range below 100°C. In Fig. 26, details of the fracture surface of a Compact Tension specimen broken at 300°C in the ductile to brittle transition range is shown. Cleavage fracture is preceded by ductile crack growth in the centre of the specimen. The temperature dependent fracture toughness is plotted in Fig. 25. Although no valid KIc-values could be obtained so far, the curve indicates that the DBT temperature is in the range of about 300°C. Tensile tests in this temperature range show a predominantly ductile behaviour with total elongations up to 25%.

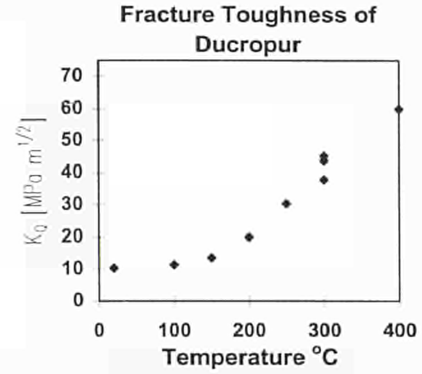


Fig. 25: Fracture toughness values KQ as a function of temperature. The values at low temperature are upper bounds estimated on the base of non valid tests.

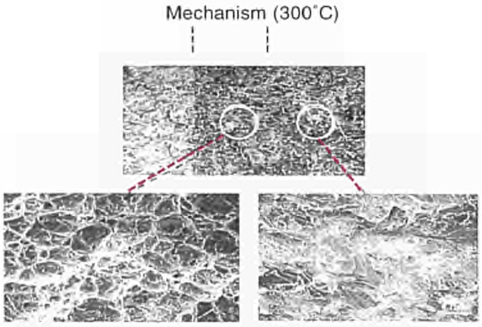


Fig. 26: Details of the fracture surface of a CT-specimen broken at 300°C Cleavage fracture (below right) is preceded by ductile crack propagation showing the typical dimple structure (below left). No second phase particles were found at the bottom of the dimples.

Mechanisms of Irradiation Embrittlement

An extensive review on theoretical concepts for describing the DBT transition in materials exposed to irradiation has been performed in collaboration with AEA-Technology under the umbrella of the AMES network. A problem which encounters the description of the DBT in commercial steels is the appropriate modelling of the rather complicated microstructure. The shows that progress in the description of phenomena like irradiation embrittlement and ageing on the DBT can only be achieved, if the modelling is formulated on the dimensional scale which is concerned by the damaging processes. A satisfactory, simple macroscopic model which describes the influence of easily measurable macroscopic properties (e.g. the flow stress) on the DBT is not yet available. Also the statistical models widely used by engineers lead finally to the conclusion that intrinsic mechanisms in single grains are controlling the DBT. This underlines the importance to include a mesoscopic approach based on dislocation dynamics to explain the DBT. Since the purely statistical evaluation also points to a DBT phenomenon, the combination of both approaches could lead to a better understanding of the complexity of the experimental observations.

ADVANCES IN CERAMIC JOINING

The activities of the project in the reporting period were focused on:

1 the development of techniques for fabricating strong, refractory ceramic joints by:
   a) applying and further evaluating the earlier reported hybrid brazing-diffusion novel bonding route,
   b) assessing a recently developed high temperature filler metal for brazing ceramics

2 the characterisation of ceramic joints and specifically on establishing the methodologies for measuring:
   a) interface fracture toughness
   b) residual stresses and stress distribution,

to enable an understanding of correlation between interfacial physicochemical properties, mechanical response and joint fabrication procedures.

A number of interlayer/multilayer systems, critically selected according to established criteria to satisfy the principles of the novel bonding route, were investigated as joining agents for Si_3N_4. These targeted both, a low temperature of bonding and/or a high service temperature for the joint. Such systems were, AgTi/Bi, AgTi/Sn and CuTI/Pd, respectively. Most promising systems were valued to be: a) the AgTi/Bi multilayer which allows bonding of Si_3N_4 to occur at 450 °C and b) the CuTi/Pd interlayer that can yield usefully strong joints (σf » 200 MPa), which can retain their strength up to 800°C in air.

Throughout these evaluations the effects of the liquid former and active component in the multilayer system and the amount of liquid formed during bonding have been clarified.

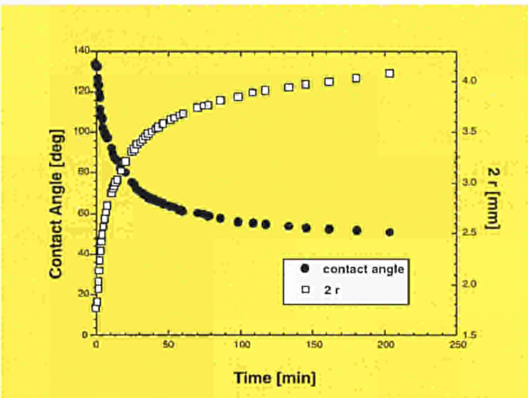


Fig. 27: Variation of contact angle with time and spreading rate of a sessile drop of Au-37.7Ni-4.8V alloy on Si3N4 (1050°C, 10-6 mbar)

The suitability of new filler metal alloy based in the Au-Ni-V system has been evaluated extensively for brazing Si_3N_4 and preliminarily for ZrO_2 and Al_2O_3. The wetting and spreading characteristics of the alloy were determined (Fig. 27) and although they do not match those of the Ag-Cu-Ti based filler metals, they are deemed good. The strength results (Fig. 28) of joints fabricated under optimized conditions demonstrate that brazing with the V-active filler metal permits strong joints to be fabricated. However, what is of more interest is that the high temperature testing of the joints (Fig. 28) has shown that this system yields a substantial improvement on the HT capabilities of brazed joints and in particular of the Ag-Cu-Ti brazed Si_3N_4 joints. Similarly and equally positive is the fact that annealing of the joints for 100 hr at 800°C in air did not deteriorate their as-bonded strength characteristics.

Brazing of Zirconia ceramics to Ti, Ti6Al4V alloy with a view to biomedical applications was also studied. Promising results were achieved utilizing Au, Ag interlayers and mimicking an in-situ brazing process.
A large number of interface fracture test results for the diffusion bonded $Si_3N_4/Fe/Si_3N_4$ and $Si_3N_4/FeCr/Si_3N_4$ joints were established. Experimental procedures have been optimized, particularly with respect to interface pre-cracking. For both systems the interface fracture toughness increases with increasing mixed mode (opening and shear) of loading and with increasing the Fe alloy interlayer thickness. The yielding conditions of the constrained metal interlayer were determined and development of pertinent fracture criteria is ongoing.

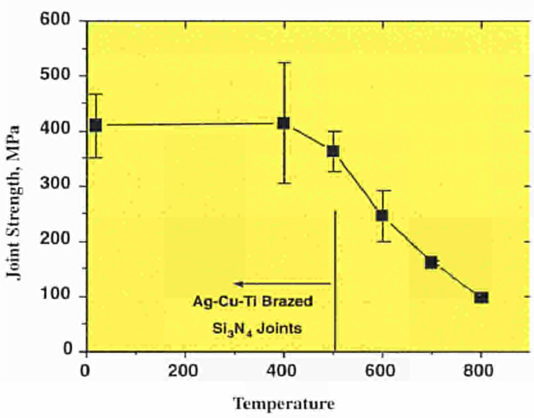


Fig. 28: Flexural strength of Au-Ni-V brazed Si3N4 joints as a function of test temperature. The limited service temperatures of Ag-Cu-Ti brazed joints are schematically shown.

The effects of joint geometry on the residual stresses have been evaluated for the Si_3N_4/Kovar® and Si_3N_4/AISI 316 joints. The material removal correction procedure that is been developed, as been reported previously, for metal/ceramic joints was fully verified. Establishment of procedures to enhance the spatial resolution of the XRD measurement technique are currently under progress.

Results achieved from the execution of the project studies in 1995 were diffused in the open literature by means of 10 publications in referred Journals, 5 in Conference Proceedings and 9 presentations in international Conferences.

SURFACE PERFORMANCE

The objective of this project is to study surface degradation processes due to thermal cycling and wear by a combination of various advanced techniques, including Thin Layer Activation (TLA). It also studies the possible improvement of material performance by surface modification. The advanced, and partially even unique, facilities are made accessible to several organisations in Europe through collaborations. In the year under review, a Research Agreement was signed with the IAEA in the frame of modification of materials by ion treatments for industrial applications.

A significant problem in the progress of understanding cyclic corrosion behaviour of materials is the large difference in testing conditions as employed by the various investigators. This makes comparison of data from laboratory to laboratory almost meaningless. The Institute is active in this field by performing pre-normative research to analyse the possible critical parameters in cyclic corrosion testing. This is a contribution to the formulation of test guidelines or test standards.

The use of the IAM high current ion implanter as a tool to study the high temperature scaling and spallation behaviour of advanced materials was further exploited. This included TiAl-based intermetallics in the form of thin sheet, in collaboration with Plansee AG, Austria. A participation within the Concerted European Action on Structural Intermetallics ("CEASI") falls also in this frame. On the basis of previous experiments the studies were targeted especially on TiAl-based alloys with small amounts of niobium. Ion implantation offers the advantage of adding an element in a surface layer in a well controlled manner. In various studies it was shown that ion implantation can be beneficial to obtain a better understanding of the underlying degradation mechanisms. Additionally, it can also serve as a screening technique for alloy development.

In the field of lubricated wear testing of technical components, TLA has significant technological and economic benefits. The antipathy towards radioactivity, however hinders a significant expansion of the market. The Institute decided to offer a complete packaged service to industry, instead of the specific technique per se. The construction of the IAM Facility for Engine Testing for this purpose was delayed due to complex safety and infrastructural procedures.

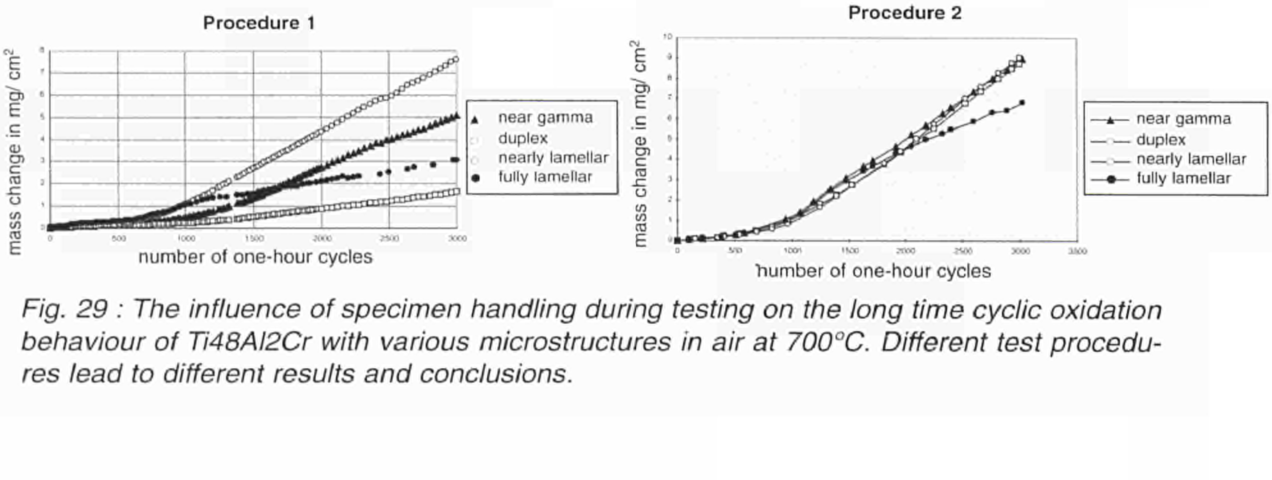


Fig. 29 : The influence of specimen handling during testing on the long time cyclic oxidation behaviour of Ti48Al2Cr with various microstructures in air at 700°C. Different test procedures lead to different results and conclusions.

INDUSTRIAL TECHNOLOGY SURFACE ENGINEERING FOR IMPROVED PROPERTIES

PACVD

The activities in the Plasma Assisted Chemical Vapour Deposition (PACVD) project have been directed to the understanding and development of process optimization, with a particular emphasis on in-situ diagnostics for process control and novel coatings for industrial applications.

In the field of process development, a new Dual Frequency Inductively Coupled Plasma Source for coating complex tridimensional parts has been developed and characterized in the frame of the EUREKA project IPACERC, led by JRC. This apparatus features a high-density inductive plasma source, plus an optimized direct heating of the substrate by means of a supplementary inductive set-up. This concept relies on the well-established performance of both inductive plasma generation, and direct inductive heating of the substrate. The novelty resides in combining both features in a single, optimized piece of equipment where two inductive set-ups operating at different frequencies are superimposed for simultaneous operation.

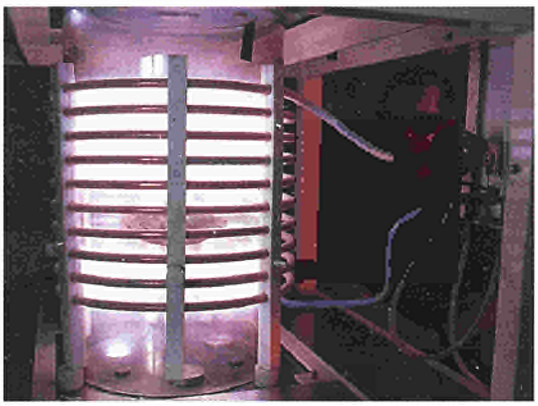


Fig. 30: Inductively Coupled Reactor with an Ar plasma

The first phase of this project has been to built the reactor and address the first technological challenge, i.e. to effectively prevent both power supplies from potentially destructive interference. This has been solved by designing appropriate power filters that allow reliable operation of the reactor equipment in nominal conditions (maximum power from both generators). It has required the determination of an equivalent circuit of the two coupled inductors in order to simulate the electrical interactions between both systems. Then, the power filters have been increased in size in order to minimize these electrical interactions. The electrical superposition of the two systems has been performed, simultaneously providing the plasma creation and the heating of the substrate.

The second phase of the project has consisted of assessing the performance of the reactor with respect to the plasma generation and the heating of the substrate. Ion density measurements have been performed to characterize the plasma quality. In nominal conditions, the measured ion density is greater than 10^{12} cm^{-3}, i.e. two orders of magnitude larger than state of the art PACVD sources (Fig. 30). To optimize the plasma source frequency, temperature measurements on a metallic substrate with thermocouples and IR emission were performed with different frequencies and different powers. The optimal frequency for heating was found to be 100 kHz providing a temperature gradient across the heated part less than 10 °C which is a good value for the deposition uniformity and coating microstructure. These experimental results are very promising for the PACVD reactor performance, and the process development for deposition of

W/WC multilayers will be started during 1997. The project led to a patent application and two publications.

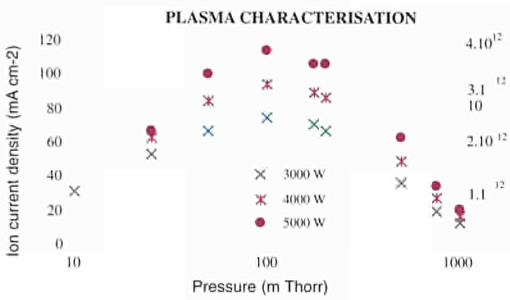


Fig. 31: Ion current density and ion density of the Inductively Coupled Plasma reactor for an Ar plasma as a function of pressure and Rf power.

In the field of coatings for hard surfacing for tribological use, the activity has focussed on the influence of process parameters on the deposition of Boron Nitride. The attempts to deposit cubic boron nitride from PVD or PACVD have all demonstrated the role of ion bombardment on the formation of the cubic phase. However, the films obtained have high residual stresses which limit their applicability. In the present work, we have tried to evaluate the possibilities offered by competing deposition/etching mechanisms: Plasma Post-treatments of boron nitride coatings have been performed using pure argon, Ar/H_2 and Ar/Cl_2 mixtures. The study revealed the effects of ion bombardment, H/H_2 atoms, and Cl/Cl_2 on the cubic-BN content in the films. It has been found that, in addition to ion bombardment, hydrogen atoms and more efficiently Cl and/or Cl_2 can be considered as chemical etchants of sp^2 bonded boron nitride. For instance, the concentration of cubic BN increased from 5% before treatment to 20%, 40% and 55% for Ar, Ar/H_2 and $ArCl_2$ respectively, opening new possibilities route for c-BN deposition of films with low residual stress.

SURFACE MODIFICATION FOR CORROSION PROTECTION

The technique of chemical depth profile analysis by Radio Frequency Powered Glow Discharge Optical Emission Spectroscopy (GD-OES) has been investigated for application in the analysis of ion implanted surfaces. The results show a good qualitative agreement (Fig. 32) between theoretical (TRIM) depth profiles and experimentally determined data obtained using GD-OES and Rutherford Backscattering (RBS). The capability of the technique to analyse non conducting layers has been used in the examination of corrosion scales on TiAl intermetallics and nickel base superalloys after high temperature oxidation. Preliminary results show the method to be applicable to fundamental corrosion studies where knowledge of scale and scale/metal interface composition may be used in the elaboration of corrosion mechanisms.

Calibration of the instrument using certified standards has allowed the quantitative analysis of thick (80mm) MCrAlY coatings after high temperature exposure with the results being applied to the quantification of aluminium depletion by the combined effects of oxidation and base alloy interdiffusion during long term exposure at temperatures of 800-950°C. The nature of this interdiffusion is dominated by the movement of aluminium into the base alloy where precipitation of Ni_3Al occurs resulting in the formation of a zone which is simultaneously enriched in aluminium and depleted of chromium.

During the working lifetime of certain turbine components it may be necessary that protective coatings be removed and replaced several times before the effective mechanical lifetime of the component is reached. In previous studies of this process a laboratory scale method of electrochemically (galvanostatic) stripping exhausted MCrAlY coatings was developed. In a continuation of this work, tests have been undertaken to assess the corrosion resistance and integrity of superalloys following stripping of the coating to the original alloy interface and recoating using

VPS. Corrosion tests have been performed of recoated UD520 and IN738 alloys exposed to chemical and thermal conditions characteristic of gas turbine operation. It was found that, although external corrosion behaviour was indistinguishable from that of the normal MCrAlY, certain samples exposed at 800°C exhibited damage by internal corrosion attack (Fig. 31) of the coating/alloy interface. The mechanism of this phenomenon is thought to involve initial attack of the interface, across localised coating defects followed by propagation along the interface which has been rendered vulnerable to internal oxidation as consequence of the higher than normal Al/Cr ratio. This has been demonstrated by GD-OES compositional depth profiling.

The use of the Chemical Vapour Deposition (CVD) technique has previously been investigated as a promising method of closing exterior porosity in ceramic coatings. It has the possibility to reduce the oxidation rate of the metallic bond coat of yttria stabilised zirconia thermal barrier coatings (TBC). Further detailed studies have concentrated on the testing of SiO_2 deposited from tetraethyl-orthosilane (TEOS). These show that the application of ~4µm of SiO_2 to the surface of the YSZ ceramic could reduce the apparent open porosity of the ceramic by 15%. Such samples when exposed to high temperature(1000°C) corrosion testing over 1000 hours in air showed 35% reduction in bond coating while identical testing in more aggressive atmosphere of $Ar/10\%O_2/0.3\%SO_2$ did not show any comparable improvement.

Some parts of the above described work were partially, or completely, conducted within the framework of a research contract between JRC-IAM and ENEL.

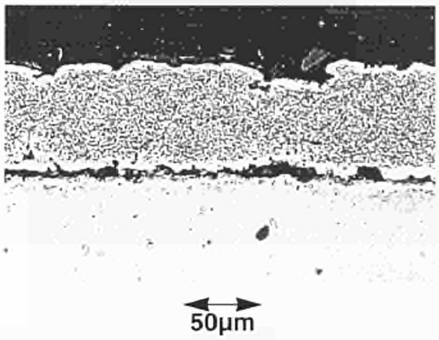


Fig. 32: Internal Corrosion Attack of Stripped/Recoated UD520/MCrAlY after 500h at 800°C

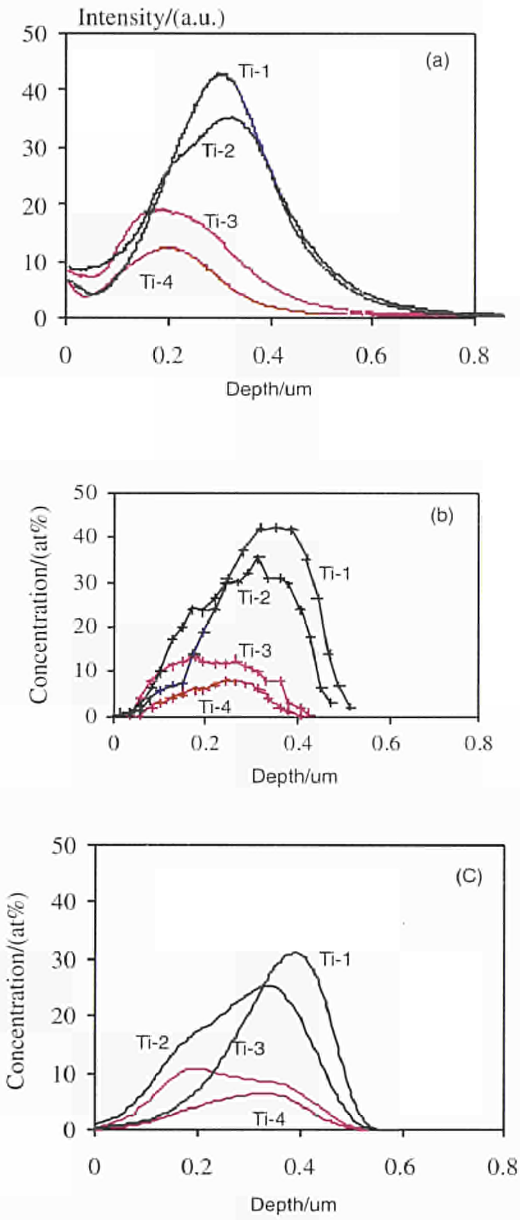


Fig. 33: Depth Profile Analysis of Carbon Implanted Titanium (a) GD-OES, (b) RBS, (c) Theoretical Profile (TRIM)

SURFACE MODIFICATION FOR FATIGUE RESISTANCE

The treatment of metallic surfaces by laser beams yields high heating rates in a thin surface layer followed by fast cooling due to self quenching. With this treatment microstructural changes are introduced such as phase transitions to metastable phases or grain refinement due to fast solidification after surface melting. These changes can be beneficial for reducing lifetime limiting surface damage and are now established methods for surface hardening or for improving the corrosion resistance. As fatigue properties are greatly influenced by surface conditions, the improvement of the fatigue resistance by laser treatments is investigated in this project.

