ANNUAL·REPORT·91



# Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

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# I Introduction

# Introduction

This report reviews the progress made at the Institute during 1991. As 1991 was the final year of a four year JRC programme, it is appropriate that we should look back over the achievements of the past four years, and look forward to the new challenges of the next JRC programme covering the three years from 1992-1994.

In the new programme, our accumulated expertise in the field of high temperature materials will be carried forward, adapted, and expanded to meet the evolving needs of the European materials community. Our awareness of current and future needs derives from our many contacts, in both research and industry, with a primary input coming from our Institute Advisory Committee. The decision to set up an Advanced Coating Centre was a major response to ensure that the Institute will continue to provide technologically relevant facilities. An influx of new, younger staff, and a new building housing both staff and laboratories is providing the Institute with the resources required to meet these new challenges smoothly and efficiently.

## Programme Structure of the Institute

The Institute is conducting its work under several Programme forms, which have individual natures and separate budgets. As before, we report under these headings:

- The Specific Programme on Advanced Materials
- Contributions to other Specific Programmes
- Projects in Support to Commission Services
- Exploratory Research Programme
- Third Party Contract Research
- Complementary Programme of the High Flux Reactor.

## The Specific Programme on Advanced Materials

Here, projects derive from the Specific Programme on Advanced Materials of the Framework Programme of the C.E.C. The main research areas and objectives are:

- Properties, Performance, Characteristics and Improvements of Structural Materials

  *Alloys*
  To study the performance of alloys in simulated industrial environments with physically based modelling and experimental verification to predict behaviour in service.

  *Engineering Ceramics*
  The investigation of engineering ceramics behaviour in simulated industrial situations. The analysis and engineering of microstructural and interfacial factors influencing materials properties.

  *Components and Thermal Fatigue*
  Measurement and modelling of crack propagation in cyclic thermal gradient fields with and without simultaneous irradiation damage and creep: to predict component behaviour for industrial applications where thermal fatigue is a life limiting factor.

  *Operational Defects in Materials and Lifetime Prediction*
  Development of methodology to identify and quantify the microstructural defect state in those components which determine the lifetime and performance of structures in industrial service, leading to the formulation of codes for life-time prediction and design.

  *Reliability*
  Development and application of diagnostic techniques and non-intrusive methods (coherent light and thermal emission) and acoustic emission for materials and components. Numerical simulation of creep and fatigue behaviour.

  *Modulation of Surface Properties*
  *Wear & Corrosion Resistant Coatings*
  To develop new procedures for the synthesis of protective (wear, corrosion resistant and thermally insulating) coatings by PVD, CVD, LPPS and by treatment with ion beams.

  *Surface Treatments for Improved Performance*
  Improvement of surface properties of metals and ceramics by ion implantation, laser treatment, electron beam melting, sputter coating and combination of these methods, determination of hardness, wear resistance, friction, corrosion resistance.

- Properties, Performance, Characteristics and Innovation of Functional Materials

  *Composite Materials Properties Improvements*
  To characterise selected composite materials

(phase dispersed alloys, particle dispersed alloys, fibre strengthened alloys) by microstructural and compositional analyses and mechanical testing.

*Chemical Sensors*
To develop or improve chemical film sensors for environmental as well as industrial gaseous atmospheres ($H_2/OH_x/NO_x,SO_x$) with high performance.

- Data and Information Management for Advanced Materials

  *Data Banks*
  Provision of computerised databases for materials properties used for data management, data evaluation and input to computer-aided engineering, finite element methods, computer-aided processing and data information services.

  *Information Centre*
  To provide an information bureau, a meeting forum and a means for cooperation, the promotion and dissemination of information on materials research in the Community and to act as continuous interface to industry.

Contributions to Other Specific Programmes

The Institute is contributing to the following Specific Programmes in the research areas:

- Reactor Safety
  *Project for the Integrity of Steel Components (PISC)*
  Assessment of the effectiveness of the inspection techniques and procedures and of their reliability when applied to structural components; emphasis on the in-service inspection of the primary circuit of nuclear reactors.