Laser Surface Treatment

Surface modification of Al-alloys (7075) by laser surface remelting has been investigated using a Nd:YAG laser. The laser process parameters for remelting a thin surface layer without damaging the protecting oxide layer have been determined experimentally. Microstructural investigation and fatigue testing have been initiated.

Fatigue Damage Monitoring by X-ray Diffraction

The method consists of measuring the peak breadth (ß) of a suitable X-ray diffraction line at critical positions on the surface of a material during its fatigue life. Such monitoring, which was validated by in-depth ß-measurements, gives information about the spatial and temporal evolution of the fatigue damage and suggests different damage evolution modes for laser treated and untreated materials.

The surface ß-measurements were performed along the tensile axis of laser treated (surface remelting) and untreated AISI 316L flat specimens subjected to interrupted high cycle fatigue tests in load-control pull-pull mode in order to determine the work hardening state. The results of surface ß-measurements (example in Fig. 33) revealed that the work hardening of the surface increases during fatigue life. At each fatigue life fraction a maximum work hardening level bmax is reached in some point of the gauge length. A critical value of $ß_{crit} \cong 1^{\circ}$ can be associated with incipient fracture. The ß-curves obtained for laser surface melted materials suggest that the increase of surface work hardening is quite uniform until the very last fraction of the fatigue life when a sharp maximum develops in the fracture zone. This localization may be associated with non-homogeneously distributed damage nucleation sites. A threshold value of $ß_{crit} \cong 0.8^{\circ}$ can be associated with incipient fracture in this case. A correlation between $ß_{max}$ and the fatigue life fraction (Fig. 34) was detected and could be used, in principle, to estimate the fatigue life. The lower ß-values observed for laser treated specimens both in Fig. 33 and Fig. 34 correspond to the lower amount of work hardening that these specimens can accumulate during fatigue with respect to the untreated ones due to their initial work hardened condition induced by the laser treatment.

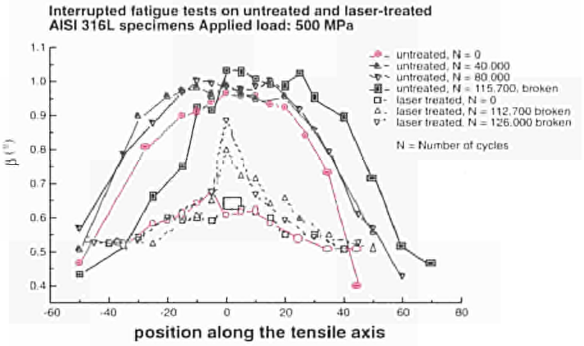


Fig. 34: Results of ß-measurements on the surface of untreated and laser treated AISI 316L specimens fatigued 500áMPa. Position "0" is chosen in the middle of the gauge length for unbroken specimens and close to the fracture surface of the broken ones.

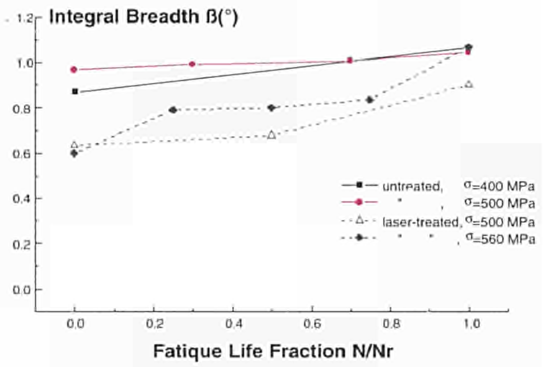


Fig. 35: The integral breadth maximum as a function of the fatigue life fraction N/N

Since the experimental procedure for ß surface monitoring is quite simple and relatively rapid, it is planned to test the method for other materials (e.g. Al alloys) and surface treatments.

Surface Characterization by Photothermal Measurements

An improvement of the surface properties, and possibly the fatigue behaviour, can be achieved by laser transformation hardening instead of surface remelting. In this case the quality of the hardened surface is determined by the hardness value at the surface and the hardness profile achieved in the subsurface region. Photothermal measurements have been found to be a promising technique for non contacting hardness and hardness profile determination based on the correlation between hardness and thermal diffusivity.

In 1996, efforts for establishing a European network for qualifying this hardness measurement method for industrial use were successful. The IAM will act as co-ordinator of a total of 11 participants from research laboratories and industry and contribute by microstructural studies to the characterization of the hardened surfaces.

THE SLOW DISSOLUTION OF REFRACTORY CRUCIBLES

Local erosion of solids in contact with gas/liquid or liquid/liquid interfaces is a well known phenomenon. The purpose of this project is to study the erosion of a partially immersed vertical crucible wall into a molten substance, to analyse and understand the different forces and the basic generic laws contributing to this phenomenon, which is common to both glass making and metallurgical industries.

The slow dissolution of a partially immersed solid at the vertical interface leads to a concentration gradient. This can give rise to longitudinal interfacial tension gradients, the direction of the convection currents depending upon whether the solute is positively absorbed or negatively adsorbed. This is essentially the mechanism that has been suggested to explain the preferential attack of refractory at the surface line of molten slag and glass in steel and glass making furnaces. It has been recognised that the process of corrosion of refractory materials is actuated by currents which sweep away the dissolved wall material into the bulk and consequently increase the concentration gradient of dissolved crucible material at the wall surface.

A mathematical model has been developed on the assumption that the wall erosion profile can be explained by the disruption of the dissolution concentration gradients by buoyancy and surface tension driven convective flows. The problem is formulated by considering the Navier-Stokes equations coupled with the energy and diffusion equations. The velocity field is determined by the equations of motion and energy, whereas the concentration distribution is determined by the convective diffusion equation. All these equations are mutually dependent since the surface tension is a concentration function. The concentration distribution depends on the velocity distribution, and the driving mechanisms of the flow are the buoyancy and surface tension forces.

In this study, it was assumed that the slow dissolution of the refractory wall is a diffusion controlled process and is governed by the concentration gradient of the dissolved crucible material. Results are presented for the selective case of concentration density driven flow. In the case of pure natural convection due to concentration gradients resulting from the dissolved wall material where the density of the crucible material is less than that of the melt, creates an upwards circulation pattern. This type of flow pattern sweeps away the dissolved material at the wall, from the bottom to the top, by continuos replenishment with fresh bulk molten material with lower concentration of crucible species. The dissolved material from the wall accumulates at the top part of the side of the wall of the refractory.

BIOMEDICAL APPLICATIONS: ION IMPLANTATION TO REDUCE WEAR OF UHMWPE IN JOINT PROSTHESES

Summary

The most frequently used friction-couple in artificial hip joints is UHMWPE (ultra-high molecular weight polyethylene) for the pelvis-socket liner with ceramic or metal for the femur head-ball (Fig. 35).

Concerning metal head-balls, stainless steel AISI 316 and the Co29Cr6Mo alloy are the most frequently used, on account of their good tribological properties. The Ti6Al4Va alloys lead to a much higher wear of the UHMWPE socket.

The purpose of the exercise reported here was to investigate whether ion implantation could:

- improve the titanium alloy to such an extent as to reduce PE wear to a level comparable to that achieved with the other two metals;

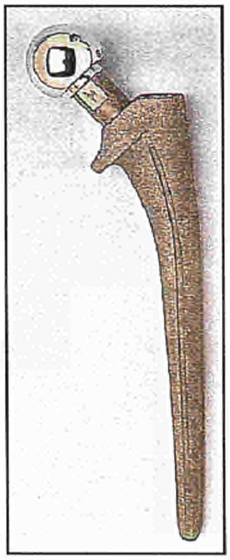


Fig. 35: Artificial hip joint

- further improve the behaviour of the Cobalt alloy.

Our results suggest that:

- ion implantation with chromium can improve the titanium alloy even beyond the level currently achieved today by industrial implantation with nitrogen;
- ion implantation with nitrogen or oxygen can further improve the tribological behaviour of the Cobalt alloy.

Equipment and method

Ion implantation was performed by means of a high power implanter manufactured by DANFYSIK (Fig. 36).

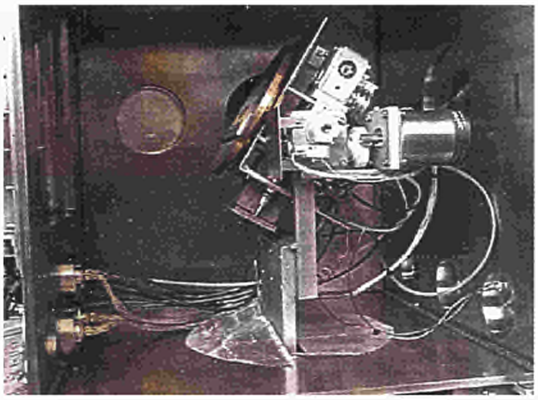


Fig. 36: DANFYSIK 1090 implanter: the target chamber

The metal specimens were discs of 40 mm diameter, mirror polished before implantation. The uniformity of implantation was checked with XPS analysis. The UHMWPE specimens were pins of 9 mm diameter, 13 mm height. They were loaded with a 23.5 kg mass, thus achieving a specific load of 3.54 MPa in the reciprocating "pin-on-flat" machine shown in Fig. 37.

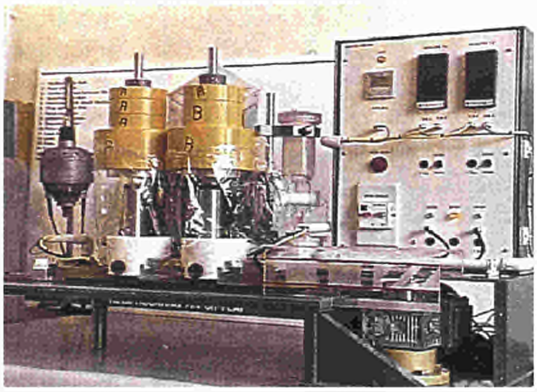


Fig. 37: "Pin-on-flat" machine

The pin wear was measured by weighing. The lubricant was bovine serum. Each test was repeated three times. Tests and methods comply with ASTM F 732-82.

Results

The most important data are summarised in Table III and Fig. 38.

	AISI 316	Ti	Ti+N	Ti+O
Wear	1±0.3	100±10	80±10	0.2±0.05

	Ti+Cr	Co	Co+N	Co+O
Wear	0.04±0.01	0.25±0.03	0.12±0.01	0.17±0.02

Table III: Wear of UHMW-PE specimen after 10^6 cycles (mg); (Ti stands for Ti6Al4V, Co stands for Co29Cr6Mo)

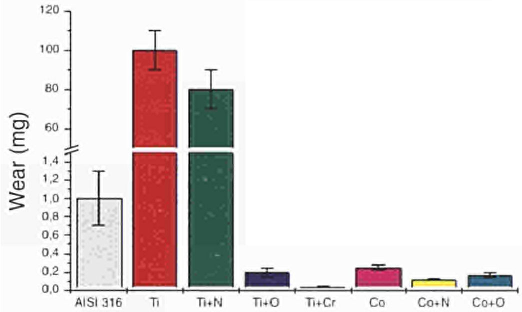


Fig. 38: Wear of UHMW-PE specimen after 10^6 cycles (mg)

Conclusions

The industrial practice of implanting the Ti alloy with nitrogen can be improved by implanting chromium. Costs are slightly higher, but the reduction of wear seems to justify them.

Oxygen implantation on the Ti alloy leads to a reduction of wear to the same level as with the Co alloy.

Chromium implantation with the Ti alloy leads to a reduction of wear to about 1/3 of the best level achievable with the implanted Co alloys, which in turn is about half of the level achievable with the non-implanted Co alloy.

It is interesting to note that O implantation is much better than N implantation for Ti, whereas the opposite is true for the Co alloy.

Concerning the biocompatibility of chromium as compared to nitrogen, undoubtedly the latter is better; however the amount of chromium released by the implanted Ti alloy would be much lower than that released by the CoCr alloy, which has long been recognised as acceptable.

SUPERHARD SURFACE

Introduction

Light element coatings are the subject of intensive research. Besides the well known coatings such as diamond, diamond-like coatings, h-BN, c-BN and B_4C, there is interest also in CNx coatings and coatings within the B-N-C system. The Thin Film Laboratory (TFL) has investigated both types of coatings, the latter in the framework of a 'Human Capital and Mobility' network project (FUNINCOAT).

Results and Discussion

CN_x coatings

CN_x coatings were sputter deposited by a dual ion beam facility. One ion beam was used to sputter a carbon target with nitrogen ions. This provokes the release not only of carbon atoms but also of CN based compounds which are deposited at the substrate. The second ion beam was used to supplement the growing film with nitrogen ions. Reduction of the substrate temperature from 300 K to 77 K in conjunction with low sputter beam voltages (< 200 V) caused the nitrogen concentration to attain a maximum value of 44%, the optical band gap to increase from 0.2 eV to 2.2 eV (Fig. 39), the sheet conductivity to decrease from $10^{-2}(\Omega.cm)^{-1}$ to less than $10^{-9}(\Omega.cm)^{-1}$ and the density to be reduced from about 2 to 1.6 g/cm^3.The chemical structure was investigated by Fourier Transformation Infra-Red spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and Auger Electron Spectroscopy (AES). The increasing transparency is accompanied by structural changes indicating a transition from a predominantly sp^2 bonded amorphous sp^2/sp^3 C-N network to a more linear polymer-like structure consisting predominantly of doubly and triply bonded C and N atoms.

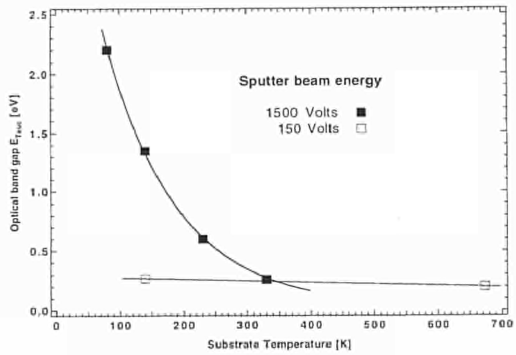


Fig. 39: Effect of substrate temperature and sputter beam voltage on the optical band gap.

B-N-C coatings

B-N-C coatings of the 'FUNINCOAT' project were synthesised in various laboratories of the participating institutions. Characterization was carried out using several techniques, in various laboratories, and it was the main task of the TFL to perform glancing angle X-ray diffraction measurements on the films. Density measurements using X-ray reflectivity were also carried out successfully on some coatings, but the curvature of the samples limited the useful application of this technique to only a few films. Most investigated films within the B-N-C system were mainly amorphous, whereas BN films displayed broad diffraction peaks more characteristic of a nanocrystalline structure as already found in our laboratory.

Conclusions

Substrate temperature and nitrogen ion energy have a strong influence on the optical and electrical properties of CNx coatings. B-N-C coatings were found to be amorphous with hardness values usually between 10 GPa and 20 GPa.

LASER SPALLATION

Introduction

Measurement of interface properties is not new in the thin film and coatings technologies. However most of the available techniques provide only a rough measure of the strength of interfaces. Such interface strength between thin coatings and substrates can be effectively investigated by a pulsed laser induced spallation technique, utilizing shock waves produced by an energy pulse of short duration, and coupled with simultaneous measurement of the spalled film free surface velocity by means of a Laser-Doppler-Interferometry device (VISAR). The great advantage of this approach is that it is the only method by which the strength of adhesion of a thin film, as well as its Modulus of Elasticity, can be evaluated accurately and unambiguously. Details about the experimental and theoretical/numerical techniques developed are included in the 1993, 1994 and 1995 editions of the IAM Annual Report.

Objectives

To develop and validate advanced experimental techniques based on a pulsed laser induced spallation method and laser Doppler interferometry for the investigation of bonding strength and modulus of elasticity in coatings and thin films with application to optics, micro-electronics and fibre reinforced CMCs.

To develop and validate state of the art computational methods for the prediction of a.) the pressure pulse generated by the interaction of short energy pulses with energy absorbing matter and b.) the threshold tensile stress normal to the plane at the material interface during spallation.

Methodology

Development of a spallation test based on a high energy density pulse interacting with a thin energy absorbing film deposited on the rear surface of a flat substrate/coating specimen. Determination of the required threshold energy level; monitoring of the film velocity profile during exfoliation yielding the film modulus of elasticity and its bonding strength.

Development of a theoretical/numerical model simulating the spallation test based on a hydro-dynamic formulation. Implementation of appropriate equations of state for the energy absorbing material leading to an estimate of the generated pulse profile and computation of the tensile strength of the material interface by elastic wave propagation.

Results

The performance of the new pulsed laser system based on 300 pico-second pulses was investigated. This system yields pressure pulses of higher amplitude and much shorter duration, compared with the 8 nano-second pulses, allowing for testing of films of sub-micron thickness.

Spallation tests were performed on copper and aluminium films of 0.5 to 2 micron thickness deposited on silicon wafers with variable success due to the relatively low strength of silicon compared to high film bonding strength exhibited in some cases.

Deliverables

Reference measurement and theoretical/numerical modeling methods for the evaluation of the coating and thin film deposition process techniques. Parameters based on quantitative investigation of a.) the bonding strength of the material interface and b.) the modulus of elasticity of the deposited thin film.

We claim that our methods are indispensable for the investigation of fundamental mechanical properties of material interfaces associated with thin films. In fact this is the only clean test that can lead to the evaluation of the mechanical properties of the film.

Industrial relevance

Reliable quantitative estimates for the bonding strength of material interfaces associated with coatings are indispensable for the development of advanced composite materials and their implementation in industrial systems.
The investigation of the mechanics of material interfaces associated with thin films is essential for the development of several optics and micro-electronics applications.

Achievements

"Validation of thin films bonding strength measurement system", Project proposal to be submitted to the VALUE programme.
"Numerical simulation of a pulsed laser induced spallation test", Ph.D. thesis by M. Kiriakopoulos, University of PATRAS, Greece, May 1996.

"Experimental and theoretical/numerical investigations of bonding strength in thin films", Invited lecture, European Research Conference on interfacial Engineering of Materials, Il Ciocco, Italy, 9-14 October 1996.

"High speed interferometry with a new kind of VISAR", Proceedings, 22nd International Congress on High-Speed Photography and Photonics, Santa Fe, New Mexico, USA, 27 October- 1 November 1996.

Two scientific papers by A.G. Youtsos and M. Kiriakopoulos, invited to be published in Theoretical and applied fracture mechanics, are in preparation.

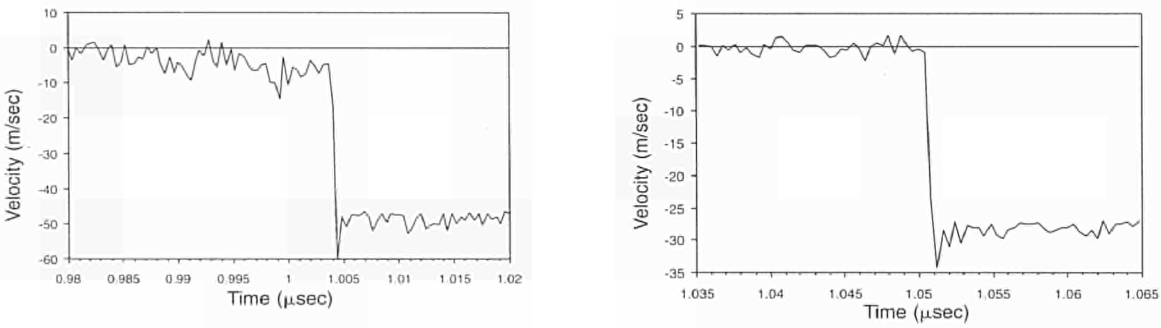


Fig. 40: Velocity profile of a 5 μm SiC coating deposited by PECDV during Exfoliation from Al_2O_3 Substrate

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INTERFACES IN FIBRE COMPOSITES

The interest in continuous fibre reinforced Ceramic Matrix Composite materials (CMCs) lies in the ability of fibres to prevent catastrophic brittle failure in ceramics by various energydissipation processes during crack propagation, and thus providing defect tolerance. This behaviour depends on the special properties of the interface between the fibres and the matrix.

The five year project to engineer the fibre-matrix interface structure and chemistry by precoating of fibres, in order to optimise toughness and defect tolerance, focused from the beginning on unidirectional ceramic matrix composites (C/Si_3N_4) fabricated by liquid infiltration of long carbon fibres with submicron Si_3N_4 powder slurries, followed by hot-pressing.

This methodology was then adapted for the newly developed ceramic SiC-based fibre (Hi-Nicalon, Nippon Carbon) where a deposition of a 200 nm pyrolytic carbon layer (courtesy of "Laboratoire des

Multimatériaux et Interfaces, University Claude Bernard-Lyon) on the fibres prior to fabrication was necessary to decrease the interfacial bond strength and to avoid the brittle fracture of the composites. Under these conditions, a "pseudo-plastic" mechanical behaviour and a great increase in toughness by matrix microcracking and pull-out of the broken fibres has been observed. The sample dimensions were then scaled-up to perform mechanical tests under tensile load. The unloading-reloading cycles performed in the non-linear G-region (Fig. 43) illustrate the damage consequences, i.e., a progressive decrease in the material's longitudinal stiffness, an extension of the residual strain and an increase of the surface area of the hysteresis loops. This phenomenon depends mainly on the characteristics of the fibre/matrix interface and more specifically to the interfacial shear stress due to fibre/matrix sliding and debonding.

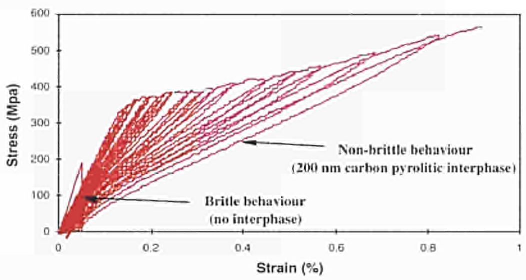


Fig. 41: Tensile stress-strain curve with hysteresis loops for a SiC (Hi-Nicalon) reinforced Si_3N_4 composite

The above technology is being transferred to textile preform infiltration. A useful textile preform for structural reinforcement of ceramic matrix composite materials is the bi-directional (2D) woven fabric. Fig. 42 illustrates the schematic diagram of the plain weave where two sets of yarns are interlaced at right angles. The major difficulty with slurry infiltration is in filling the interfibre spaces uniformly and densely with the matrix phase, ensuring a minimum of entrapped voids. The variables of particular importance are: stable and well-dispersed suspensions, excellent wetting of the fibres by the slurry, small ceramic particle size, a controlled consolidation technique and finally, a judicious choice of 2D fabrics which are characterised by the spacing between adjacent yarns, the number of filaments per yarn and the bundle size. Fig. 43a illustrates the interwoven pattern of carbon fibres in a Si_3N_4 matrix. The composite is not microcracked after processing and the 2D fabric is fully infiltrated with uniform distribution of carbon fibres in a dense Si_3N_4 matrix (Fig. 43b). The Institute has already patented the C/Si_3N_4 composite for wear applications.

As a further competence, the project has designed and constructed a manufacturing technique for filament winding. This method consists of winding continuous fibres onto a rotating mandrel from a moving carriage in a specific predetermined pattern. The equipment is fully programmable from a PC in order to control the variables of the process such as fibre architecture, infiltration rate, winding speed, and tension. Filament winding is an important processing technique for producing complex shapes of rotational symmetry (e.g. CMC tubes for heat exchangers).

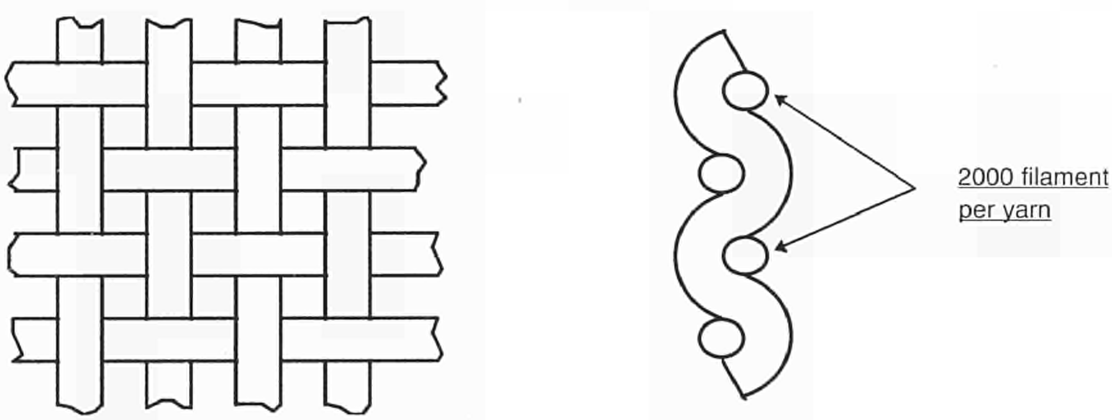
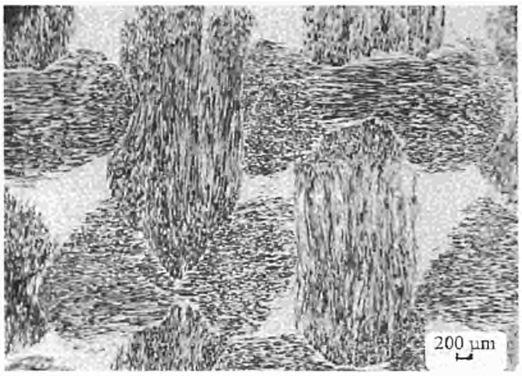


Fig. 42: Schematic diagram of plain weave with sets of yarns interlaced at right angles



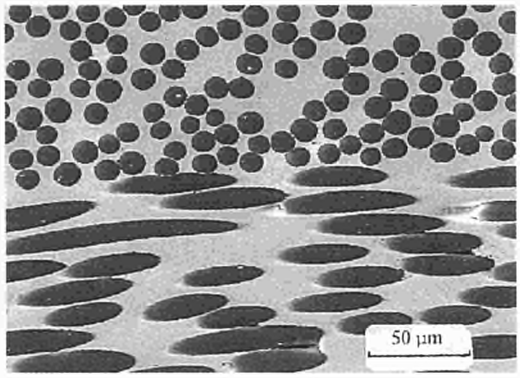


Fig. 43: Bi-directional (2D) woven fabric infiltration a) interwoven pattern in 2D C/Si3N4 and cross-section of fibre bundles showing degree of infiltration

Continuous Fibre-reinforced Ceramic Composites (CFCC) are important candidate materials for high temperature applications in power generation systems. Their strength, toughness and damage tolerant behaviour, as well as the resistance to oxidation environments are critically dependent upon the nature of the fibre-matrix interface. The engineering of fibre-matrix interfaces is currently performed in conventional Chemical Vapour Deposition (CVD) installations by testing at laboratory scale different coating layers onto single filaments. The industrial production of coated fibres for the manufacture of CFCCs must be adapted to the particular nature of the substrate and the functions of the coating. The reinforcing multi-filament fibre tows require a reproducible deposition of uniform coating layers on thousands of individual filaments of the yarn, over lengths of the order of hundreds of meters.

A pilot plant CVD facility fulfilling these stringent requirements has been recently designed, constructed and commissioned in the Institute of Advanced Materials to support the on-going development of CFCCs for industrial applications. This prototype installation executes in a single run fibre pre-treatments (desizing, etc.) and continuous deposition of multi layer coatings (SiC, Si_3N_4, p-C, BN, B_4C, etc.) onto individual filaments of endless fibre tows in a wide range of deposition temperatures and pressures (up to 1300o C and between 10^{-4} Torr and atmospheric pressure, respectively). A key component of this facility is the fully automated bi-directional fibre tow transport system located in a single vacuum chamber (Fig. 44) adjacent to the CVD reactor, which includes the following features: constant speed operation irrespective of spool size, reversible fibre motion, automatic fibre tension adjustment and spreading, and synchronized transport tape to prevent fibre interweaving and sticking in multi layer coatings. This device is protected by the EUROPEAN PATENT nr. 96114406.0. Further extensions of this patent to USA, Canada and Japan have also been applied for. Preliminary tests have shown that the present facility is capable of producing fairly uniform coatings with controlled microstructures, as shown in Fig. 45 for a p-C coating layer deposited onto FT-500, 3K multi-filament carbon fibre tows.

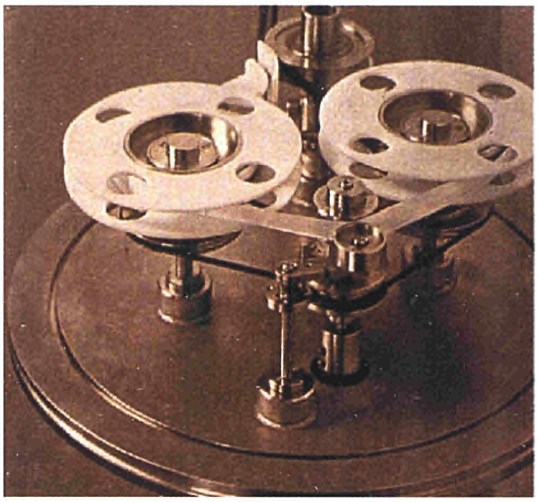


Fig. 44: Fibre tow transport system.

Fig. 45: Pyrolytic Carbon coating layer.

INDUSTRIAL TECHNOLOGY: ADVANCED MATERIALS-STANDARDS FOR APPLICATION

ECO MATERIALS

The objective of this project is to study, in collaboration with the Environment Institute, the emission of eco-unfriendly metals in the environment and the effects thereof, especially by radiotracer methods. It also studies possible ways to increase the environmental compatibility of materials.

The IAM has further expanded its activities in the use of radiotracers for surface degradation studies, such as corrosion and wear using the innovative Thin Layer Activation (TLA) method. The principle of TLA is the creation of radionuclides in a surface layer by exposure to a high energy particle beam, for example from a cyclotron. The activated layer emits nuclide specific γ-radiation. Any loss of the activated material due to a material degradation process will result in a loss in γ-activity of the activated component, directly related to mass or depth loss. Specific properties of TLA include area selectivity, high sensitivity, speed and applicability as a non-contact in-situ method. In this area, the IAM benefits significantly from its unique position of being an institute for materials research with cyclotron facilities. Besides a variable energy cyclotron, for studies involving TLA, the IAM is in the possession of a γ-spectrometry laboratory and specialized cyclic corrosion and wear test facilities.
The TLA method is expected to contribute significantly in the field of biomaterials because in these applications the release of even small amounts of materials can be critical. Conventional test methods are often limited by their relatively low sensitivity. In the year under review, the IAM TLA/pin on disk lubricated wear test facility was successfully employed in the development of ZrN coatings for biomaterial applications. This activity is performed in collaboration with the IAM Surface Engineering Unit. The TLA method appeared to be a very fast screening method, due to its sensitivity which is order of magnitude higher than classical gravimetry. Thus it does not only supply quantitative data of the amount of degradation, but also gives more insight into the degradation processes.

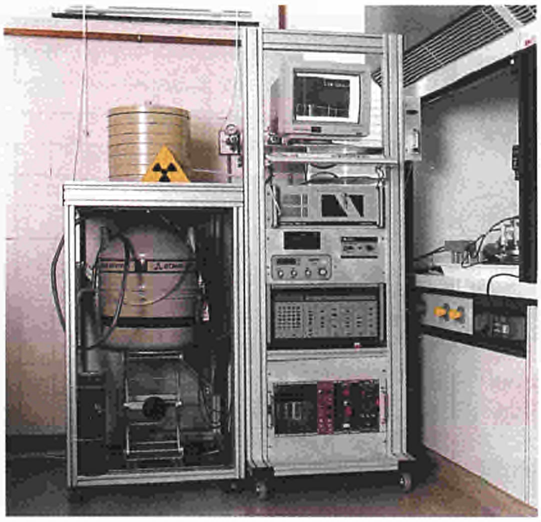


Fig. 46: The IAM facility for electrochemical release testing using Thin Layer Activation.

In 1996, a TLA electrochemical test facility was developed and became operational. The experimental installation allows simultaneous online measurement of corrosion parameters and concentrations of released radiotracers. The latter analysed with a dedicated gamma spectrometry facility, allowing for very small concentrations to be monitored.

The facility's key application is in the area of heavy metal release due to corrosion under conditions relevant to technical components in the food processing and pharmaceutical industries for example. Preliminary results on the release of AISI 316L components in glucose solutions indicated the TLA data to be complementary to the electrochemical data. The facility was designed in a multi-purpose way, to be useful for a wide range of applications, for example to address the subject of metal release related to biomaterial applications. These activities are strongly related to life science.

COATINGS FOR DRY MACHINING AND AS A SUBSTITUTE FOR ELECTROZINC

Introduction

As a result of intensive R&D work in recent years hard coatings for various applications have been developed and are now in industrial use. Nevertheless, there is a continuous need for the development of new types of coatings. Presently, the machining industry requires coatings to be used on cutting tools for lubricant-free machining in order to reduce the increasing costs associated with cutting fluid and chip disposal and to reduce environmentally dangerous waste.

To synthesize suitable materials, the Thin Film Laboratory of the IAM/Ispra developed a concept based on multiphase coatings. Multiphase coatings can be considered as composite materials. They are in most cases nanocrystalline and also very hard. If one of the constituent phases has lubrication properties it should be possible to obtain coatings with a very low friction coefficient whilst maintaining hardness values above 25 GPa (hardness of TiN).

Results and discussion

In this reporting period, coatings within the ternary system Ti-B-C were investigated. Fig. 47 shows the phase diagram of this ternary system together with the composition of the synthesized coatings. The coatings were deposited by magnetron co-sputtering from a TiB_2 and a graphite target. To obtain coatings of different composition, stainless steel substrates were placed on a substrate holder in different positions relative to the TiB_2 and the C target along their centre line (Fig. 47). Sputtering from the TiB_2 target was performed in d.c. mode and from the C target in r.f. mode. The sputter power applied at the TiB_2 target was 300 W d.c. and at the C target 1.0 kW r.f. Deposition rate under the targets were 14 nm/min for TiB2 and 16 nm/min for C. The bias voltage at the substrates was applied by r.f. via a charging condensor. Argon was used as sputter gas at a flow rate of 137 sccm. Evacuation of the vacuum chamber was accomplished by a 3000 l/s cryogenic pumping system down to 10^{-7} mbar.

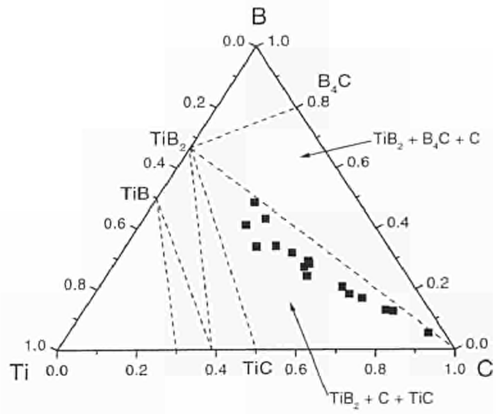


Fig. 47: Phase diagram of coatings within the Ti-B-C system

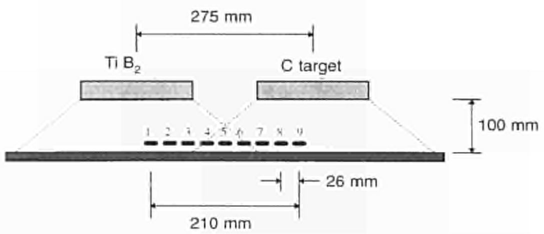


Fig. 48: stainless steel substrates placed on a substrate holder in different positions relative to the TiB_2 and the C target along their center line to obtain coatings of different composition.

All deposited films were examined by glancing angle x-ray diffraction. The diffraction spectra of films of various composition ranging from $TiB_2CO_{0.3}$ to TiB_2C_9 is shown in Fig. 49. Due to the low atomic numbers of B and C with respect to Ti, the Ti containing phases are expected to dominate the XRD spectra except at the higher carbon contents. At lower carbon contents the spectra show essentially a structure typical for TiB2. It is interesting to observe that with increasing carbon content the peak positions are slightly shifted to lower diffraction angles indicating a slight lattice expansion probably due to interstitial carbon atoms. At even higher carbon contents a strong line broadening and a line shift can be observed. Such diffraction spectra are typical for Ti being incorporated in the amorphous structure of a diamond-like carbon coating of the type a-C(Ti). Such coatings are known to be formed under the deposition conditions of a substrate position near or directly below the carbon target. The spectra do not display peaks which might be interpreted as being due to the presence of a TiC phase which is expected also to be present in that area of the phase diagram, where the coatings are positioned (Fig. 47). However, a closer inspection shows that the coatings are expected to contain, in the equilibrium conditions valid for the phase diagram, only a minor TiC phase fraction due to the vicinity to the quasi-binary phase line TiB_2-C.

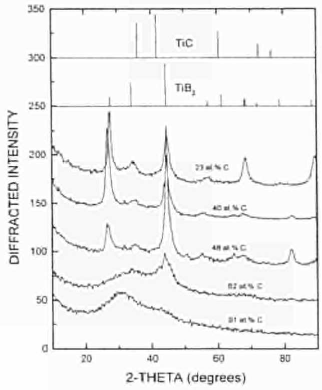


Fig. 49: diffraction spectra of films of various composition ranging from $TiB_2CO_{0.3}$ to TiB_2C_9

The chemical composition and the type of bonding of the coatings was investigated by XPS/AES spectrometry by using a Riber Nanoscan 50 spectrometer incorporating a semi-imaging MAC2 analyzer working in the constant analyzer energy mode. The XPS C1s peak of the coatings $TiB_{1.1}C_{3.4}$ and $TiB_{1.3}C_{8.4}$ are shown in Fig. 50. The two main components correspond to graphitic and or DLC type phases and $Ti(BC)_x$ phases in the material. As the C content is increased, the graphitic (low friction) component becomes dominant.

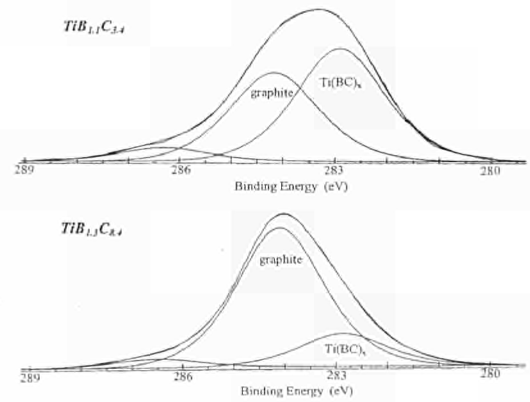


Fig. 50: XPS C1s peak of the coatings $TiB_{1.1}C_{3.4}$ and $TiB_{1.3}C_{8.4}$

The results of hardness measurements performed with an ultra-low load and depth sensing nanoindenter are shown in Fig. 51 as a function of the carbon content of the coatings. As can be seen, there is an almost linear dependence which is due to a continuous decrease of the hard TiB_2 phase content at the expenses of an increase of the amorphous a-C(Ti) phase.

This a-C(Ti) phase can display high hardness values up to 35 GPa [ref] if it is synthesized with a high sp^3/sp^2 bonding type ratio which, however, seems not to be the case with our coatings. Coatings which were deposited under substrate bias voltage show generally a higher hardness, as expected.

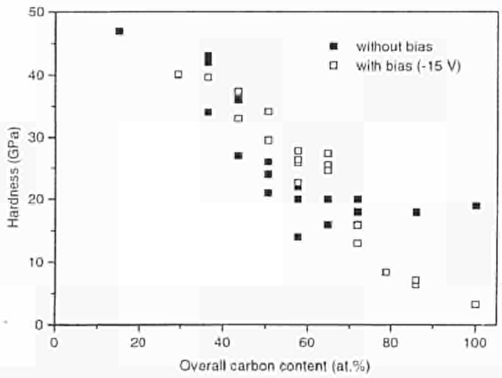


Fig. 51: Hardness measurements performed with an ultra-low load and depth sensing nanoindenter as a function of the carbon content of the coatings

Fig. 52 is a plot of the friction coefficient, as measured as a function of the carbon content with a pin-on-disk tribometer at a velocity of 0.1 m/s and under a load of 5 N using a 316L steel ball as counterface. As expected, the friction coefficient decreases with increasing carbon content down to a value of less than 0.2 for a carbon content of about 60 %. At this value the hardness of the coating is still greater than 20 GPa, a value typical of the TiN coatings which are used for many cutting tool applications. Interestingly, coatings sputtered with bias voltage have at intermediate carbon contents a higher friction coefficient a behaviour which cannot be explained simply.

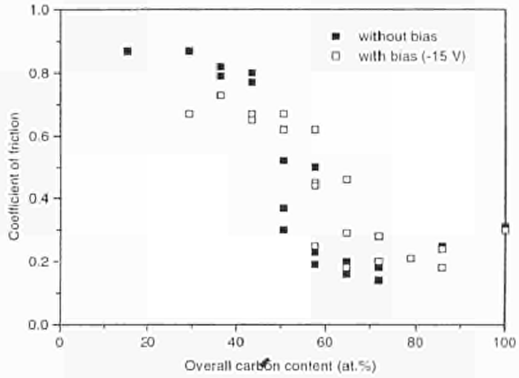


Fig. 52: Plot of the friction coefficient, as measured as a function of the carbon content with a pin-on-disk tribometer

Fig. 53 shows a plot of the hardness/friction coefficient ratio as function of the carbon content of the coatings. The highest ratio is obtained at 65 % carbon content with H = 25 GPa and C = 0.18. This value should be compared with values of 'conventional' coatings presently used for cutting applications such as TiN with H = 22 GPa and C = 0.6 and TiAlN with H = 28 GPa and C = 0.7.

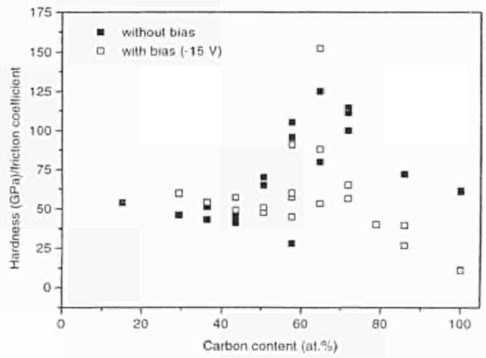


Fig. 53: Plot of the hardness/friction coefficient ratio as function of the carbon content of the coatings

Conclusion

Applying the concept of multiphase materials containing lubricating and hard phases, coatings of high hardness and low friction coefficient could be synthesised.