- Radioactive Waste Management
  *Materials Research Aspects*
  To describe the interactions between conditioned waste (vitrified high level waste and alpha-contaminated waste in concrete) and the surrounding materials in final storage conditions, essentially for the development of risk assessment models.

- Fusion Technology and Safety
  *Materials Integrity*
  To provide experimental information on properties and on irradiation behaviour of AISI 316 steel for NET first wall candidate material. Study of effects of plasma disruptions and determination of thermal fatigue behaviour of first wall elements. To obtain data on Pb-Li properties and the compatibility with structural materials.

Projects in Support of Commission Services

A number of projects are directly sponsored by other Services of the Commission in different Directorates Generales. These refer closely to the specific interests and responsibilities of the Services. These projects are:

*Standards for Advanced Ceramics (DG III)*
- Support to and stimulation of the development of European standards and pre-standards
- Execution of R & D actions within European standardisation activities.

*Standards and codes for Non Destructive Testing of Pressure Vessels and Weldments (DG III)*
- Support to the development of European (Pre-) Standards and Codes.
- Stimulation and coordination of Round Robin actions.

*Materials Science and Technology for Aeronautics Applications (DG III)*
- To produce an analysis of the European position with respect to construction materials employed in the aeronautic industries.
- To elaborate a strategic plan for Materials Science and Technology to support European industrial competitivity.

*Standardization of Quality Control Protocols Produced Radio-Pharmaceuticals (DG XI)*
- Support to the Council Directive on radiation protection of persons undergoing medical examination and treatment
- Pre-normative R & D for protocols for chemical, radiochemical, radionuclidic and biological purity.

*Ceramic Catalyst Carrier (DG XI)*
- Assessment of all relevant factors (state of the art).
- Investigation of the degradation mechanism of ceramic catalyst carriers.
- Studying the efficiency of ceramic soot filters.
- Development of methodologies for performance inspection.

*The Role of materials in environmental Problems arising from Power Stations (DG XI)*
- To evaluate the Materials Science and Technology contribution to reduce environmental problems, arising from fossil fuel fired power stations.
- To develop a strategic plan to realize this goal.

*Technology Transfer and Utilization of Research Results (DG XIII)*
- Oxygen sensors
- Passive Downward Heat Transport.
- Ultrasonic Reference Transducers.

*Materials Databanks (DG XIII)*
Support to DG XIII (Information Services Market) on:
1) Organisation and evaluation of the Materials Databanks Demonstrator Programme
2) Organisation of pilot demonstration projects for the industrial integration of materials information services
3) Development of standards for materials databanks.

## Exploratory Research

Here, a small contribution from the value of all the specific programmes of the Joint Research Centre provide a budget for supporting research which is aimed to stimulate originality and fresh directions in the scope of the Institute. During the period under review, the Institute has won a number of such projects, as follows:

- *Design and Construction of an Epithermal Neutron Beam for a Boron Neutron Capture Therapy Facility at the High Flux Reactor in Petten.*
  To design and construct a BNCT facility at Petten for the treatment of certain types of cancer by means of radiation.

- *Joining of Ceramics to Metals*
  To study experimentally the interfacial chemical relations controlling the joining of ceramics to metals and to explore the use of ion beam surface preparation techniques for promoting joining in order to optimise joints for high temperature applications under stress.

- *Micro-Hydrodynamics of Laser Melted Pools*
  To model with computer techniques and study experimentally the expected shapes of molten pools produced by laser melting of engineering alloys.

- *Development of Intelligent Processes for Sub-Micron Ceramic Microstructure*
  To improve the quality of materials by actively steering processing operations towards goals set in terms of materials properties.

- *Development of ceramic fibres for high temperature composites*
  To explore the potential of conferring an adequate corrosion protection of carbon fibres to allow its exploitation in high temperature ceramic composites.

## The Programme of the High Flux Reactor

To exploit effectively the High Flux Reactor in Petten, an outstanding European materials testing facility, for the benefit of member states' reactor technology programmes, for the programmes of the Commission and for other Third Party requirements.