INDUSTRIAL TECHNOLOGY NDE AND INSPECTION

APPLICATION AND QUALIFICATION OF NDE TECHNIQUES

Introduction

The role of inspection and application of NDE techniques is increasingly important for industry, both for advanced materials and high strength steels and alloys. Application of new joining techniques leads to the development of new methods of manufacturing industrial components and consequently to the adaptation of the NDE techniques for quality control and ageing monitoring.

Non Destructive Evaluation guarantees reliable economic and safe operation of capital intensive plants by reducing the number of failures and unforeseen shut-downs. Characterisation and monitoring of materials integrity and degradation are essential for the correct maintenance and life management of industrial equipment.

The need to apply effective NDE requires further evaluation of inspection capability by a neutral organisation such as JRC.

To apply NDE in a controlled way and to allow effective competition between service vendors, qualification of inspection techniques in the sense of demonstration of capability is an important request of industry and standard bodies.

Damage of pressure equipment due to ageing and aggressive environments requires monitoring techniques to supplement the damage evaluation and modelling.

Transfer of results/technology from the nuclear field

The satisfactory application of probabilistic safety assessment methods calls for validated distribution of input parameters. Flaw size, shape and distribution for real components arise from NDE reports. The PISC data base has been proposed for use in the evaluation of the probabilities of both "non detection" and "incorrect evaluation" with respect to the PISC assemblies and to suggest potential correlations of flaw parameters, component types and NDE inspection methods with those probabilities of non-detection and incorrect evaluation.

The final report on the PISC III Action 5 "Steam Generator tubes inspection" was prepared and distributed for comments.

The main objective of this action was the experimental evaluation of the performance of test procedures and techniques available to inspect steam generator tubes in-service. The exercise was characterised by the geometry (thin walled tubing), the material (Inconel 600), the large number of flaws and the inspection techniques (predominantly eddy current with a few beams using ultrasonics).

Identification of the lack of NDE performance and the importance of human factors greatly concern the heat exchanger tubes inspection community: vendors, utilities, safety authorities. Agreement for open final publication is expected to ask for more discussion and iteration.

Ultrasonic examination of austenitic stainless steel welds

Reliable ultrasonic inspections of austenitic stainless steel welds to detect and classify flaws which could cause weld failure, are mainly hampered by the high attenuation of the ultrasonic beam. The ultrasound is scattered and mode-converted at the grain boundaries of the austenitic weld metal leading to a lack of confidence in the NDE evaluation of such welds.

Techniques for flaw detection characterisation and sizing using ultrasonic signals need the physical understanding of ultrasound grain scattering in anisotropic austenitic welds.

Centrifugally cast and wrought material used during the PISC III Action 4 have been selected for the experimental determination of the attenuation coefficients and the grain scattering. These will be used to demonstrate the capability of the improved examination procedure which is to be developed as a result of this theoretical study.

An important spin-off of this action is an SMT contract on ultrasonic scattering in austenitic welds with industrial inspection companies and a national research centre.

Contribution to standards

The European Methodology for Inspection Qualification developed by the ENIQ networks was officially registered by the CEN Technical Committee 138 Non Destructive Testing and is used as a basis for discussion and development of non-nuclear standards of inspection.
Assemblies simulating piping, pipelines, collectors and vessel welds were certified with respect to defect simulation in view of inspection qualification.

Spin-offs

As mentioned above several applications were paid for by industry. New proposals were made either directly to adurty or through several Shared Cost Action proposals with a sting component of on co-operation with industrial companies. Contracts were obtained from Brite-Euram and SMT to be executed in the period 1996-1998.

MATERIALS CHARACTERISATION

Post manufacturing quality control and in-service inspection of TBC's are gaining importance. Several European industries, such as energy production companies, have already shown interest in this field. However, research has usually been achieved individually, with few contacts with other potential partners. Moreover, European industry is still behind America with regard to coating technology. Being able to share resources and access a common database of experience would anist in cloring the gap with the American market leader.

Non-destructive evaluation (NDE) of TBC's is still in a critical phase. The immediate creation, of a network for the co-ordination of research and the improvement of communication between laboratories and industries dealing with TBC technology would certainly give a valuable impetus to the European activities.
Work done at the Ultrasonics Development Laboratory of the IAM – at JRC Ispra, has shown that the ultrasonic NDE of commercial metallic coatings is perfectly feasible. It can be very useful for the post manufacturing determination of coating characteristics, such as the layer thickness, the elastic constants and the density, as well as for the detection of defects, such as debondings and surface-breaking cracks.

Similar research is currently being carried out at the JRC Ispra and JRC Petten (NL) on plasma-sprayed and electron beam physical vapour deposited (EB-PVD) TBC's. It appears that ultrasonics can give valuable information on the microstructure of TBC's, as well as on the porosity content or the presence of defects. These characteristics define the quality of the coating and are critical for its lifetime. The development of a new probe prototype for the NDE of TBC's, which could easily be turned into a commercial sensor, has already started at Ispra. Other labs across Europe, in industry and at universities, are currently working on ultrasonic NDE of coatings, with promising results.

The creation of a network on TBC's involving the NDE aspect of coating technology is desirable since it would help avoiding the loss of resources due to duplicated activities. All the literature concerning the application of ultrasonics to coatings could be made easily accessible from a database, possibly using Internet or the World Wide Web. Finally, if the network includes an electronic messaging system, new findings could be shared by the other members of the network more quickly than through the conventional scientific publishing system.

EPERC

EPERC is a European network of national networks and of individual industrial and R&D institutions with the objective of strengthening the co-operation spirit of the pressure equipment (PE) industry. EPERC aims to increase the effectiveness of R&D work done on the subject in view of codes and standards, of developing the transfer of information to the benefit of Small and Medium Enterprises.

Since January 1996, work has been in progress to establish a wider national representation within EPERC and we are happy to report today that representation has been established for France, UK, Germany, Italy, Netherlands, Denmark, Sweden, Greece, Spain, Finland, Belgium, Austria and Switzerland. Negotiations are underway with potential representatives from Ireland.

Administrative Task Groups

As announced in 1996 the business of EPERC was initiated by means of a series of 5 administrative Task Groups as defined below. Progress and actions undertaken by these groups in the past period are detailed below.

TG1 – Business management of EPERC
Considerable progress has been achieved with the realization of the first version of the Business Plan. This document provides the modus operandi and the function for EPERC, and will be used as a basis for promotion and marketing purposes. It has been designed in such a way as to convince potential partners and clients of the strengths and professionalism of the EPERC approach. This document has been distributed for comments.

TG2 – Current European and International R8 D activities
This group has been active in a number of areas particularly in the execution of a survey of research currently undertaken by the PE community in Europe. This report will be completed by March 1997. Another important action which has been addressed is the preparation of the terms of reference for some dedicated research calls of the European Commission's SMT program. This work was performed on behalf of CEN TC54 and it is hoped to establish similar arrangements with other CEN Technical Committees associated with PE.

TG3 – R & D needs of Industry
TG3 has been active over the last period with the execution of a survey of the RED needs of the European PE industry via a detailed questionnaire. The preliminary evaluation of this data has now been completed and first results indicate that technical actions should be proposed for Fatigue Design, Inspection Management, High Strength Steels, Gaskets.

TG4 – Support of European policy and CEN
This group has been active in bringing the existence of EPERC to the attention of CEN and to specific Directorate of the European Commission. In the case of

DGIII (Industry) this has led to the raising of a funded action in the field of Design by Analysis (DBA).

Further proposals were made to DG III regarding specific actions on Inspection harmonization.

TG5 – Technology Transfer
Regarding the Technology transfer actions of TG5 the decision has been taken to establish an Internet World Wide Web site (WWW) for EPERC to facilitate both the transfer of technology actions but also to serve as an administrative engine for the Network and the secretariat. In this respect all EPERC members are strongly encouraged to realize an Internet WWW connection and email link before the end of April 1997.

Technical Task Forces (TTFs)
There has been much comment and discussion regarding the fact that EPERC should be seen to be activating technical and not only administrative actions. In response to these wishes EPERC is to initiate a number of technical action lines, 3 in the first instance. These will be referred to as EPERC Technical Task Forces (TTFs) and the action themes will be those priority topics arising from the TG3 survey of industrial needs, moderated with respect to the results of the survey of current European RID topics performed by TG2.

The Initial objective will be to recruit a small number of experts in the field of each of the TTFs. This group will be mandated to establish the Programrne for the TTF. This will consist of a Mission statement, detailed in the form of a series of self contained R & D Actin Packages (APs). The philosophy behind this approach is that it would be difficult, if not impossible, to realize funding for a single large action. By breaking each TTF down into a series of small and realizable APs there exists some potential that these may be independently funded, so allowing the work to commence. The challenge will be to design the APs in such a way that they fit together seamlessly as the pieces a jigsaw puzzle to meet the overall mission of the TTF.

Having established the Program for each of the TTFs EPERC members will receive an Open Call for Partipation to each of the TTFs and its associated APs. These calls will be coordinated by the secretariat but will be implemented via the established national networks.

NON NUCLEAR ENERGY: MATERIALS FOR CLEAN TECHNOLOGIES

CERAMIC CATALYST SUPPORT

This activity studies the degradation of automotive catalyst supports and their associated catalyst systems via accelerated controlled laboratory testing. The results are intended to contribute towards an improvement in operational lifetimes of these environmentally important automobile components.

The catalytic converter for automobile exhaust gases consists of a metallic housing which contains the catalyst material on a metallic or ceramic support. Catalytic activity is conventionally achieved by a γ-alumina washcoat, containing a fine dispersion of an alloy of precious metals (Pt, Rh, Pd), on a highly porous, thin-walled honeycomb structure of cordierite ($2MgO.2Al_2O_{3.5}SiO_2$). This material is chosen because of its low thermal expansion coefficient, good thermal stability and high thermal shock resistance.

Previously, in thermal cycling in dry air, coated cordierite materials have been shown to suffer significant weight losses through spallation and cracking of the washcoat. The weight loss was correlated with the coating thickness. In a further test series, thermal shock has been studied by cycling between a high temperature environment generated by a propane û air burner and an air jet at ambient temperature.

Replicate specimens of four materials (coated and uncoated, thick- and thin-walled variants) were tested up to 1000 cycles at a maximum temperature of 1000°C. To determine the effect of thermal shock, the modulus of rupture (MOR) was measured in four-point bending mode.

The results of residual strength measurements of all materials are summarised in Fig. 54 and Fig. 55. Each material is represented by a single result corresponding to its mean MOR after a certain number of thermal shock cycles. Although there was considerable scatter in the results for individual specimens, as might be expected for this type of porous honeycomb material, as a guide to the scatter, the mean of the estimated standard deviations of each data set was found to be 0.12MPa [range 0.04 – 0.25].

The results of tests from 600°C (Fig. 54) show that there is little, if any, effect due to thermal shock, for a given material. When account is taken of the scatter of the data, it is safe to state that there is no significant trend with increasing number of cycles. After the tests at 1000°C (Fig. 55), however, there is certainly a reduction in residual strength for all materials, between 23 and 36%. Most, if not all of the reduction occurred after the first 100 cycles. Thereafter, the data is not precise enough to deduce a significant trend.

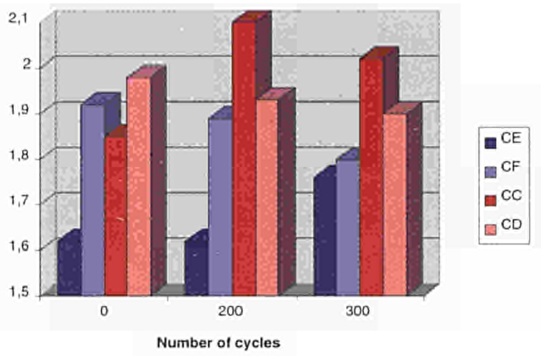


Fig. 54: Mean strength versus number of cycles for all four materials after exposure to thermal shock from 600°C; [CE = thick-walled, uncoated, CC = coated; CF & CD = thin-walled, uncoated and coated, respectively].

It might have been expected that the thin-walled material (CF) would be significantly weaker than the thick-walled (CE). This is clearly not the case and, indeed, there is a more than a suggestion of the reverse, which is maintained even after thermal shock cycling from either temperature. With the coated materials, CD and CC, respectively, there is from this data no such significant difference. In addition, there was no significant difference between the strengths of the two coated variants before testing.

Future work will concentrate on simulated environmental performance testing of conventional as well as novel catalyst support materials.

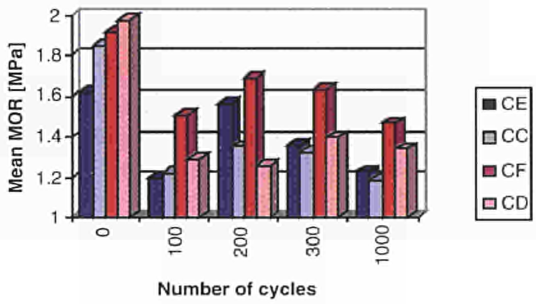


Fig. 55: Mean strength versus number of cycles for all four materials after exposure to thermal shock from 1000°C

MICROSTRUCTURAL ENGINEERING

Catalytic converters for car exhausts operate under especially demanding conditions with respect to temperature range and fuel/air ratio. A wash coat consisting of a γ-Al_2O_3 catalyst support with a dispersed cerium oxide "oxygen storage" component and noble metal (Pt, Pd, Rh) catalyst is generally used to coat a cordierite honeycomb structure. The ceria has a multifunctional role: it acts as an "oxygen storage" component to buffer transient shifts in the fuel/air ratio, it acts to stabilise the γ-Al_2O_3 structure and the noble metal dispersion, and it has a synergistic effect on the noble metal catalysis of reactions such as that between CO and NO. All such catalysts rely for their efficiency on a fine and uniform dispersion of a catalytically active phases on the surface of porous substrate. Nanoporous materials with nanoscale dispersions of the various phases, i.e. nano-nano composites, should be particularly effective for these applications, providing they can demonstrate sufficient high temperature stability. Methods for producing suitable nanostructures are therefore the key to further progress.

With the above requirements in mind, our aim was to find a simple and inexpensive way to make a nanoscale dispersion of cerium oxide in nanostructured alumina (a nano-nano composite). Moreover, the product should also initially consist of boehmite to allow thin coatings to be produced on a suitable substrate, i.e. cordierite honeycomb, by a simple dipping process prior to transformation to transition alumina during calcination. A sol-gel method was used to hydrolyse a mixture of aluminium and cerium salts in the presence of urea. This precipitation method yielded a voluminous transparent gel which was calcined to yield a ceramic powder. The properties of the gel and the calcined product were characterised using SAXS, DTA/TG, XRD, TEM and HRTEM techniques. The effect of the processing parameters, especially the effects of microwave and autoclave treatment of the gel, on the properties of nano-nano composites was the main subject of investigation.

Alumina-ceria powders produced by homogeneous precipitation were shown to exhibit a variety of morphologies depending on the processing route. Microwave drying of the gel produce a true nano-nano composite very suitable for catalysis applications. The structure remained amorphous to about 900°C before phase separation of CeO_2 and the crystallisation of transition alumina. Autoclave treatment at 200°C resulted in the formation of boehmite and the precipitation of agglomerates of $CeOHCO_3$ which resulted in a relatively coarse microstructure on calcination (Fig. 56).

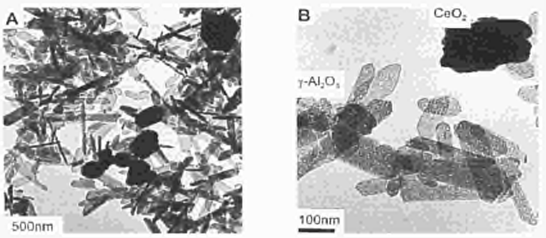


Fig. 56: Autoclave treated alumina-ceria precursor gel a) agglomerates of $CeOHCO_3$ in a boehmite matrix directly after autoclave treatment b) a CeO2 agglomerate in a matrix of γ-Al_2O_3 boehmite pseudomorphs after calcination for 1h at 800°C.

Nevertheless, this material had good thermal stability against transformation to a-Al2O3, resisting 1h at 1200°C. Moreover the presence of boehmite allowed the easy production of thin films by a simple dipping technique. The autoclave treated material therefore is suitable for car catalyst applications whereas the microwave treated material would therefore be more suited for use in pellet form rather than thin films. In principle, the prevention of agglomerates of the Ce phase during autoclave treatment should be achievable which should further improve the potential of the autoclave treated material. Incorporation of nanoscale noble metal catalyst into such materials was also achieved and samples of catalyst coated honeycomb were produced for characterisation.

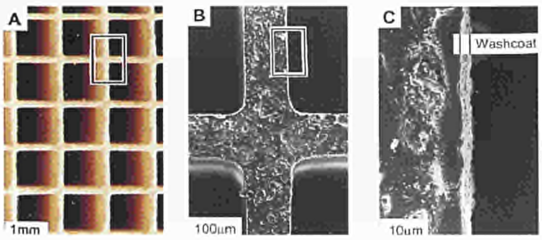


Fig. 57: Cordierite honeycomb coated with a 3 micron layer of alumina-ceria washcoat containing Pt/Pd catalyst. a) 400 channel/square inch honeycomb b) uniform coating c) detail of coating.

SIC / SIC COMPOSITES PROPERTIES

Characterization of SiCf/SiC Composites

The development and characterization of ceramic composites as materials for environmentally friendly energy applications was concluded in 1996.

Thermomechanical Behaviour of SiCf/SiC Composites

Bending tests at 800°C on 2-D SiCf/SiC composites, produced by Chemical Vapour Infiltration (CVI), were carried out for samples annealed in vacuum and in He-atmospheres. Temperatures of 800°C were considered for the isothermal ageing cycles, for 100 hours periods. The helium atmospheres contained residual amounts of oxygen and hydrogen, which could be detrimental for the integrity of the fibre/matrix interface. The results indicate that thermal ageing effects at this temperature on mechanical properties are negligible (Fig. 58). Moreover, SEM fractographic studies did not reveal differences in the fibre pull-out extent in the fractured surfaces, when compared with the 'as-received' condition. The observed differences on the stress-strain curves are more likely to be dominated by morphological features of the specimens, such as the macroporosity (inherent to the CVI matrix processing),or surface defects.

Room temperature thermal diffusivity shows that no relevant modifications of the thermal properties occurred following ageing periods of 100áh and 1000áh with or without traces of oxygen or hydrogen. The thermal behaviour, however, strongly depends on the porosity of the material (Fig. 59). The thermal conductivity data, calculated from thermal diffusivity and bulk density experimental values, were compared with the existing models for multiphase materials, assuming the porosity as third phase of the composite. Fibrous, spherical, and lamellar pore shapes were considered in Fig. 59. All the experimental data fall in between the two curves calculated using two different approaches (porous matrix and porous composite) and assuming lamellar pore shape (F=0.055). This value corresponds to ellipsoids with an axial-ratio of about 12. This result confirms microstructure investigations for which macroporosity can be roughly described with oblate shape, exhibiting a very high thickness- to-width ratio.

<u>*Thermal Properties of SiC-fibres*</u>

A novel three dimensional photothermal method was proposed for the direct measurement of longitudinal thermal diffusivity of fibre type samples. Two SiC single fibres, Textron SCS-6 (Textron Speciality Materials, MA, USA) and AVCO SCS-6 (AVCO, MA, USA) were investigated. The other sample was a Nicalon^{TM} Si-O-C fibre (NLM207, Nippon Carbon, Japan). A composite model was applied in order to arrive at the thermal diffusivity of the CVD-SiC found on the SCS-6 fibres.

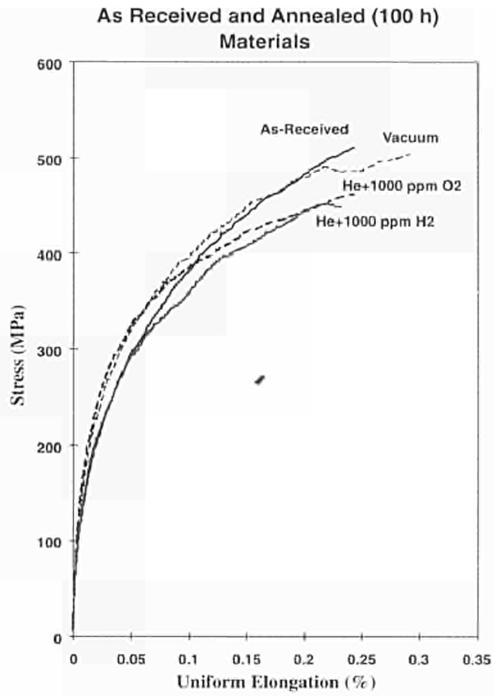


Fig. 58: Bending tests at 800°C on 2-D SiCf/SiC composites after 100 h annealing periods in vacuum and in different He-atmospheres

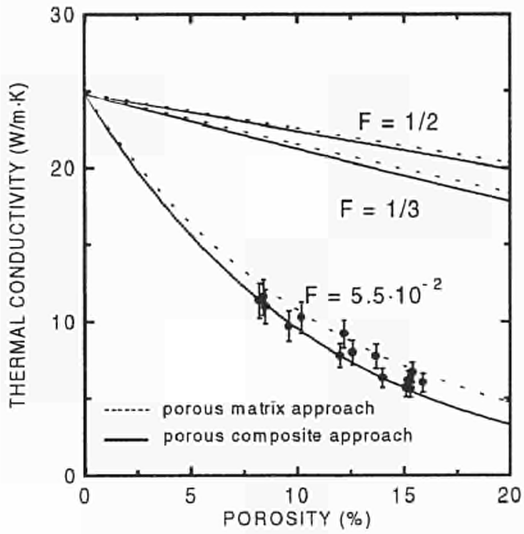


Fig. 59: Thermal conductivity of porous SiC /SiC composite normalised to the thermal conductivity of zero-porosity composite. The F parameter is determining distinct pore shape models.

NON NUCLEAR ENERGY: PHOTOVOLTAIC

PHOTOVOLTAIC ENERGY

The Capacitance Effects in High Efficiency Cells Project

At ESTI, measurements of the Current Voltage characteristics of photovoltaics devices are conveniently performed using flash solar simulators. In certain cell technologies these transient measurement techniques can lead to capacitance effects which complicate the accurate measurement of the performance of these high efficiency solar cells. Because of increasing quality assurance requirements and the growing importance of high-efficiency cells, the understanding of this phenomena is essential for the correct calibration of photovoltaic devices. In the Research Project "Capacitance Effects in High Efficiency Cells" a new method (Photo-Current-Response (PCR) -method), to measure the light induced capacitance in high efficiency solar cells was developed. The capacitance is induced by a fast pulsed LED-array. Effective capacitance measurements in dependence of bias voltage, light intensity and temperature can be made. To investigate the temperature behaviour of the capacitance effect it is necessary to reach temperatures from 100°K to 400°K. These device temperatures are achieved by a combination of cooling the device fixture with liquid nitrogen and heating with oil. At temperatures below room temperature (77-300°K) moisture condensation will occur on the surface of the probe. Such condensation usually provides a current leakage interfering with the reliability and accuracy of the measurements. This is avoided by keeping the probe station dry, which is done by putting the probe station of the PCR set-up under vacuum. For this, high vacuum conditions of approximately 10^{-5}mb are sufficient.

The photograph (Fig. 60) shows the vacuum chamber developed at ESTI. By putting a solar simulator in front of the view port, the vacuum system is also used for V_{oc} or I_{sc}-characterisation of solar cells at low temperatures

Standardisation

Qualification Specifications for Thin Film Modules

During 1996 the ESTI sector of the EST Unit has modified and revised the working practices for the qualification and type approval testing of amorphous silicon thin film Photovoltaic modules. This work building upon both internal research and industry driven demand for an acceptable and recognisable international standard for the qualification and type approval of commercially available thin film modules has led to renewed interest by prominent thin film Photovoltaic producers. The fruits of this effort on the definition of testing and qualification procedures has already been seen in 1996 with the confirmed order for a full qualification test sequence and requests for several others at an advanced stage of preparation.

In addition following the initiative of the EST unit, a proposal has been submitted to the JOULE III programme by a consortium led by EST to redefine the existing internal test procedures (known as CEC 701 specification) and to extend this to cover not only amorphous silicon thin film materials but also the new and exciting developments in the fields of Cadmium Telluride, Copper indium di selunide and multi junction structures . This proposal has been accepted for funding by the JOULE Committee, and with its combination of leading European manufactures in thin film technologies, combined with the experience in standard preparation and testing procedures of the EST Unit, is set to become an exciting and dynamic development for testing laboratories and thin film produces around the world.

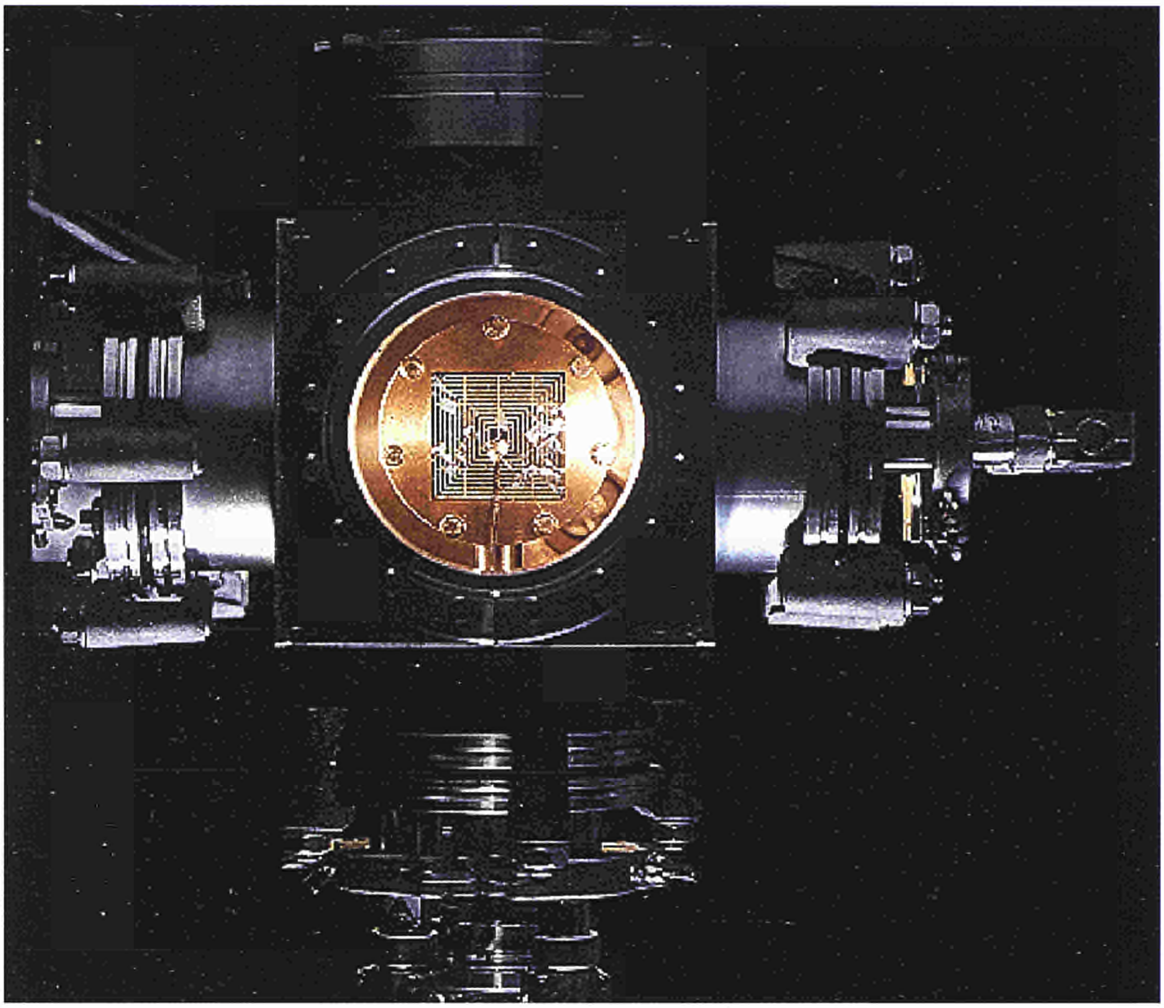


Fig. 60: Vacuum chamber used for characterisation of solar cells under illumination at low temperatures at the ESTI laboratories.)

<u>*Preparation of IEC standard for photovoltaic Systems*</u>

As one of the spin-offs from APAS project 'ElectricHome' ESTI together with three European partners submitted a THERMIE B proposal to the European Commission which aims to develop a standard for stand-alone photovoltaic systems for non-electrified areas.

The overall objective of the proposed project is to develop and promote global draft standards for the installation and integration of stand-alone photovoltaic (PV) systems in non-electrified houses in Europe and elsewhere as a follow-up of a research project supported by the EC.

These standards which will be developed in close cooperation with representatives of international standardisation organisations will help to lift photovoltaic components and systems to a higher level of quality and reliability and make PV a serious alternative to diesel generators or grid-connection.

Enhanced deployment of reliable and high-quality PV systems in isolated and rural areas in Europe will help to raise the quality of life closer to the standard of urban settlements. It has the potential to stop or even reverse rural exodus and thus significantly improve economic and social cohesion.

This planned initiative provides advice to the European PV industry (mainly SME's which dominate this sector) how to improve the quality of PV systems and components and to increase their industrial competitiveness. This initiative also provides advice to end-users on the technical requirements to be demanded for PV systems on remote locations. Furthermore, the deployment of RE and in particular PV at a wider scale provides significant market opportunities for local SMEs acting as suppliers or distributors of PV systems.

ENERGY MONITORING IN BUILDINGS

Monitoring Energy Performances of the EcoCentre Buildings.

To evaluate the energy retrofits of the ECOCENTRE buildings it is necessary to monitor the behaviour of all single components and how they influence the global energy consumption. The buildings, object of careful monitoring during the 1996, are the "Mensa" (building 8) and the "Technological Hall" (building 45).

The existing monitoring system was extended in order to give greater accuracy of the expected results and supply data to the Infrastructure Unit for the final acceptance of the works. In building 45 and in building 8 the monitoring system is now able to provide hourly, daily and monthly consumption of heating, cooling and electrical energy. It is also possible to view daily internal and external temperature and relative humidity trends. This has been made possible through dedicated software running on a PC connected to the local PCs that perform the data acquisition. Extra data analysis allows evaluation of the energy performance of each single building component.
The main result of this activity is the development of monitoring and tele-monitoring techniques and expertise, as well as procedures for acceptance of a retrofitted building based on the measurement of its energy performance.

Assistance has also been given to checking the environmental conditions of building n.8 (the Mensa building) in relation to the settings of the regulation system. Comfort problems associated with incorrect operation of the regulation system have been detected and reported.

Environmental Indicators for the Ispra Centre.

Without a suitable definition and monitoring of environmental quality indicators, for an area where environmental retrofit actions are carried out, it is not possible to demonstrate scientifically the improvements made by the project. For this reason a first study was carried out during 1996 (see F. Conti, 1996, EUR 16404 EN) in order to identify a set of environmental indicators applicable to the area of the Ispra research establishment. After having reviewed the environmental parameters used in other urban situations, 7 areas of environmental rehabilitation actions appropriate for the Ispra research centre, grouped into 3 broad categories, have been identified. These 3 categories are: Consumption of natural Resources (Energy, Water, Ground, Air), Social Involvement and Human Behaviour (Management & Funding, Transport & Mobility, Staff Involvement, General Information) and Biodiversity (Flora & Fauna).

Different types of indicators have been defined:

- ***global indicators***, which monitor the overall area situation (e.g. the total consumption of primary energy or the global emission of air pollutants from the Centre),

- ***partial effect indicators:*** referring to previous example on energy, indicators could be defined for levels of consumption of heating, cooling and electricity;

- ***targeted indicators,*** to monitor the development of the situation concerning a particular action (e.g. the recovery of green areas, the recycling of used paper, the purchase of electric or low emission cars, or the improvement of use of the water treatment plant, etc.),

- ***specific indicators,*** which connect the performance as a function of the specified user (e.g. standard performance with respect to the number of people served, to the area of floor heated, etc.).

Due to the large number of indicators for different categories and types, they have to be reduced to a few meaningful and readable number of indices so that environmental progress in various fields can be quickly highlighted.

The choice of an indicator implies expenditure for data collection and calculation time. Therefore, particular care has been taken in order to propose indicators that can be quantified by means of data already collected for other reasons and not linked only to the environmental monitoring of the centre. The sub-project of starting the collection and evaluation of environmental data for the Ispra campus will be discussed in the 1997, after specific presentation and debate of the environmental indicators between the widest number of interested people from the Centre.

REACTOR SAFETY COMPONENT AGEING AND INSPECTION

NESC

Introduction

The NESC Network has been conceived to integrate the different technical constituents of structural integrity, namely, materials characteristics, inspection, fracture mechanics, stress analyses and integrity analysis. Hence this European network assembles, in very topical projects, the experts of the different disciplines or branches of structural integrity essential for the safety of nuclear power plants. These experts participating actively to the projects of the network are representing industry (manufacturers, engineering companies, utilities), service vendors, regulatory authorities, R&D instituti/ns in support to reactor safety and regulators.

The first NESC project is managed by AEA Technology and is based on the problem of the pressurised thermal shock (PTS) in a reactor pressure vessel. The vessel is represented by a cylinder with end of life material characteristics and the PTS is simulated in the Spinning Cylinder Test Facility at the Risley laboratories of AEA.

The Steering Committee of NESC develops also other projects to be launched as a function of the contribution in kind that can be given by members and of the financing that can be found.

The NESC 1 project

During the past year the project has continued to be faced with unexpected problems. Through the determined resolve of the Project and Network management team combined with the excellent international co-operation and good will engendered by all the members, the agreement to finally go ahead with the Spinning Cylinder test could be achieved within the Steering Committee. The main activities of 1996 were as follows:

Cylinder preparation

During the qualification phase of the as manufactured cylinder, and prior to the round robin inspection trials an unexpected complexity of the through clad defect was discovered. Although this adds co.siderable inspection challenge to the project, it was decided that this could no longer be considered as the key through clad defect in the cylinder. This defect was so complex in shape that it could not be accurately sized and consequently no structural mechanics assessment could be carried out. In view of this and the lack of a reasonable comparison with the behaviour of the underclad defect, it was decided to attempt to introduce a new through clad defect after the inspection round. A semi-circular EDM notch, typical in shape but much larger than those used for ISI inspection exercises, was inserted by the Reference Laboratory, JRC/IAM (Figure ?). This was subsequently fatigue sharpened by AEA Technology in order to increase the chance of crack growth during the test.

Task Group 1 – Inspection

The inspection of the cylinder had started as expected in December 1995 at TRC/ABB Sweden. All necessary guideline documents were prepared by the Reference Laboratory both for the handling and inspection of the cylinder as for the reporting. The inspection continued until the end of June with teams from

Finland, Russia (inspection in Finland), France, United States of America (inspection at JRC Petten), United Kingdom and Germany taking part. Invigilation of these inspections as well as the analysis of the results were performed by the Reference Laboratory to communicate the range of measurements of five of the detected defects to the Structural Analysis Group in order to enable the pre-test fracture mechanics calculations.

Task Group 2 – Materials

The group, constituted from 7 mechanical testing laboratories from the United Kingdom, Germany, France, Italy, the Netherlands, Finland, organised and executed a complete materials characterisation programme. Much additional work had to be carried out to carefully characterise the Heat Affected Zone (HAZ) beneath the cladding which had not been considered in earlier evaluations but would play a crucial role in determining the propensity for crack growth

Task Group 3 – Structural analysis

From the pre-test calculations, the group had to advise on optimum test conditions for the Spinning Cylinder test but first they had to evaluate whether there was any possibility of crack growth from any of the defects under the most extreme test conditions possible. This doubt arose from the high ductility of the HAZ which had been measured. The defects were expected to grow lengthwise from their extremities, and the maximum crack driving force was situated just in this HAZ region and across the interface to the base metal. Without the additional HAZ ductility, the test conditions were already approaching the limit to facilitate crack growth. Although many alternatives to modify the test conditions such as strip cooling, undercooling the coolant etc. were presented and discussed, it was finally agreed that crack growth could be anticipated from at least the large defects under the thermal and mechanical stress systems which could be achieved in the Spinning Cylinder commissioning trials. Some 16 organisations carried out the calculations to support this and final agreement was obtained by the NESC Steering Committee at the end of the year.

Task Group 4 – Instrumentation

While the Structural Analysis group were deliberating on the chance of a successful outcome of the test, the Instrumentation Task Group carried on with the instrumentation plans of the cylinder.

New NESC Projects

NESC was inaugurated as a Network not a single project, hence, in parallel, the NESC Network management has devoted considerable effort to assist in realising successors to the NESC I project. Two routes have been followed to this end, the first utilising fully defined projects (NESC II, PTS simulation)and the second seed projects around which full scale projects can eventually be built (Shared Cost Actions such as BIMET).

AMES

Introduction

Within the European Network AMES, Ageing Materials Evaluation & Studies, a large number of experimental studies on ageing, both thermal and irradiation induced, and annealing are carried out.

Most of the activities are international projects involving several European Member States laboratories and several of the mentioned activities are financed as SCA competitive projects by the Nuclear Fission Safety Programme of the EU.

Large parts and tasks of these projects are carried out in Petten by the so called "AMES Reference Laboratoryö; a joint effort of IAM and ECN in order to provide all the necessary facilities and tools required to conduct material ageing studies on both un-irradiated and irradiated samples.

General objectives

The general objectives of the AMES Reference activities are given in Table IV.

	Objectives:
a)	Carry out experimental study on ageing/annealing **thermal ageing** **irradiation embrittlement**
b)	Setting-up develop and maintain: **thermal ageing laboratory** **irradiation facility form HFR** **certified impact testing laboratory, etc.**
c)	Collect Reference Materials **reference steels (JRQ, HSST etc.)** **materials coming from aged industrial structures and/or private organisation (GKSS irr.mat.)**
d)	Maintain & develop competence in: **neutron dosimetry** **material damage** **indexation, etc.**

Table IV: GENERAL OBJECTIVES of the AMES Reference Laboratory projects

Further developments

In order to further improve the Petten site capability in this field, particular emphasis is given to develop new dedicated unique facilities. Just as unique example, a completely new type of irradiation rig has been developed for the very high demanding AMES requirements and specifications. The new irradiation facility for the HFR, called "LYRA" is a re-loadable, located at the Pool Side Facility, PSF. A system of γ-heating shields are designed in order to minimise the thermal gradients and the samples target temperature is maintained by means of a complex system of independently controlled heating plates. The rig temperature range is between 200 to 450 °C. The required fluence levels typical of RPV end-of-life can be obtained in irradiation time of the orders of 6-8 weeks. A space of up to 59x64 mm is available to accommodate sample holders with different loadings for a maximum length of sample column of up to 350 mm. As many as 140 Charpy V-notched samples (10x10x55 mm) or 10 CT specimens can be loaded. The necessary instrumentation is provided in order to demonstrate the achievement of the irradiation requirements; including sufficient number of calibrated thermocouples distributed between the samples, a sufficient number of flux detectors distributed between samples, and Self Power Neutron Detectors, SPN's, one in front and one on the back of the sample holder) in order to verify the fluence rate gradients and to be used, after calibration against the flux detectors, as fluence indicator for the following irradiation experiments.

Activities

The list in Table II represents a short summary of the most important activities and projects for which an important share of the work is carried out at Petten and that can be considered as spin offs of the AMES work.

Project name	EU contract number	Objectives & **partners with JRC-IAM**
AMES DOSIMETRY	NFS SCA PL950011	Harmonise dosimetry practices for ageing studies and establish the dosimetry of AMES activities **Tecnatom, ECN**
REFEREE	NFS SCA PL950073	Assess the correlation between different fracture toughness properties of aged steels; Charpy impact versus dynamic & quasi-static toughness transition shifts measurements **NE, SCK/CEN, VTT**
RESQUE	NFS SCA PL960344	Validation of CV-n sample re-constitution techniques for obtaining more experimental fracture tougness data limiting the amount of material used. Different welding & joining techniques are compared. **SCK/CEN, ECN, VTT, NE, AEA, RCR, Siemens**
SYNTER	NFS TN PL960346	Propose and study safety related innovative nuclear reactor technology elements for present and future type of plants **KFA, CEA, ECN, VTT, ENEA, ENEL, PSI**
MADAM	NFS TN PL960395	Generation of a possible conversion table of material damage indexes for possible comparison of results coming from different test programs and real operating plants **Tecnatom, VTT, NE, ECN, SCK/CEN**

Table V:Brief description of SCA financed projects relevant with AMES tasks.

ENIQ

The European Network for Inspection Qualification (ENIQ) groups the major part of the nuclear power plant operators in the European Union (and Switzerland). The main objective of ENIQ is to co-ordinate and manage at European level expertise and resources for the qualification of NDE inspection systems, primarily for nuclear components.

One of the important activities of ENIQ in 1996 has been the discussion of the second issue of the European Methodology for qualification of non-destructive tests. The approval of the document is expected for the beginning of 1997. It was decided to issue a second issue in order to take into account the many recent developments in this field, induced by, among others, the publication of the first issue in 1995. This document is in good agreement with the common position document on qualification of NDT systems for pre- and in-service inspection of light water reactor components, prepared by the Nuclear Regulator's Working Group, sponsored by DG XI. This shows that in the EU there is not only agreement among the utilities but also between the utilities and regulatory bodies on the general principles for inspection qualification

Many EU countries are already implementing the general principles of the European methodology in their national qualification programmes. Furthermore the International Atomic Energy Agency is preparing guidelines for inspection qualification for VVER operating countries. These IAEA guidelines take into account the European methodology, as proposed by ENIQ. This is very important because inspection qualification is considered as one of the important tools to improve the safety of nuclear power plants in Eastern Europe. That is why, as already mentioned before, ENIQ is having regular contacts with relevant institutions from Eastern Europe in order to exchange information.

The Steering Committee of ENIQ has decided to conduct a pilot study to explore ways of how to apply the European methodology for inspection qualification to a specific component.

The objectives of the ENIQ pilot study are:

- to apply the general principles of the European methodology to a specific component in order to explore ways how to apply them
- to verify the feasibility of the principles of the European methodology

The example chosen for the ENIQ pilot study is the qualification of an inspection of austenitic pipe to pipe and pipe to elbow welds. All aspects of the inspection are qualified. The procedure and equipment qualification involve open trials on test pieces containing defects while that of the personnel is done through blind trials. In addition to practical trials, qualification involves also the production of a technical justification as required by the methodology document.

The inspection procedure under qualification is an automated one involving a scanner and digital flaw detector. The inspection procedure was produced specially for this exercise and is tailored to the particular requirements of this inspection.

Qualification involved a combination of satisfactory practical trial results and a convincing technical justification. If qualification revealed shortcomings in some aspect of the inspection, modifications were made and the qualification was repeated.

Once the inspection has been qualified, it will be applied in 1997 to a number of "real" components, some containing defects removed from operating reactors and others containing simulated defects but welded using the same materials and procedure as the qualification test pieces. The results obtained will be compared in detail to those in the first qualification part of the pilot study. From this comparison, conclusions will be drawn about the value of qualification in providing confidence in the inspection.

The status of the pilot study as it stands end of December 1996 is as follows. All necessary documents have been prepared (inspection procedure, technical justification, qualification procedure, etc.). The practical trials start beginning of 1997 and are expected to be finished mid next year. The full results of the pilot study should become available end of next year.

Despite the fact that the pilot study is not finished yet there are already a number of lessons learned from the pilot study.