## Achievement Highlights from the 1987/1991 Programme

The achievement highlights in materials research are grouped here under three headings: irradiation of materials, characterization of materials, and improvement of materials - particularly of surfaces and interfaces. The work derived from the exploitation of large facilities, from large projects, and from smaller exploratory projects where ideas for future projects are tested.

One achievement is not materials related; it concerns the medical use of neutrons for cancer therapy.

*Irradiation of materials*

The Institute possesses two large facilities for materials irradiation, the high flux reactor, HFR, at Petten and the cyclotron at Ispra.

The HFR has performed many in-core irradiations of fuels and structural materials for thermal, fast, and fusion reactor types.
LWR fuel testing included experiments with MOX fuel and high burn-up PWR and BWR fuel rods in which power cycling behaviour, transient fission gas release and iodine release during simulated LOCA conditions were investigated. Irradiations for wide-ranging studies of LMFBR fuel behaviour were performed covering both oxide and nitride fuels. The release of volatile fission products from coated HTR spherical fuel particles was studied under steady state and transient conditions. Extensive irradiations to characterize graphite over a wide range of temperature and neutron fluence were also performed for the same HTR application.

A significant contribution was made to the materials programme within the European Fusion Technology programme concerning austenitic stainless steels, vanadium alloys, refractory ceramics, lithium ceramics, and Pb-Li liquid metals. The cyclotron also contributed to radiation damage studies on fusion materials.

The commercial production of radioisotopes in the HFR for medical and industrial purposes was increased to an annual turnover in excess of 1 MECU. In addition, the cyclotron was used to develop an efficient technique for producing medical Ga-68, and production of I-123 was set up to serve as the principle source in southern Europe of this very short-lived isotope.

Activation analysis is carried out in the HFR as a general service to industry and universities. The cyclotron is also applied to activation analysis but specializes specifically on the analysis of the wear of moving components and to spallation behaviour in corrosion studies.

Neutron beams from the reactor are used for neutron radiography and together with spectrometers, neutrons are also used as analytical tools in a wide range of fundamental and applied research.

<u>Characterization of material properties and behaviours</u>

Materials characterization encompasses alloys, ceramics and composites.

Extensive investigations into the corrosion, creep and fatigue properties of alloys for high temperature applications in gas turbines and corrosive environments were pursued with the aims of predicting and improving in-service behaviour. Liquid metal embrittlement and corrosion studies on stainless steels have contributed to the European Fusion Programme and have lead to the development of a ternary oxide corrosion barrier. New techniques were introduced such as in-situ computer vision monitoring of fatigue crack growth, and improved ultrasonic detection of creep induced intergranular micro cracks. The PISC programme has complemented this laboratory scale work by demonstrating how NDI methods can be optimized on full scale industrial components, resulting in the revision of several European industrial NDI procedures.

The reliable measurement of long term behaviour, e.g. creep and fatigue, is very expensive. Consequently it is important to extract the maximum value from the data. The Institute operates a high temperature materials data bank (HTM-DB) for alloys which features a highly developed set of data evaluation programmes that maximizes the predictive usefulness of the data.

On ceramics, the corrosion mechanism for $Si_3N_4$ and SiC in sulphur containing atmospheres was elucidated, and work on the uniaxial testing of engineering ceramics contributed to European standardisation of testing procedures.

Creep and tensile testing of continuous fibre ceramic matrix composites (alumina fibres in SiN) has lead to a model to predict primary and secondary creep. Characterization of metal matrix Al-SiC composites stimulated ideas for a new composite produced by vacuum plasma spraying.

## Improvement of materials

The Institute invested heavily in a surface modification centre equipped with ion-implantation, laser, electron beam, plasma sputtering, and plasma spraying equipment together with the necessary complementary surface analysis equipment i.e. AUGER, ESCA, SEM, TEM, XRD, nanoindentor etc. This facility for improving materials surfaces has been used both for the production of ultra hard wear and corrosion resistant coatings and also in experiments in more innovative applications such as producing compact, cheap, and robust chemical sensors for gases such as $NO_2$ and $Cl_2$

Ceramics joining is another methodology which uses surface engineering. New developments involving ion beam amorphization of $Si_3N_4$ and implantation with Cr and Ni allows joining of $Si_3N_4$ parts with Ni-Cr interlayers: this yields strong and reliable joints.