The Steering Committee of ENIQ agreed to set up a Task Group on Risk Based Inspection at its meeting in April 1996 in Madrid. The general objective of this task group is to study aspects of ISI or any other surveillance method in view of both a more selective application and optimisation in order to reduce the inspection efforts whilst at the same time increasing the ISI effectiveness. Actions have been decided for gathering and transfer of information on the different aspects of risk based inspection and, possibly at a later stage, writing of a " European methodology" document on RBI. The organisational framework of this task group is determined by the ENIQ agreement:

1. utility driven
2. linked to ENIQ but might evolve into a separate network
3. narrow contact/co-operation with the regulators welcomed
4. European core

O.V. Chapman from Rolls Royce and Associates was nominated as chairman of this Task Group. S. Crutzen acts as co-chairman of the Task Group.

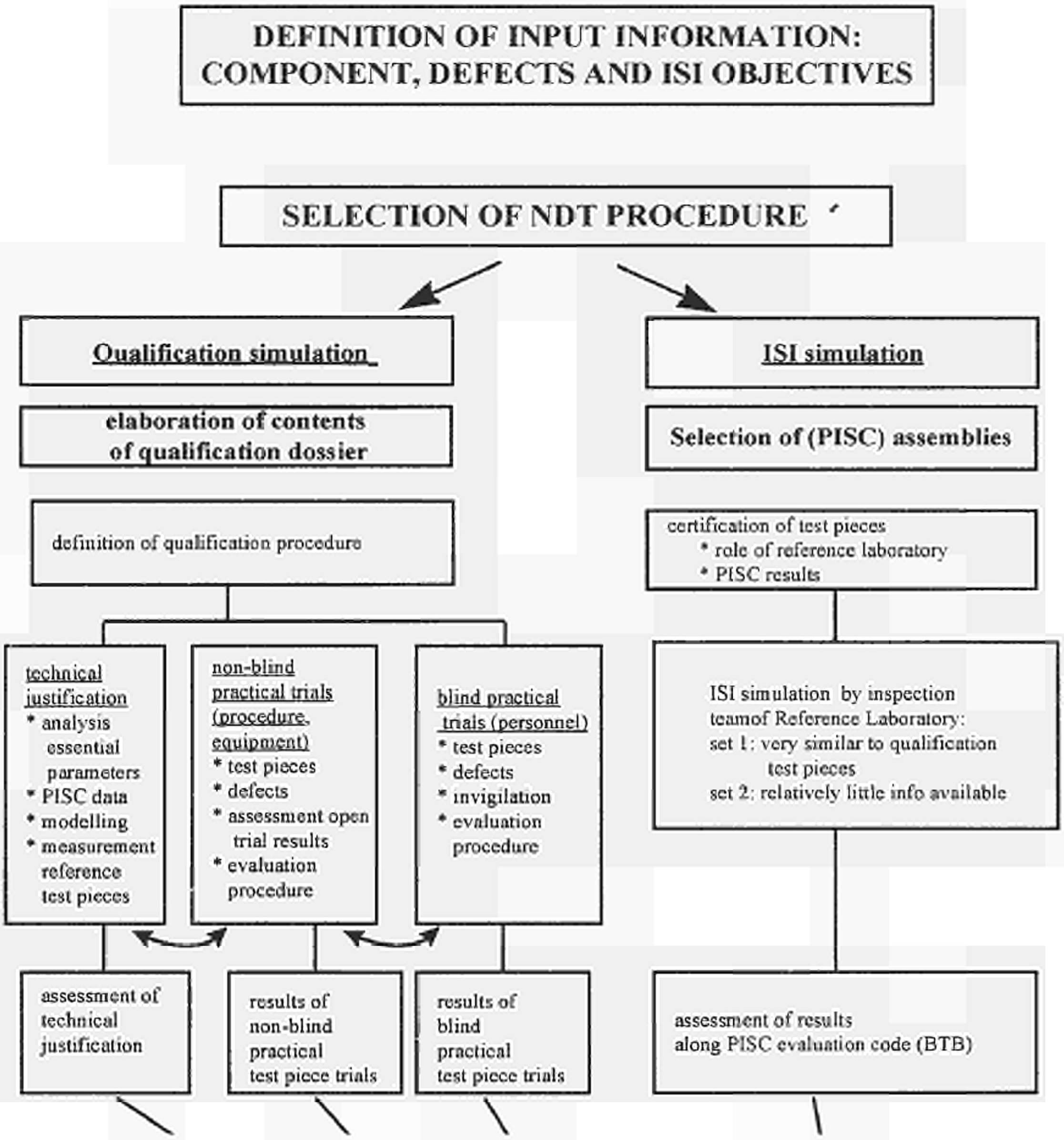


Fig. 61: Schematic overview of the ENIQ pilot study

NETWORKS IN SUPPORT TO COMMISSION SERVICES

Through the European Networks ENIQ, AMES and NESC, JRC as Operating Agent and Reference Laboratory is collaborating with DG XI.C2 to reach consensus on several technical issues regarding Nuclear Installation Safety. In this sense, JRC IAM is also an ideal organisation to support, help to manage or even execute projects of technological character required by the DG XI Working Groups.

DGXVII pursues activities in view of the development and better acceptance of the nuclear industry in Europe as well as for its competitiveness compared with the United States and Japanese industry. In this context since 1992, DGXVII has supported the PISC activities (Programme for Inspection of Steel Components) managed and executed by the JRC/IAM. A logical continuation of some PISC activities is the ENIQ network programme.

The networks developed and operated by JRC/IAM have the following broad objectives:

- the integration of fragmented R&D work on structural integrity through the execution of studies and projects at European level
- the support or introduction of a long term strategy in some of the European groups or actions conducted by the Commission
- the use of European networks influences studies and project results in the direction of codes and standards in Europe and for the harmonisation of codes in general

The following results have been reached in 1996 apart from:

- technical and scientific advice during the different meetings (playing the role of Reference Laboratory for the round robin trials on austenitic fast breeder welds organised by AG1 which includes also evaluation of the obtained inspection results and reporting
- taking the minutes of the WGC&S, AG1-2-3 meetings

ENIQ

JRC Petten is providing support to the Working Group Codes and Standards (WGC/S) Activity Group 1 (AG 1) active in the field of inspection and manufacturing standards.

During 1996 discussions were held within ENIQ in order to come up with a second issue of the European methodology. This second issue is in good agreement with the "Common Position" document of the NRWG Task Force, supported by DG XI. It shows that at EU level there is not only agreement between the different utilities themselves but also between utilities and regulators on the general principles of inspection qualification. The publication of this second issue as an official EUR-report is expected for the beginning of 1997.

The NRWG Task Force on qualification, supported by DG XI, is also following the ENIQ pilot study conducted to show the feasibility of the European methodology. The pilot study is also considered as a test for the common position document of the NRWG's Task Force. Documents for the pilot study and the inspections were prepared. Two progress meetings were held with representatives of the NRWG Force on inspection qualification in order to report on the progress of the work done.

ENDEF

A second meeting was held in Moscow and a third in Petten with Russian and Ukrainian representatives from authorities and industry to present and discuss the strategy to implement an effective in-service inspection system for VVER and RBMK reactors. The strategy paper had been developed within ENDEF in previous meetings. The Russian and Ukrainian representatives supported the work and were ready to continue to cooperate on this subject. A first meeting was held in Brussels with Phare beneficiary countries to present and discuss the ENIQ and ENDEF activities and their relation to these specific countries.

A comparison between the steps in the strategy document for the implementation of ISI qualification and the ongoing Tacis, Phare and bilateral projects was carried out.

AMES

JRC Petten is providing support to the Working Group Codes and Standards (WGC/S) Activity Group 3 (AG 3) active in the field of materials.

Three further reports have been published by AMES as EUR reports for the widest distribution: 'A Review of Formulas for Predicting Irradiation Embrittlement of Reactors Vessel Materials – EUR16455EN', 'The AMES Reference Laboratory – EUR16409EN', 'Dosimetry and Neutron Transport Methods for RPV's – EUR16470EN'. These studies were also supported by AG3.

NESC

JRC Petten is providing support to the Working Group Codes and Standards (WGC/S) Activity Group 2 (AG 2) active in the field of fracture mechanics. The NESC1 project has become a focus of activities in the frame of structural integrity and is now connected to AG2 studies.

The structural integrity assessment procedures required for NESC1 rely on the successful transfer of small scale specimen data to large structures. WGC/S has funded a small generic project to establish the state of the art in terms of transferability and to highlight those aspects relevant to NESC1.

INFORMATION MANAGEMENT AND TRANSFER

European Networks Advanced Information System (ENAIS)

The European Networks Advanced Information System continued its professional support for the transfer of information generated by the networks PISC, AMES, NESC, ENIQ and EPERC to industry, codes and standards and other fields of R&D. Numerous actions were undertaken to divulge the know-how to the interested bodies via modern communication systems like INTERNET.

Project descriptions, progress and results are progressively displayed on the World Wide Web as well as calls for participation and registration to specialised groups of external initiative.

Such a transfer of information obviously implies advanced information handling and treatment both for the analysis of results, and their presentation and publication. Codes were developed or adapted and applied in support of data evaluation, project management and quality certification. Data bases of experimental data are further elaborated and kept available for future studies as the result of a duty put on the JRC by the CSNI (OECD) after the closure of PISC.

ENAIS continued in 1996 to evaluate NDE results from inspection exercises and RRT's, mainly in the frame of austenitic steel components inspection.

Spin offs

Evaluation procedures of NDE data, codes adapted or developed and the use of up-to-date information tools lead the ENAIS sector to elaborate several Shared Cost Action proposals. The contract on Neural Network support for Non-Destructive Evaluation, obtained from the programme VALUE in 1995, was rounded-off successfully by the end of 1996.

In support to SINTAP (Structural Integrity Assessment Procedures for European Industry), ENAIS helped to provide information on statistical treatment of NDE data as well as guidance on issues relating to the interaction between NDT and fracture mechanics assessment, including probability of detection and sizing errors. To this end, it made use of examples of what can been obtained from existing PISC databases.

CONTROLLED THERMONUCLEAR FUSION

FUSION MATERIALS

Thermal Fatigue Component Testing

In agreement with the NET and ITER teams, the Thermal Fatigue Test Facility (TFTF) at IAM-Ispra is operative for testing and validation of first wall and shield blanket specimens in support of ITER's Technological Needs for the selection and characterization of Material through ITER related testing. The activities focused on the completion of the current mock-up testing campaign and the initiation of a facility upgrade to adapt the system to new NET/ITER requirements,

Testing of CuCrZr-stainless steel 316L explosion welded first wall mock-up

The mock-up was manufactured by VTT, Manufacturing Technology, Materials and Structural Integrity (Finland) and consisted of a block of Cu-Cr-Zr (dimensions 163x100x40 mm) in which 6 cooling channels were introduced along the block length. Three steel plates, having a total thickness of 5 mm, were explosion welded one after the other onto the surface opposite to the heated one. No additional heat treatment was carried out after the manufacturing process. A few micron thick black-chromium coating was deposited onto the heated surface by means of a galvanic process to increase the absorptivity and thus to increase the heat flux effectively absorbed by the component. Plate to plate and tube to plate interfaces were examined by ultrasonic inspections at VTT.

The component was subjected to thermal fatigue for 13 000 cycles at a maximum heat flux of 0.57 MW/m^2 and cycle duration of 117 s. A comparison between numerical and experimental results showed that the measured temperatures were appreciably higher than those numerically computed. This could be explained assuming that the cooling tubes were not properly bonded all along the component length and that thus the thermal resistance of the joint interface was appreciably higher. There were no indications that the debonded regions should not have grown during the test. The mock-up could complete the foreseen number of cycles without the appearance of any macroscopic defect. Therefore, the defect tolerance of this concept proved to be quite high.

Upgrade of the Test Facility for ITER-needs

NET/ITER requirements for testing the next generation of mock-ups necessitate an upgrade of the TFTF to meet the following needs:

- Testing of multilayer mock-ups containing beryllium
- Testing with coolant temperature of 140á°C and coolant pressure of 40 bar

Special filter and ventilation systems are being installed in order to handle Be mock-ups. The cooling circuit is being modified to the test conditions mentioned above for long term thermal fatigue tests (13.000 cycles).

Dispersion strengthened (DS) Cu/stainless steel and Be/DS-Cu/stainless steel multilayer mock-ups are being prepared for the next test campaign in 1997 (Fig. 62). They will be tested at heat fluxes of about $0.8MW/m_2$

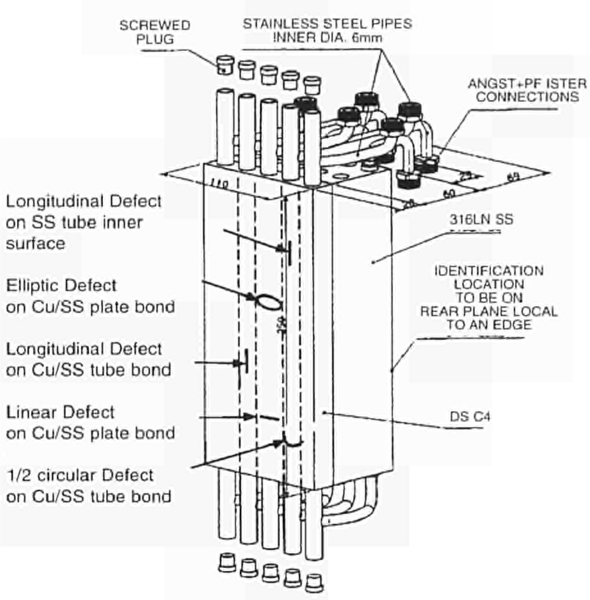


Fig. 62: Dispersion strenghtened copper / stainless steel multilayer specimen

In-Pile Thermal Fatigue: Project FAFNIR

The major objective of the FAFNIR project is the measurement and the modelling of crack propagation in cyclic thermal gradient fields in a multiaxial stress state with and without simultaneous radiation damage. The project is performed in collaboration with HFR Unit, IAM-Petten,

The project output will extend the data base necessary for the design of ITER first wall to more realistic conditions, in particular with the assessment of the effect of simultaneous neutron irradiation on the crack growth (thermal fatigue/irradiation creep interaction). Data obtained from thermal fatigue of first wall components at IAM-Ispra the will be taken into account in the present project

The construction of the in-pile rig is in progress. The measuring equipment for the measurement of the crack growth propagation (ACPD Technique) has been improved and completed.

An agreement with the ITER Home Team has been reached on the necessity of testing the in-pile experimental facility with 316 LN steel in order to complete the Thermal Fatigue Testing of Small Mock-ups already done at the IAM-Ispra.

Fusion Materials Data Bank

Materials Properties Handbook and Interim Design Criteria for ITER

The objective of this ITER-task is the collection and evaluation of data from the R&D programme for the ITER Materials Properties Handbook, which is aimed to be an important tool for the design and the manufacturing of ITER.

The EU fusion material data bank, after having being improved for the ITER-EDA, is constantly supplying data to the ITER-EDA MPH in the format agreed among the different partners. As required by the design group during this period fracture toughness data on irradiated AISI 316L (IG) have been collated and evaluated.

The collection and evaluation of data coming from different laboratories on fatigue, creep and fracture toughness of AISI 316 LN (IG) steel with particular emphasis on welded material has been completed. Data on cryogenic steels are included according to their availability.

A revision of the data, in terms of quality, will start with the ITER partners, i.e. USA, RF and Japan.

Low Activation Materials

Characterization of SiCf/SiC Composites

The development and characterisation of low activation ceramic composites for the Long-Term Technology Programme has been continued in 1996.

Thermomechanical Behaviour of Irradiated SiCf/SiC Composites

Bending tests (in helium at 800°C) of neutron irradiated 2D SiCf/SiC composites were performed. Degradation in strength occurs with increasing fluence (Fig. 63), due to fibre densification (Fig. 64) which leads initially to an improved damage tolerance. (1ádpa) and progressive embrittlement of the composite for 2 and 5 dpa. Considering the dpa neutron irradiated specimens and comparing with the results shown in Fig. 65 for He-implanted samples it can be stated that reduced strength induced by He is caused by displacement damage which is enhanced by distinct He-retention and induced volumetric changes of the ß-SiC-matrix and the Si-C-O fibre. As shown in Fig. 66, a significant thermal conductivity degradation is observed during irradiation. A steep decrease occurs at rather low doses and without further reductions at higher doses.

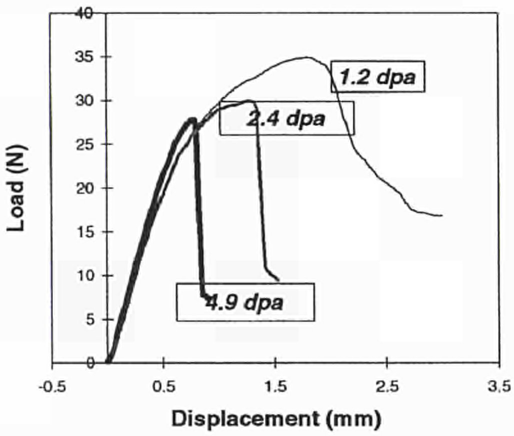


Fig. 63: Force-displacement curves for neutron irradiated SiCf/SiC composites. The attained fluences correspond to damage levels of 1.2, 2.4 and 4.9 dpa, respectively.

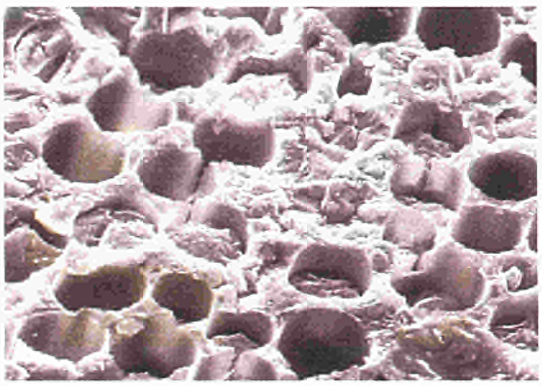


Fig. 64: SEM image of neutron irradiated (1.2 dpa). Lateral surface perpendicular to the 2D fibre plane.

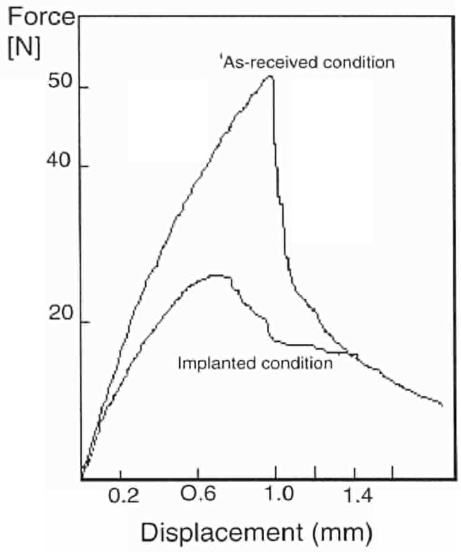


Fig. 65: Force-displacement curves for 'as-received' and for He-implanted SiCf/SiC composites. Implanted doses were around 2500 appm

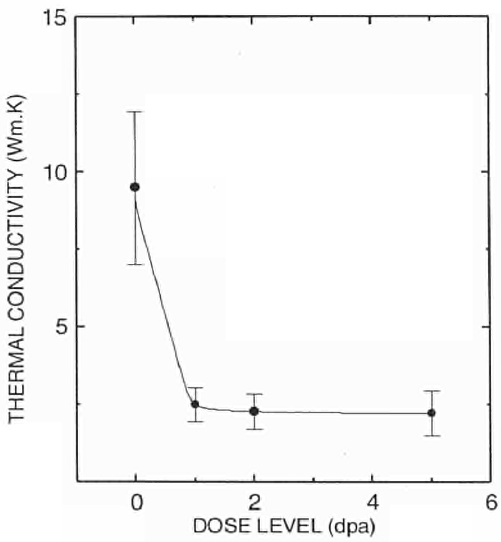


Fig. 66 Thermal conductivity of neutron irradiated SiCf/SiC composite for different fluences

Irradiation creep of SiC Fibres

Creep tests were conducted during irradiation with 14áMeV deuterons at 600á°C on TEXTRON SCS-6 fibres which consist mainly of stochiometric ß-SiC. The fibres are produced by applying a CVD procedure and have a structure which is similar to the one of the matrix of SiC composites produced by chemical vapour deposition. The irradiation creep curves can be rationalized by assuming that the total creep strain is composed of two components, a transient component and a steady state component. The transient component increases slightly with the applied stress, while the steady state component is a linear function of stress for shear stresses ageing from 75 to 300 MPa.

Next Steps

New 3D- SiCf/SiC (Guipex™) will be characterized in the framework of the TAURO blanket design. Studies after He-implantation and neutron irradiation will be performed. In a collaborative effort with ORNL (USA) the influence of He-implantation on thermal diffusivity and electrical resistivity of polycrystalline CVD-SiC bars will be investigated.

Non Ferrous Alloys

The thermal and mechanical properties of high-purity chromium (Ducropur) and the chromium alloys Cr 5Fe $1Y_2O_3$ and Cr 44Fe 5Al 0.3Ti $0.5Y_2O_3$ are being determined in order to assess the potential of these materials for use as heat sink and structural materials in plasma facing components and first wall structures. The materials were produced by the Metallwerke Plansee using a sintering process. The studies described here are strongly related to those reported in the 'Brittle Materials' project.

Thermal diffusivity measurements were performed using the laser flash method. As it is illustrated in Fig. 67, the thermal diffusivity of pure chromium is rather high which is favourable with regard to thermal stresses induced by transient thermal loads, especially if the low thermal expansion coefficient (about $6x10^{-6}$ 1/K at room temperature) is taken into account. Tests for determining the thermal expansion coefficient in dependence of the temperature are currently performed.