Exploratory research projects may provide unexpected spin-off. Modelling of surface tension driven flows (Marangoni effect) in laser-melted liquid metal pools in laser surface treatment lead to the realization that the model also has potential for solving flux line erosion of refractories in the steel and glass making industries.

## Outlook into 1992/1994: The new Programme

The Institute specific programme falls into six research areas listed below:

- Materials for extreme environments
- Reliability and life prediction
- Measurement and validation methodologies
- Surface modification technology
- Fusion materials
- Materials information and data management

The focus is on advanced materials used to the limits of their capabilities. While continuing to build on past achievements, the new programme increases the emphasis on improving the performance of existing materials through surface treatments and coating. The industrial use of such materials in terms of market size is growing rapidly; these materials are part of a vital enabling technology of strategic importance for European industry in maintaining its position at the forefront of technology. We believe that this is one area where the IAM, as a public-funded materials research institute, can most efficiently contribute its effort for the benefit of European industry.

## Budget and Resources 1991

The table below lists the budget appropriations and the manpower resources allocated to each of the research areas during 1991.

| **Programme** | **Research Staff** | **Research Budget (Kecu)** |
|---|---|---|
| 1. Specific Programme | | |
| Materials | 77 | 1140 |
| Fusion Materials | 17 | 300 |
| Reactor Safety (PISC) | 21 | 320 |
| Other Specific | 3 | 40 |
| 2. Support to the Commission | 18 | 540 |
| 3. Exploratory | 9 | 190 |
| 4. High Flux Reactor | | |
| Complementary | 40 | 680 |
| Common | 2 | 100 |
| Totals | 187 | 3310 |

**Notes 1.** In addition to the above resources, 11 research staff were engaged in contract work for third parties. Contracts to a value of more than 3.5 Mecu were signed in 1991 for execution in 1991 and later years.

**2.** The research budget for the HFR excludes the reactor running costs.

## Research Personnel

There was a significant influx of younger staff, some recruits with mostly boursiers and visiting research fellows, about half of them from the newer member states: this is a vital element in the scientific spirit of the Institute.

## Coating Centre

The Advanced Coatings Centre (ACC) is a joint venture between the Netherlands Energy Research Foundation (ECN) and the IAM. Installation of equipment in this centre was completed and the centre now offers research facilities and expertise to industry for the development of coatings for severe environments.

A very wide spectrum of techniques is available which permits the optimum solution to be selected:

Thermal spraying, comprising: thermal spraying at atmospheric and low pressure for ceramic and metallic powders, high velocity spraying for carbides

Chemical vapour deposition: low pressure CVD for temperatures of 600-1200$^0$C, plasma assisted CVD for 100-600$^0$C, and plasma ion nitriding.

Physical vapour deposition: DC and radio frequency sputtering for low temperature deposition of metals and alloys, dual ion beam sputtering for ceramics.

High energy beams: Ion-implantation, electron beams for surface alloying and hardening, $CO_2$ laser for post-coating treatment (densification) of coatings.

For characterizing the coatings, the ACC can call on the established resources of the Institute.

## IAM Advisory Committee

The Institute Advisory Committee met for the second time in 1991. This committee has the remit to advise on scientific and technical matters relating to the work of the Institute.

The advice of the committee was sought on various materials initiatives stimulated by the Institute and on future strategy. The advice of the committee was useful in formulating the new multi-annual programme, and the emphasis on high temperature materials, fusion materials, life prediction and surface engineering was welcomed.

The Members of the Committee are:
Prof. H. Czichos, Dr. C. Djololian,
Dr. A. Garcia-Arroyo, Dr. G.W. Meetham,
Prof. C. Rizzuto and Prof. G. Sørensen.

## BNCT

The HFR is one of the few neutron sources in Europe with the potential for use in cancer therapy by the technique of boron neutron capture therapy, BNCT. The basis of this technique is to administer boron to patients in a chemical form that accumulates preferentially in tumour tissue. Exposure to a beam of epithermal neutrons then results in a higher damage rate to tumour tissue than to surrounding healthy tissue. The Institute BNCT project, which forms part of a coordinated European effort, advanced to the stage where tissue damage studies were begun in order to estimate dosage required for the treatment of brain tumours.

## Third Party Contract Research

Commitments for third party contract in 1991 amounted to 3.5 Mecu. This included a contract for 2 Mecu for the production of isotopes in the cyclotron in Ispra. The total volume of contract work is approaching the target of 4 Mecu/year set for the 1992-1994 programme. This comprises a large number of individual contracts covering a range of industrial sectors.

# II Scientific - Technical Achievements

# 1. Specific Programme: Advanced Materials

## Properties, Performance, Characteristics and Improvements of STRUCTURAL MATERIALS

# Alloys

This project aims to study the performance and deterioration of materials in simulated industrial environments by means of physically based modelling and experimental verification to predict behaviour in service. A further important requirement is to develop test and assessment methodologies suitable for industrial needs.

As in earlier years of the programme, the test and assessment methodologies activity has continued to absorb the major effort. Much of the scientific activity of the project has been carried out in cooperation with industrial and research institutes throughout Europe in the frame of COST 501 Round II, Joule, BRITE-I and BCR and in addition two scientists (from component and fatigue areas) have the responsibility to manage WP5 of COST 501 which involves coordination of testing and modelling activities of over 30 European research projects.

The project activities are described in terms of the principal type of degradation involved.

### a. Corrosion

In line with previous years, there has been a continuing trend towards obtaining a greater proportion of the Group's funding from external sources in the form of research contracts. This has resulted in a number of reports being written specifically for clients which in turn is reflected in the publication of a number of joint scientific papers.

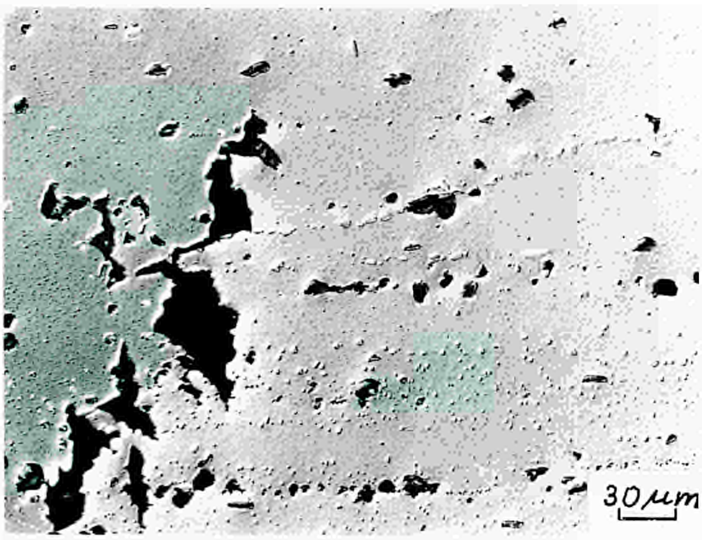
Within the specific programme, collaborative studies in the framework of COST 501/II progressed well and the validity of applying laboratory-derived data to corrosion experienced in real plant situations has been assessed. Test specimens exposed in a pilot coal gasification plant have been examined and compared with similar samples exposed in laboratory autoclaves using gaseous atmospheres which model those measured in plant. Four alloys were studied, i.e. AISI 310, Alloy 800H, Fecralloy and MA 956.