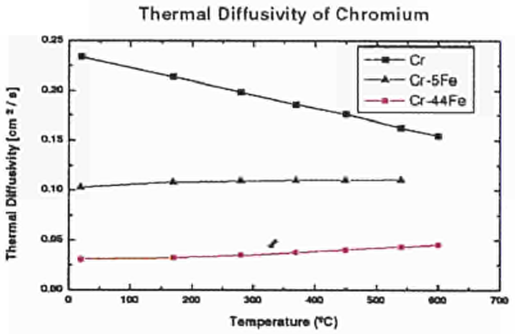


Fig. 67: Thermal diffusivity as a function of temperature for Ducropur and the alloys Cr 5Fe $1Y_2O_3$ and Cr 44Fe 5Al 0.3Ti $0.5Y_2O_3$

Tensile tests were carried out for the alloy Cr 5Fe $1Y_2O_3$. In the temperature range up to 100°C very brittle behaviour is observed. At higher temperatures i.e. 300á°C and 400°C total elongations of about 2%-4% and 12%-14%, respectively, are observed. As in the case of Ducropur, fracture mechanical tests will be performed to assess the ductile-to-brittle transition temperature.

Low Temperature Irradiation Creep Studies on Stainless Steel

The low temperature irradiation creep studies with the aim to obtain relevant data for the ITER design have been continued in the HFR.

Exp. E167-80

Low temperature irradiations of reference steels 316L, US 316 at 370 K are performed at stresses of 70 and 100 MPa. Elongation measurements were performed after 0.1, 0.4, 1.5, 2, 2.5, 3 dpa. The total neutron fluence attained corresponds to 3.8 dpa and elongation measurements at this dose are in progress in the hot cells.

Exp. E167-90

Low temperature irradiations at 370 K, on welded specimens, namely on EB-welds and on TIG welds are performed at damage rates of 0.12 dpa per reactor cycle (25 days).

EB-weld materials are being irradiated only in the as-received state. TIG weld materials are being irradiated in the as-received state and after annealing at 1073 K. The E167-90 rig contains also specimens of 316 L steels and of AMCR type materials with certain additions. Stresses of 70 and 100 MPa are used in this irradiation run. The neutron fluence attained was corresponding to 0.81 dpa. Measurements of elongation are in progress.
In both experiments, irradiation will continue up to fluences representative of the ITER technological phase.

WASTE MANAGEMENT AND DISPOSAL

Tasks which pertains to the action Safety and Environmental Assessment Long-term of the European Fusion Technology Programme. SEAL 10,1 aims at developing a waste management strategy for fusion activated materials which reduces the amount of permanent nuclear waste. Three waste management options are investigated:

1. Clearance, i.e. declassification to non-active waste (NAW),

2. Recycling, i.e. reuse in the nuclear industry,

3. Disposal. as radioactive waste for the material not fulfilling conditions for options 1 and 2.

Recycling procedures are developed, depending on the surface dose of the activated materials after an interim storage period (±50 years) at the plant site.

A surface dose of 10 µSv/h is taken to be the upper limit for hands-on recycling, in compliance with the ICRP 1990 recommendation on dose limitation, whereas 10 mSv/h represent the upper limit for recycling by remote handling. Intermediate values of dose rates define partial hands-on- (e.g. by limiting the exposure time) and limited remote handling-procedures.

Clearance feasibility is evaluated from the specific activities $A_1 ... A_i ... A_n$ of the n significant radionuclides contained in the waste and the unconditional clearance levels $L_1 ... L_i ... L_n$ which are the specific activities allowing the clearance of a waste containing the single radionuclides alone.

Following an IAEA Report[1], the clearance levels are evaluated from parameters like the energy of gamma and beta emissions, the allowable limits of intake by inhalation and ingestion which define the potential hazard of the radionuclides. Clearance of the waste should be possible if the sum of the ratios A_i/L_i called "clearance index"I_c, is less than unity.

Whereas the analyses performed on 1995 concerned waste from materials irradiated in ITER conditions [2], the present analyses [3, 4] deal mainly with the power reactor design SEAFP Model 2, SM-2 with water-cooled Pb-17Li blanket, in-vessel structures made with the reduced activation ferritic steel LA12TaLC, divertor made with beryllium armour, copper heat sink and water coolant.

Additional data were evaluated on SEAFP Model 1, SM-1, a more advanced design with He-cooled Li2O blanket, V-5Ti as the structural material, snd a divertor with beryllium armour, V-5Ti heat sink and helium coolant.

The presence of tritium inventories in the irradiated material is also taken into account.

In the case of materials from in-vessel components, prevalently destined to recycling, it is assessed that the outgassing of the residual tritium after the detritiation procedures would not give any significant dose in a vented working environment.

Similarly, it is controlled that tritium concentrations expected in materials from out-of-vessel components would not affect significantly the feasibility of clearance shows the clearance indices of materials from SM-2 components. It can be seen that clearance is feasible for waste arising from out-of-vessel components and also from the outboard zone of the vessel.

Component	Materials	I_c	Clearance
Inb.+Outb.Shield	OPTSTAB	>>1	NO
Outboard Vessel	OPTSTAB	0.9 (10 a)	YES
Inboard Vessel	OPTSTAB , Lead, B4C	>> 1	NO
Outb. T.F.C.	AISI 316LN, WP Mix, Insul.	0.02 (10 a)	YES
Inb. T.F.C.	AISI 316LN, WP Mix , Insul.	> 1*	NO

Table VI: Clearance indices and clearance feasibility for SEAFP Model 2, by component. Cooling time is 50 years when not otherwise indicated.

* Clearance is possible after 100 years of decay.
T.F.C.: Toroidal Field Coils
OPTSTAB: reduced-activation manganese-based stainless steel.
WP-Mix: 7.3% Nb-Sn, 91% Cu, 1.7% Sn.

The overall results of this study are:

- The activated waste arising from SM-2 is 69000 t. 48% of this waste can be recycled, 39% cleared (i.e. declassified to non-active waste), 13% has to be disposed of. Prolonging the interim storage to 100 years, the amount of material to be disposed of is reduced at 1% (copper of the divertor structures).
- The activated waste arising from SM-1 is 58000 t. No activated waste should be disposed of, 70% could be recycled, 30% could be cleared. The material which can be declassified is less than in SM-2, due to the better shielding characteristics of the SM-2 Blanket.

These analyses show that, if recycling /clearance will be applied within the proposed limits, the amount of fusion activated waste would be significantly reduced.

FLUID SEPARATION

FUSION WORK

<u>*Effect of oxidation on tritium retention in Be*</u>

Fusion reactors will probably be lined with Be, and have Be in the blanket. Tritium will form in the Be, and should be removed continuously from the blanket to fuel the fusion process. There are computer models to predict its release rate: they ignore oxidation. We are studying how tritium release will be affected by surface oxidation by air or water, which might come from leaks or accidents. The apparatus was completed, and experiments started in September, when we received irradiated Be specimens.

We already have very interesting results: oxidation in dry air impedes tritium release (as expected from theory), but in humid air or helium, tritium release was much faster than in inert gas. So we now plan to look at tritium release from bulk beryllium during accidents, as well as the after-effects of oxidation on tritium release from the blanket.

Tritiated waste studies

In decommissioning a fusion reactor, one must remove tritium from a large quantity of various components. We studied how best to remove it from Be, graphite and steel.

We found that tritium was all in the surface-deposited sputter layer on the surface a beryllium tile from the JET torus. There was no difference between tritium release into vacuum, nitrogen or inert gas; the peak in tritium release was at 600°C. However, some tritium remained at 950°C, where vaporization of Be becomes a practical problem. However, adding water vapour or hydrogen reduced the release temperature (Fig. 68)

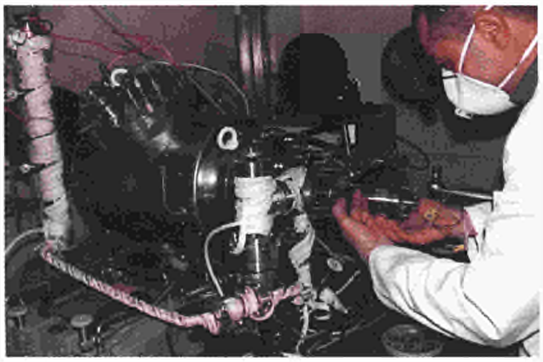


Fig. 68: Loading an apparatus for measuring tritium release from plasma-faring materials as a function of T, P and gamer gas composition

Highly-irradiated Be in ITER will be more difficult to detritiate because tritium will be distributed through the bulk. Now we are working on dissolving Be in an alloy with a low melting point: this gives guaranteed detritiation at a relatively low temperature, avoiding Be evaporation problems.

Graphite is another likely component of the plasma-facing wall of a fusion reactor. It is important to remove tritium during decomissioning. Experiments on graphite JET tiles were similar to those on Be tiles. But the results were different: although 99% of the tritium was initially in the sputter-deposited surface layer, about 30% of it entered the graphite bulk during heating, and was difficult to get out again: ~2% was retained to well over 1000°C. Water vapour additions reduced the tritium adsorption in the graphite, possibly by blocking adsorption sites. However, ITER should consider dissolving the sputter-deposited layer off their graphite tiles as a first step in detritiation.

We looked at the practicalities of removing tritium from steel, based on the process metallurgy literature as well as research results. Vacuum melting has been used: it has the advantage of giving a homogeneous product and volume reduction. However, we showed that it cannot reduce the total hydrogen content below ~1 ppm: detritiation effects are caused by accidental isotope swamping due water in refractories. The same results could be obtained more conveniently using a simple cover gas; and much better detritiation could be expected by deliberately bubbling a small quantity of hydrogen through the melt. The diagram of a suggested layout is shown thereafter (Fig. 69).

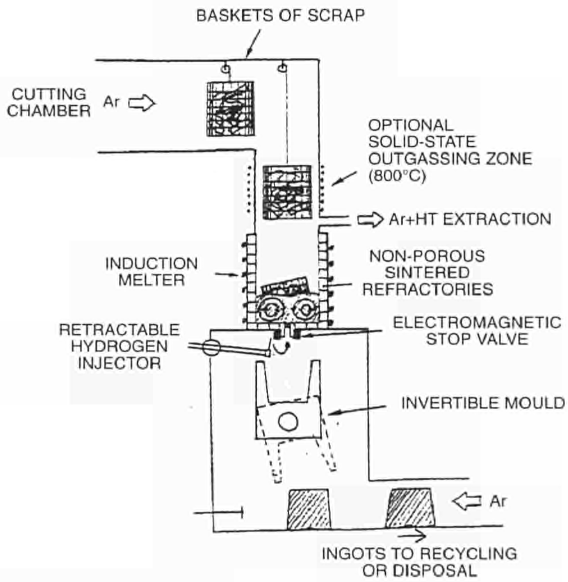


Fig. 69: Proposal for a detritiation system for metallic scrap, based on hydrogen swamping via outlet of an electromagnetic stop-valve

Reduction of tritiated water

In ITER, and all tritium labs, tritium is recovered from air or gas streams by catalytically oxidizing tritium in the gas to tritiated water (HTO) and then removing it in dryers. But one must transform the HTO to HT for isotope separation and tritium recovery. At present, the most usual method is to use a uranium getter-bed. This is very expensive and creates a solid tritiated waste. At JRC, we patented and demonstrated a simple reactor-permeator in which activated iron pellets reduce HTO vapour. When spent, the iron pellets are regenerated using hydrogen without loss of performance. The only waste is some low-level tritiated water.

After demonstration at a small scale, we made a design for a plant capable of reducing 35-70 kg of concentrated tritiated water per year of high-level tritiated water, and modelled its operation. We intend to demonstrate the full-scale plant in a high-level tritium laboratory, and sell units to other tritium laboratories and fusion research centres.

New tritium monitors
Ionization chambers are the standard way to measure tritium. But they suffer from saturation at high tritium levels, and have a "memory" caused by retained tritium. We patented two completely new tritium detectors:

- The **gas-scintillation monitor** works if tritium is in a carrier gas. There is no saturation, and the sensitivity is better than an ionization chamber of the same volume: the detector works over **17 decades** of tritium concentration. Tritium memory is also improved (Fig. 70)

- For room-air monitoring, a **scintillating zeolite monitor** selectively detects the more dangerous HTO form of tritium: it reacts faster than existing discriminating monitors, and is cheaper to make.

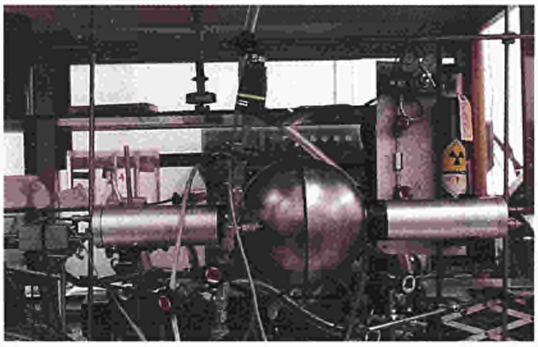


Fig. 70: Gas scintillation monitor for tritium

Zeolites with low tritium retention
Fusion reactors and tritium laboratories use large molecular sieve (zeolite) beds to dry air or other gases containing HTO. They have a problem: after drying air containing concentrated tritiated water (e.g. from a spill), the molecular sieve retains several % in its structure, even after thorough regeneration by heating in dry gas. This retained tritium contaminates (by isotope exchange) any ordinary moisture which it subsequently sees: the residual moisture coming out of a dryer on the next cycle, and next batch of water from regeneration.

We found the problem was caused partly by the choice of zeolite itself and partly by the binder used for making commercial pellets. We developed a new zeolite pellet which has now been produced at a pilot scale by industry. Initial results show that the **tritium retention has been reduced by a factor 1700** compared to conventional material.

Renewable energy (shared-cost action)
We participated in the definition stage of a project to get **hydrogen (for fuel cell) from bioethanol.** We investigated ethanol concentration processes, designed a vacuum-pressure-swing adsorption plant for separating and purifying the hydrogen product, and screened various commercial catalysts for the ethanol steam cracking. The steam cracking was performed inside a thermobalance, which weighed the catalyst (to measure carbon dumping), whilst a mass spectrometer sampled the gas just above, to measure the reaction rate.

Energy saving (shared cost action)

We helped demonstrate a novel off-shore desalination pilot plant which, by taking advantage the hydrostatic pressure in deep water, saves up to 70% of the energy costs (Fig. 71).

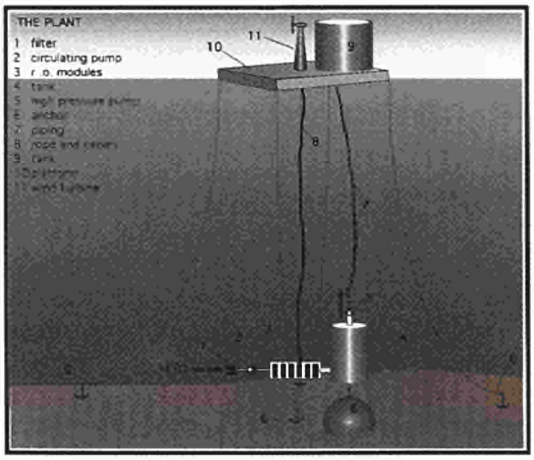


Fig. 71: Desalination Model Plant

HYDROGEN MATERIALS INTERACTION

Influence of traps on deuterium diffusion in the martensitic steel DIN 1.4914 MANET.

A time dependent permeation method is used to measure the permeability, diffusivity and solubility of deuterium in the martensitic steel DIN 1.4914 MANET (MArtensitic for NET). MANET is a candidate material for the first wall and structure of the demonstration power reactor (DEMO). The measurements cover the temperature range from 373 to 743 K that includes the onset of deuterium trapping effects. The results are interpreted using a trapping model. The trapping parameters such as the number of trap sites, N^t, and their average energies, E_t, for deuterium in MANET steel were determined. Using N_l=5.2•10^{29}sites/m^3 (this value is based on the assumption that, in bcc iron, there are 6 tetrahedral sites per host atom), N_t=1.5•10^{25}sites/m^3 and E_t=48500 J/mol.

The Arrhenius expression for MANET II for the deuterium permeability, lattice (633-743 K) diffusivity D_l and lattice (633-743 K) Sieverts' constant value $K_{s,l}$ are as follows:

$P=4.2.10^{-8}.\exp(-42380/RT)$ (mol m^{-1} s^{-1} Pa$^{-1/2}$)

$D_l=1.01.10^{-7}.\exp(-13210/RT)$ (m^2 s^{-1})

$K_{s,l}=0.27.\exp(-26670/RT)$ (mol m^{-3} Pa$^{-1/2}$)

Influence of the surface conditions on permeation in the deuterium-MANET system. Constants of adsorption and recombination sk1 and σk_2 of deuterium on martensitic steel DIN 1.4914 (MANET II) have been determined under different, well characterised, surface conditions. It was found that the condition of the surfaces is of crucial importance for the hydrogen/deuterium permeation through this steel.

Bare MANET II,

$\sigma k_1 = 5.56 \times 10^{-7} \exp(-19093/RT)$ (mol m^{-2} s^{-1} Pa^{-1})

$\sigma k_2 = 7.63 \times 10^{-6} \exp(+34247/RT)$ (mol^{-1} m^4 s^{-1})

Oxidised MANET II,

$\sigma k_{1(ox)} = 4.9 \times 10^{-7} \exp(-58383/RT)$ (mol m^{-2} s^{-1} Pa^{-1})

$\sigma k_{2(ox)} = 6.7 \times 10^{-6} \exp(-5044/RT)$ (mol^{-1} m^4 s^{-1})

The growth of an oxide surface layer (Cr_2O_3) of about 25- 30 nm in a MANET II sample heat treated in an oxidising environment, compared to the bare MANET II that have a "natural" oxide of about 5 nm has provoked a reduction of both the permeation rate and the recombination coefficient (about three orders of magnitude). In addition, the permeation governing process has changed from diffusion-limited to surface-limited.

Influence of different susceptibility to oxidation on the hydrogen diffusivity in the low activation martensitic steels LA12TaLC and LA7TaLN.

The evaluation of hydrogen diffusivity D and Sieverts' constant K_S in the temperature range 550 – 900 K at a working pressure of 10^5 Pa was carried out experimentally, by a gas evolution method in two different low activation martensitic steels (LAMS), namely LA12TaLC and LA7TaLN, which are possible candidates as armour and/or structural materials for a DEMO concept reactor device.

After a complete set of measurements, a strong reduction of "apparent" diffusivity (up to two orders of magnitude) was observed in LA12TaLC, in contrast with LA7TaLN which has given no variation of D. The different content of Cr in this two steels, (wt% 8.9 and 11 respectively), is responsible of a different oxidation resistance against gas oxidising impurities present in the gas atmosphere which give, as a result, a superficial oxide layer formation in LA12TaLC of around 270 μ, while in LA12TaLC virgin and LA7TaLN the oxide layer is only of 10-20 μ, which acts as diffusion barrier to hydrogen. The presence of Cr_2O_3 and MnO_x were found using XPS and AES analysis of the specimens' surfaces.

TiCN and surface oxidation as hydrogen permeation barrier in F82H.

The production and permeation measurements of a TiCN coating on the surface of F82H martensitic steel were studied. The coating was produced by Physical Vapour Deposition (PVD). The TiCN coating has been deposited on both sides of a disk sample. After the first two measurements at 523 and 573 K, which gave a permeation rate reduction factor of 220, an oxidation in situ of the specimens in an environment of "wet hydrogen" was performed. The oxidation temperatures were 623, 673 and 723 K. In the Arrhenius plot (Fig. 72) a gradually decrease of the permeation rate with temperature can be seen. Finally it was observed a permeation rate reduction of almost 4 orders of magnitude at 573 K.

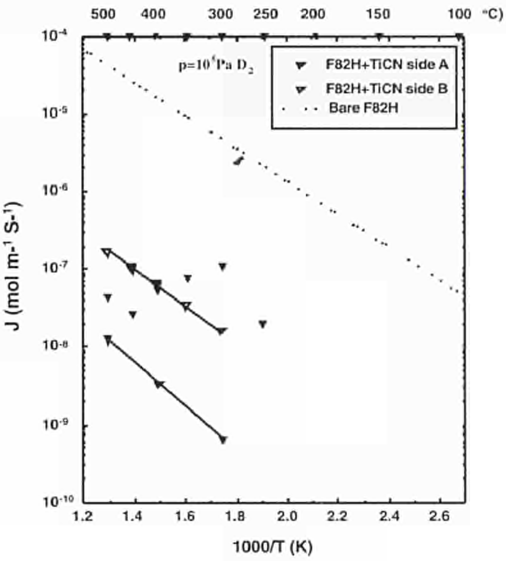


Fig. 72: Arrenius plot of the permeation rate of deuterium though bare and PVD-TiCN F82H. The upstream deuterium pressure was 100 kPa.

SEM analysis of the coated sample after oxidation revealed the presence of defects such as micro-cracks in the coating. EDS microanalysis revealed presence of oxygen in these micro-cracks. A plausible explanation for these results is that during the oxidation process the defects on the coating layers of the sample might be filled by the formation of oxides ("self healing"). These oxides can be the result of either a direct oxidation of the substrate in correspondence with the micro-cracks, the oxidation of the deposited layer (thermodynamically possible) or a combination of the two effects. Therefore an increase of the barrier efficiency was obtained and hence the observed strong decrease of the effective deuterium permeation rate. Furthermore, the change in activation energy for the permeation rate in the coated F82H after oxidation (54700 J mol-1), compared with that for bare F82H (40700 J mol-1), indicates that the reduction effect is not merely a result in surface available for permeation.

The effect of cyclic loads on the hydrogen permeation rate of structural

materials.