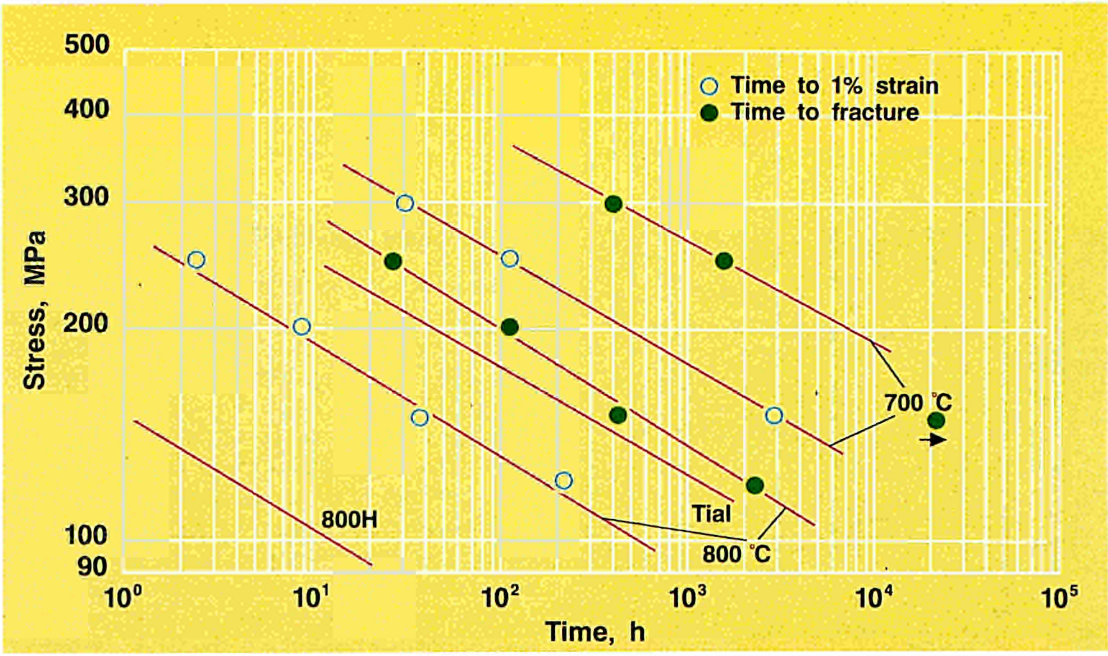
Sulphide scales had formed on the laboratory coupons, the thickness of which was significantly less for the Fecralloy and MA 956 alloys. This was associated with the formation of a much slower-growing $Cr_7S_8$ layer compared with the Fe and Ni-rich sulphide which had grown on the AISI 310 and Alloy 800H. The samples exposed in the gasifier at a similar temperature, however, were covered by oxide scales with evidence of internal sulphide precipitation. The relative behaviour of the alloys was the same as in the laboratory tests. The differences between the types of corrosion product formed in-plant compared with the laboratory experiments suggests that perhaps during the start-up of the gasifier, higher oxygen levels were present than was predicted by the earlier exposure of a group of reference metals/compounds.

Figures below and on the next page summarise in cross-section the corrosive attack experienced by the conventional austenitic AISI 310 and the more corrosion-resistant ferritic MA 956 in the two $H_2$-based laboratory environments containing 0.6% and $2\%H_2S$ and also in the plant atmosphere.

Collaborative studies between the Ispra and Petten sites of the Institute are continuing in an attempt to elucidate more fully the mechanisms by which Ce ion implantation improves the corrosion resistance of alloys exposed in these coal-gasification type atmospheres.

**Below:** *Creep damage in MA 760. T = 1050°C, tg = 4556 h.*






**Above:** *Time to 1% strain and time to fracture for $Ti_5Si_3/Ti_3Al$ at 700°C and 800°C. Examples for the time to fracture for Alloy 800H and TiAl at 800°C are included.*

## b. Creep

Much of the Specific Programme work was devoted to collaborative investigations within COST 501/II concerned with gas turbine alloys and materials for heat exchangers in coal gasification systems. Work has also been started on an intermetallic alloy in collaboration with the MPI in Düsseldorf (D).

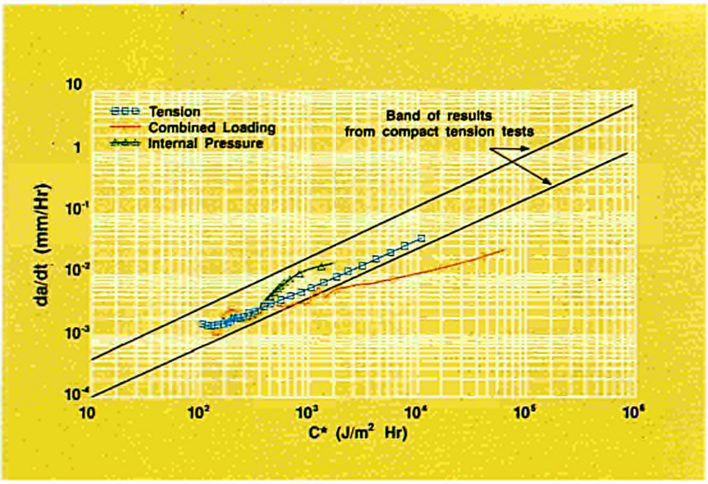
The ODS alloy MA 760 is considered as an advanced gas turbine blade alloy. Investigations at 1050°C have been completed with a microstructural examination of creep damage. Whereas samples taken transverse to the extrusion direction failed in a classical manner, i.e. by pore formation in the grain boundaries perpendicular to the stress axis, for samples from the longitudinal direction fast necking caused by the high stress sensitivity of the creep rate seems to be an important problem. Final fracture occurs by the accumulation of transverse cracks which, however, are not linked to transverse grain boundaries, Figure on page 10.

Further work on this subject will be concentrated on creep testing and microstructural analysis at 1150°C.

Studies with the heat exchanger material MA 956 have shown a systematic improvement of corrosion resistance to heavily sulphidising gases due to preoxidation.
Investigations have therefore been carried out at 600°C to determine the extent to which the scale can withstand mechanical deformation.
At low rates of creep strain, 0.5% strain seems to be critical for oxide failure and crack formation which is found to be accompanied by severe external and internal corrosion. This work will be continued for different gas compositions and also under conditions of thermal and mechanical cycling.

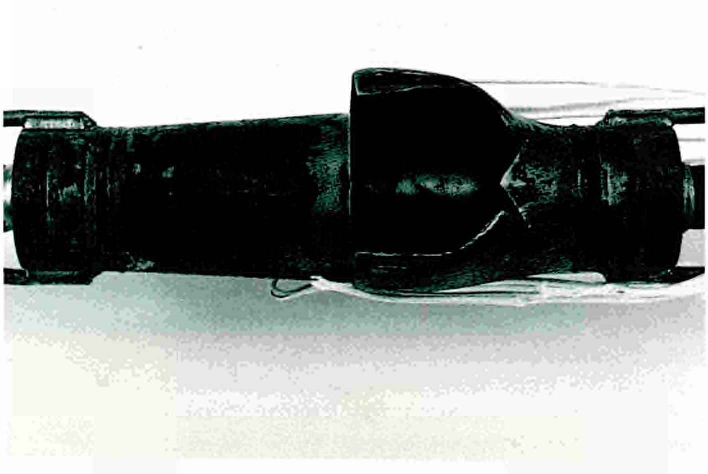
Creep and creep rupture investigations have been started on the intermetallic alloy $Ti_5Si_3/Ti_3Al$ at 700°C and 800°C.

Although this material has low ductility at temperatures below 600°C, at 700°C about 10% elongation to fracture is found under creep conditions combined with very promising strength properties, figure above. At 700°C a stress of 150 MPa is expected to correlate with a lifetime of about 50.000 h. Further work will be concentrated on structural studies in order to analyze in detail the microstructure-mechanical properties relationship.