An experimental facility has been designed and constructed to measure the hydrogen permeability of structural materials under cyclic tensile loads. A gas permeation technique has been employed to determine the hydrogen permeabilities for uncoated tubular 316 stainless steel and DIN 1.4914 MANET II specimens. The results obtained over the temperature range 450 to 873 K and for driving pressures of 100 kPa, without tensile loads, are in excellent agreement with the literature data and as such validate the apparatus. Cyclic tensile loading (up to 120 MPa, cyclic rate 10 min/cycle) of 316 stainless steel yielded no measurable effect on the permeability over the temperature range 540 to 873K.

Testing of a water vapour cold trap for atmospheric air detritiation.

A water cold trap has been tested to determine its removal efficiency. The emphasis is on the influence of the cryogenic cooling on the efficiency of the process.

The cold trap has the form of a shell-and-tube heat exchanger. It employs tube bundle of 57 vertical tubes. The bundle is immersed in the cryogenic bath accommodated in a cylindrical shell. The mechanical design of the device is shown in Fig. 73.

The overall performance characteristics were found to be moderate concerning atmospheric air detritiation requirements. The physical mechanisms do not help in obtaining extremely high removal efficiencies. Nevertheless, the cold trap can operate satisfactorily under moderate cryogenic temperatures, and it may serve as a pre-drier to remove most of the water vapour throughput (for instance, during an accidental release).

To achieve ultra-dry exit conditions, appropriate means must be taken to trap the outflowing aerosol particles. Application of filters does not seem to be an effective approach (the size of the particles is of the order of 1 μm). Possible alternatives may be: (a) creation of high turbulence in the flow to increase impaction deposition, (b) generation of electrical precipitation.

Tritium inventory and permeation analysis of the ITER Plasma Facing Components.

The TMAP4 code has been used for the evaluation of tritium inventory and permeation towards the coolant in the ITER reactor. A survey of material data available in the literature has been performed as input for the calculation code.

Data on tritium inventory and permeation fluxes were calculated for various PFC's components of the ITER reactor.

A discussion of the validity of the calculation considering the data available and recommendations for future research and development as well as some design considerations were presented.

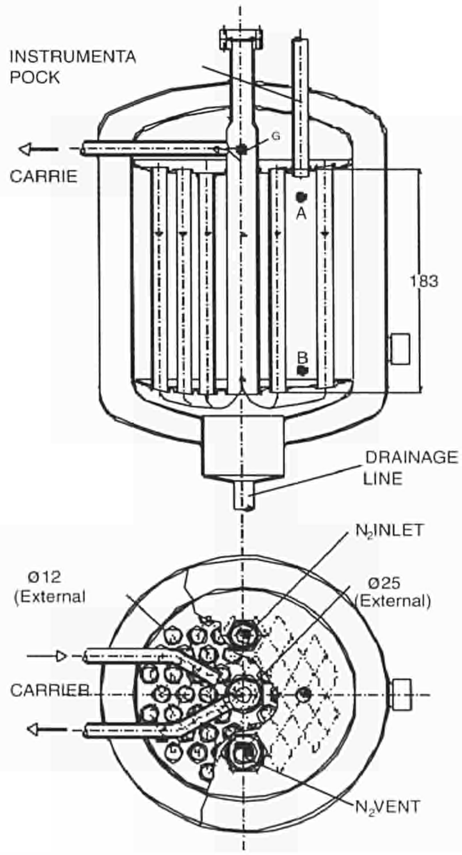


Fig. 73: Cold trap design

Institute for Advanced Materials

Technology Transfer in 1996

During 1996, IAM executed one project and won six others from DG XIII/D in the frame of the Valorisation programme. This major increase in successful projects is an illustration of the potentiality of an Institute like IAM in the process of Technology Transfer.

NEURAL NETWORK PACKAGE FOR THE ANALYSIS OF ULTRASONIC TESTING TECHNIQUES: NNPV

A joint project of IAM, ISIS and ENEL, Laboratory of Piacenza (Italy).

JRC, Operating Agent of several European Networks on NDT effectiveness evaluation and qualification, is depository of the archive and information bases generated by these co-operation such as PISC and ENIQ. JRC, due to its role of Reference Laboratory in this field of action has deeply understood the shortcomings of industrial inspection procedures based on NDT.

Besides lack of capability of several techniques uses for specific defect types and human errors induced by over estimation of capability, a typical difficulty is the one of full description of the inspection procedure used. This description must explain the contribution of the different techniques to the final result, the decision steps involved and where the skill of the operator is of importance. Capability evaluation of an inspection procedure is illusory if such a procedure is not fully documented and described ; qualification of an inspection procedure is impossible if such a procedure is not fully described and if each decision step is not clearly stated and justified.

Most of the inspection procedures applied on structural components nowadays rely in part on the skill of the expert called "level 3". This expert does not detail the rules or logic steps he follows to reach a decision about the presence of a defect, about the fact that indications are corresponding to geometric features and thus to be neglected, about the size of a defect.

Sizing of defects, in particular, represents an art that is often very badly documented in inspection procedures. Few NDT techniques exist which demonstrate a defect size without any doubt. Often such techniques are not applicable due to geometrical features of the component or simply due to the characteristics of the material – weld or base material – used to build heavy duty industrial structural components, the worst example being the one of the cast austenitic steel.

Objective Of The Study

An important contribution to the industrial application of inspection procedures, often conducted by small service vendors, would be the possibility to qualify these procedures of inspection in an official manner . Such a qualification would allow their participation to tendering and servicing by any capable service vendor on the basis of demonstrative standards more than on descriptive standards or codes. (Ref. European Pressure Equipment directive) (1). To reach such an objective, now understood as essential both for standard components inspection (e.g. pipeline welds) or in service inspection of nuclear components, the absolute need is the FULL PROCEDURE DESCRIPTION. No qualification is in fact possible on the basis of expert skill only : rules applied and decision steps have to be documented (Ref. European Methodology for Inspection Qualification/ENIQ)(2).

It is clear that the particular aspect of defect sizing makes the respect of this qualification requirement very difficult. However if, instead of declared operator skill, a sizing procedure would be accepted based on a Neural Network or model trained in correct conditioned and qualified itself for the purpose, the introduction of such a validated model in the inspection procedure would allow a full and official qualification.

Establishing a neural network (NN) for the precise scope of defect sizing is therefore the aim of the study. Training this network on relevant data and verifying its performance in view of its use in industrial inspection procedures are thus the objectives of the work. The Neural Network (NN) model used in this activity is called Multilayer Perceptron (MLP), which is by far the best understood and most widespread connectionist -i.e. NN- approach.

Complementing a procedure to allow its qualification

The study has demonstrated the applicability of the NN to replace the only " skill of the operator " in the description of an inspection procedure that has to undergo a severe qualification process which requires the full description of all the decision steps part of that procedure.

The procedure description becomes : "Neural Network Package applied using as input the operator / level 3 sizing results and the individual techniques results as such."

If the NN package is validated, it constitutes a well documented procedure, easy to follow and qualify.

This result is essential when the component to inspect, due to its geometry or material, poses inspection problems that are apparently solved by the expertise of one operator. It is obvious that qualification (that has to lead to full competition between vendors) cannot rely on the limited verification that an expert does a very reliable job on some defects when it can be demonstrated that to establish his skill in a statistically satisfactory manner hundreds of defects would be necessary. The certification of operators, as existing, is considered as a necessity in the European Qualification Methodology but not as being sufficient : a complementary qualification can be required. This complementary qualification procedure must rely on the verification of the capability to apply a fully documented inspection procedure.

Increasing reliability

The NN proposes defect sizes based on training and setting of the model which allow safety or economy .

Even without looking for safety and thus without adding any safety feature (oversizing) it was demonstrated that the NN was more systematically capable than the "PISC' operator !

INNOVATIVE LONG-CARBON FIBRE REINFORCED CERAMIC MATRIX COMPOSITE MATERIAL FOR ENHANCING INDUSTRIAL WEAR APPLICATIONS: RING SEALS AND BRAKE LININGS.

Ceramics are attractive candidate materials for wear applications owing to their excellent properties of thermal stability, high elasticity, hardness, resistance to chemical corrosion and low inertia. Monolithic oxide, nitride and carbide ceramics, as Al_2O_3, ZrO_2, Si_3N_4, SiC and others

have been recognised for use in structural applications such as seal rings, valve seats, dies for extrusion, guides, valve train components, bearing parts or cylinder liners.

The major shortcoming of the materials is their low fracture toughness, resulting in inherently, brittle components. In recent years considerable effort has been devoted world-wide to improving ceramic fracture toughness, primarily for structural engineering applications, by incorporating additional phases into the base materials generally in the form of fibres, whiskers or particles. Very few studies have considered the development of Ceramic Matrix Composites (CMCs) for tribological applications and most of these are focused on whisker reinforced ceramics. Nevertheless, the improvement of fracture toughness of ceramics designed for wear applications is of considerable industrial importance and the tailoring of the combined properties of the component phases offers an excellent opportunity to optimise the frictional and wear properties of the composite.

Carbon/carbon composites already developed for aerospace and high performance cars (racing) are not suitable for the severe braking conditions of normal world cars. Therefore there is a need to use more refractory materials. Carbon/carbon wear rates are very high giving a short service life and C/C is most efficient when hot, a condition not acceptable for normal automotive conditions.

The Institute for Advanced Materials in Petten has developed and patented a new material based on long carbon fibre reinforced ceramic matrix composite where the carbon reinforcement provides an in-situ self-lubricating facility in dry sliding applications. This composite material has demonstrated unique tribological properties showing a significant improvement when compared with monolithic ceramics (non reinforced material) already used in some industrial applications where tribology related aspects are crucial,

Two potential fields of applications of this material have been identified in industry: ring seals for pumps manufacturers and brake lining for automotive industry. These two industrial sectors have shown great interest in the IAM composite material and would like to see further development before large volume of actual components are integrated into a service application.

In agreement with potential industrial users in order to bridge the gap between laboratory scale material and industrial applications, this project intends to adapt the following preparatory stages of the material for prototype component manufacturing:

1. infiltration facility scaling up,

2. from sintering of laboratory samples to sintering of complex and large components.

The key objective of this project is to develop real components for practical industrial applications, with an initial focus on the large and important automotive sector. The successful project will improve service life and decrease component weight. This is valuable combination of technical and economical benefit.

COATING FOR THE HIGH TEMPERATURE OXIDATION PROTECTION OF CARBON-CONTAINING CERAMIC MATRIX COMPOSITES

Ceramic Matrix Composites (CMCs) reinforced with long carbon fibres or with ceramic fibres pre-coated with a carbon pyrolytic layer are the most attractive materials for high temperature structural applications because of their high specific strengths. However the potential applications are limited by the high affinity of carbon fibres and carbon pyrolytic interfaces to oxygen. This reaction leads to a maximum service temperature of 400-450°C. The proposed oxidation protection process offers applications ranges up to 1500°C.

One way to protect CMCs in oxidising environments at high temperatures is to use diffusion barriers to prevent oxygen from reaching the composite material.

The Institute for Advanced Materials in Petten has developed and patented a new protection process which is a combination of B_2O_3 and SiO_2 which protects the composite at lower temperatures by the formation of a B_2O_3 glass and at high temperatures, by reacting with the composite matrix to form a complex AlYSiOB glass. The coating process is by deposition from solution, and is therefore rapid and very inexpensive compared to conventional Chemical Vapour Deposition processes. This protection is efficient on Si_3N_4 matrix composites.

The developed protective system offers applications for engines, especially gas turbine engines and for heat exchangers up to 1500°C.

For both aerospace and heat exchanger applications, there are significant economic benefits which can be achieved. These include weight savings of up to 50%, the ability to eliminate the need to ventilate turbine blades and to run at higher temperatures and finally the economic strategic replacement of imported exotic metals such as Cr, Mo and Nb.

There is a strong evidence from industrial contacts that JRC/IAM Petten has developed, that there is a demand for a material that can operate reliably at significantly higher temperature. The use of CMCs would be acceptable if the fibre oxidation problem could be overcome. This project aims to demonstrate that there is an industrial process available that can solve this problem.

The key objectives of this project are:

1. To design, develop and provide a prototype equipment to coat composite components in a controlled, reliable and automated way.
2. To test the coated components to verify and demonstrate the effectiveness of the equipment and the value of the coating process.

CONVERSION SOFTWARE WITH GRAPHICAL USER INTERFACE FOR MAGNETIC RESONANCE IMAGING AND COMPUTERIZED TOMOGRAPHY IMAGE DATA

This proposal provides for the efficient transfer and manipulation of Magnetic Resonance Image (MRI) and Computerised Tomography (CT) data for use in the European Boron Neutron Capture Therapy (BNCT) project.

BNCT has been proposed for patients with a virulent brain cancer (glioblastoma multiform) for which the prognosis is poor with a median survival time of only 9 months. Other cancer treatments are ineffective with this cancer whereas with

BNCT encouraging results are reported from Japan and the USA.

The European BNCT project is expected to begin Phase I clinical trials with human patients in 1997 at a specially constructed facility in the Petten High Flux Reactor (HFR) which is now complete. The BNCT facility at Petten is unique in Europe and is one of only 4 centres in the world; 2 in the USA and 1 in Japan. One other reactor based facility in Europe may become available for patient treatment in 2 years time.

Detailed treatment planning is an essential prerequisite for each patient irradiation to estimate the radiation doses delivered. This will be performed at several institutes involved in the European collaboration, including JRC Petten, where specially written software for BNCT treatment planning will be used.

The BNCT treatment planning software at Petten requires as input MRI or CT data. These data will be provided by various medical centres within Europe that refer the patients for participation in the first clinical trial. However the medical centres use different MRI/CT machines which generate image data in a variety of formats. This confusion of image formats is being addressed but no standard has yet been agreed. Moreover many MRI/CT vendors are committed to their proprietary formats so that a practical standard is not expected for some years.

The treatment planning software at Petten requires image data in a specific format – incompatible with those provided by the medical centres. Consequently there is an urgent need for a software tool to convert the image data provided by the medical centres into a format suitable for use at Petten. Given the life expectancy of the patients being treated it is important to minimise the time between diagnosis and treatment. The development of an efficient and reliable tool to convert image data will significantly help in this respect.

The objective of this project is to build a software tool to convert the image data supplied by the medical centres into a format acceptable by the treatment planning software. The software conversion tool needs to be written in the C language for UNIX based workstations using the X-Windows system. In addition to converting the image formats the software should have the capability for simple editing of the data.

NEW CONCEPT OF PRESSURE VESSEL DESIGN IN ORDER TO AVOID HYDROGEN ATTACK: FEASIBILITY DEMONSTRATION AND EVALUATION OF ECONOMIC IMPACT

Hydrogen attack is a well-known phenomenon in the petrochemical industry which leads to the degradation of the mechanical properties of the steels used for pressure vessels in hydroprocessing units.

Hydrogen absorption at high temperatures gives rise to decarburization of the steel with simultaneous appearance of methane bubbles in the steel created from the chemical reaction of the hydrogen with the steel's dissolved carbon or carbides. Within these bubbles the methane pressure is extremely high creating a high internal stress which combines with the system stresses to accelerate the creep damage leading to premature failures.

To minimise this problem, steels with low carbon content are chosen to limit the carbon – hydrogen reaction. Generally however such steels are limited in creep resistance as this relies heavily on carbide strengthening. They are selected according to the "Nelson curves" (API 941) which present the temperature – hydrogen pressure resistance of the different steels according to the available data from plant experience.

At present, European industry exploits around 100 hydroprocessing vessels weighing up to 1000 tons and costing 2-3 MECU each made mainly from 2.25Cr-1Mo ferritic steel and operating at a maximum temperature of 450°C and 170 bars. Internally, a thin layer (1 cm) of a more expensive, austenitic stainless steel is clad to prevent corrosion by the contaminated gas which also limits the superficial decarburization of the ferritic steel.

The remaining problems are :

1. Operating conditions are limited because of the synergy of creep and hydrogen attack. The vessel is always overdesigned to consider hydrogen presence and the process temperature is limited.

2. The austenitic steel cladding has a much higher solubility for hydrogen than the base material, thereby providing a continuous hydrogen source for the base material.

3. During operation or cooling down of the vessels a disbonding of the clad steels is observed. This is attributed to the high hydrogen concentration in the interface (the austenitic – ferritic interface provides a discontinuity in the hydrogen concentration).

4. Extremely long and expensive studies are needed to predict the behaviour of new candidate steels for hydrogen service.

At the present time, because of its unique high pressure hydrogen test facilities, the CITE (Component Integrity Testing and Evaluation) laboratory of the IAM is involved in a European programme (BRITE-EURAM n* 1835) which aims to evaluate new steels to reduce hydrogen attack (title : Prediction of Pressure Vessel Integrity in Creep Hydrogen Service). Among the partners in this programme are important European industries directly interested in the development of such vessels. These cover market leaders in the production of special steels (for hydrogen service), important constructors of vessels as well as a European based internationally renowned exploiter of such vessels. Within this project, new generation steels are sought for pressure vessel construction able to permit an increase in the process conditions of the pressure vessels up to 500oC and 200 bars. According to a market study made by the industrial partners this should lead to savings of at least 100 MECU/year for the European industry.

As a completely alternative approach, the CITE laboratory has proposed a patent for a fundamentally new pressure vessel design which could effectively permit an increase of the process temperature without resorting to new generation steels. The new design is intended to avoid hydrogen attack of the vessel as well as to resolve the current industrial problem of "disbonding" of the cladding.

The proposed design will facilitate :

- a choice of the structural steel (outer wall) only on the basis of its creep characteristics

- a decrease in the thickness of this wall, as no hydrogen attack is expected

- an increase in the process temperature dependent only on the creep properties of the structural steel.

- an increase in the life of the vessel

- an increase in safety

All of these consequences will obviously contribute to a higher, not yet quantifiable, level of economic benefit than foreseen through the simple introduction of special steels. Nevertheless, a number of new technological problems concerning the design and construction of the inner wall have to be solved before implementation of the proposed design.

The aim of the present proposal is to :

1. confirm the principle and demonstrate the feasibility of the new design of pressure vessel, identify and resolve any technological problems

2. obtain quantitative data on the advantages provided which will help in the evaluation of its economic impact.

Achieving these aims of demonstration and benefit evaluation is expected to convince even such a traditionally conservative industry with regard to structural design of plant.

To this goal, preliminary tests on simulated vessels will be realised to enable the design, development and construction of a reduced scale industrial prototype which will be demonstration tested under industrial conditions representative of advanced operating conditions commensurate with such a revolutionary design.

PROTOTYPING OF AN AUTOMATED VIDEO IMAGE CAPTURING SYSTEM (PAVICS)

One of the major activities of the Mechanical Performance and Joining sector of the Institute for Advanced Materials of the JRC has, for a decade, been the testing and evaluation of high temperature aeroengine materials using thermo-mechanical fatigue (TMF) techniques. Due to the extreme conditions under which the tests are carried out, and the long duration for which the tests are required to be run uninterrupted, the cost for each test is high. A major fraction of this work is carried out under third-party contracts for major European aerospace manufacturers.

To obtain information about the origin of cracks and their growth throughout the specimen, time-lapse video pictures of the specimens are taken at predefined points throughout the tests, in synchronisation with the test cycles. At this moment the video imaging system used is very labour intensive, with low reliability and poor image quality. It also suffers from the additional problem that there is no possibility to directly evaluate the images within this system. Five years of working with a standard system has shown that it is not possible to extend the system to produce the functionality that is demanded by our customers.

In order to meet the demands for automatic, efficient and user-friendly monitoring of the cracking which occurs on the specimen surface during testing, an innovative automated time-lapse imaging system has been conceived. From estimates made for partial automation of the crack measurement procedure, it is predicted that the productivity increase available from the implementation of this system would be in the order of 80%.

The present proposal is for the building and evaluating of a prototype of that system, of which the Institute for Advanced Materials holds the intellectual property rights of both the system concept and the compiled software and source code. The technical work to be conducted consists of system analysis and specification, computer programming, system assembly and testing, system evaluation and documentation, and exploitation.

CORROSION TEST MANAGER – CTM

This project is dealing with the development of a computerised database and information tool for the daily logging, configuration and presentation of raw experimental corrosion test data

The Corrosion Test Manager (CTM) is a computerised data management and information relational database system designed specifically for the storage, handling, record-keeping and analysis of raw corrosion test data on a daily basis. It consists of two inter-linked modules, the first dealing exclusively with experimental data (CTM/D) whilst the second is concerned with microstructural images which result from the structural analysis of the exposed test specimens (CTM/I).

The programme will be able to run on either local laboratory PC's for daily data logging or as a centralised archiving and information-management system. The CTM system runs under WINDOWS (including the 32 bit Windows 95 system) and in a Network multi-user environment.

The System is being developed primarily as an in-house tool but its use can be broadened and extended to help other research centres and industries more efficiently store and evaluate the copious amounts of experimental data which have been or are being generated.

The objectives of the project are:

1. to extend the present system to include other types of corrosion test used in-house and elsewhere, e.g.

    - thermogravimentic studies for the uninterrupted determination of corrosion kinetics

    - burner rig studies for gas-turbine-type conditions

    - thermal-cycling tests in corrosive atmospheres

2. the installation of a measured metal-loss (due to corrosive attack) data system of importance in real engineering considerations

3. to develop and improve the software package of the CTM system so that it meets high quality commercialisation standards, i.e.

    - optimise and protect the system maintenance tools with password procedures

    - create Help file and complete user documentation (instruction manual, reference approval programming manual etc.)

    - complete system installation and set-up

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ABSTRACT:

The 1996 Annual report of the Institute for Advanced Materials (IAM) of the JRC highlights the achievements of the Institute during this past year.

An overview of the management of IAM is presented in the introduction. Contributions to research and technological development achieved whilst the execution of specific R&D programmes. Technology transfer achieved in 1996 also is thoroughly reported.

The Operation of the High Flux Reactor (HFR) resulting from the Supplementary Programme is presented in a condensed form. A full report for the HFR is published separately.

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