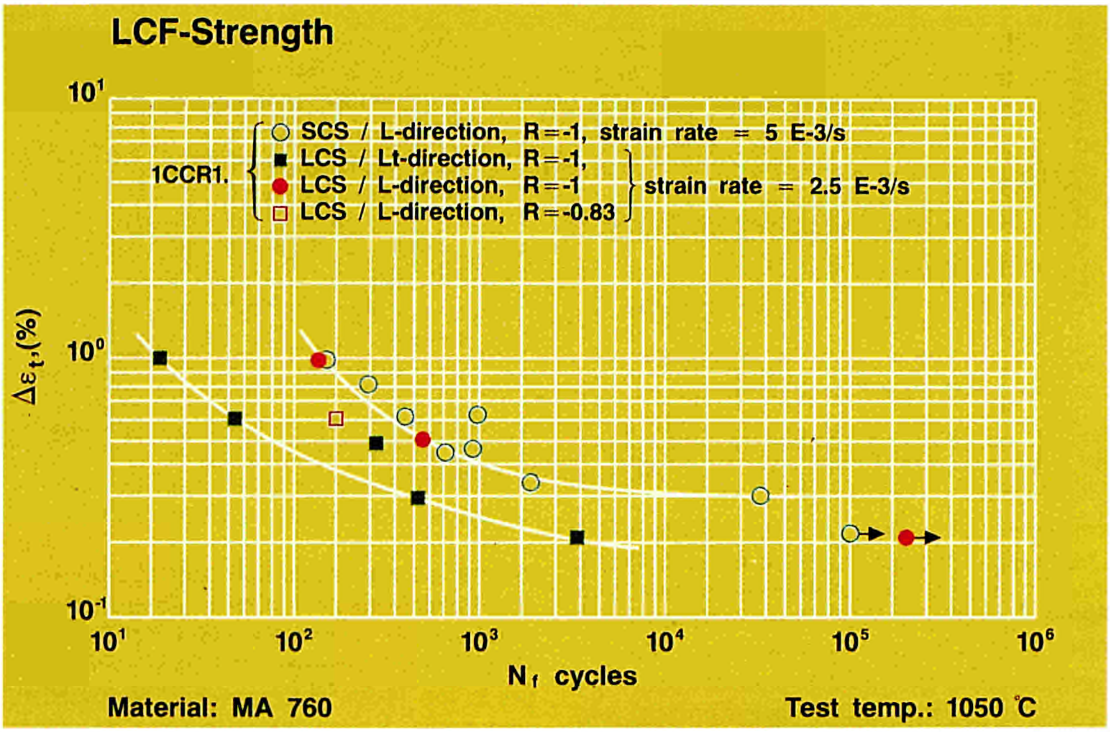

**Above:** *Creep crack growth curves for circumferentially notched 2¼ Cr1Mo tubular components under different loading conditions compared with compact tension test data base at 500°C*

**Below:** *Internally notched 2¼ CR1Mo steel tube failed under internal pressure at 600°C*



## c. Component Behaviour

All the work in collaboration with COST 501-II WP5 has concentrated on a 2¼Cr1Mo steel with the test pieces being removed from a 70 mm wall steam pipe. Multiaxial creep tests have been carried out under axial, internal pressure or combined loading conditions on tubular components at 550 and 600°C. By using uniaxial creep data supplied by other partners in the project, predictions of the strain development under multiaxial stresses could be deduced from the previously developed continuum damage mechanics based model. Creep crack growth measurements on the same alloy have continued using the potential drop method. Tests have been completed on both longitudinally and circumferentially notched tubular components stressed through tensile and/or internal pressure loading. In the analysis of the data, a limit load analysis is applied in order to derive the C* integral as the creep crack develops thereby enabling a successful juxtaposition of the data determined directly on components with that obtained conventionally from compact tension specimens as shown in figure above. The parallel programme on creep crack growth in ferritic steels for hydrogen service has advanced on two fronts. Firstly, the basic creep crack growth data in air has been provided from miniature compact tension specimens and a special rig has been designed and is under construction for testing identical specimens in a 200 bar $H_2$ environment. Secondly, baseline tests have been carried out on components containing internal defects under internal pressure, a failed test piece being shown in figure below. The potential drop technique has been adapted for this situation which will facilitate experiments where defects are exposed to the high pressure hydrogen environments under conditions close to those observed in petrochemical plant. The study of oxide dispersion strengthened materials for high temperature heat exchanger applications has suffered delays due to lack of this special tubular test material supplied by a COST 501 collaborator, and difficulties in sealing tubes for internal pressure testing. Three potential joining methods are being examined in this respect, namely electron beam welding, diffusion bonding and explosive welding. This aspect is not only critical for the testing programme but also for the high temperature heat exchangers constructed with this advanced material.



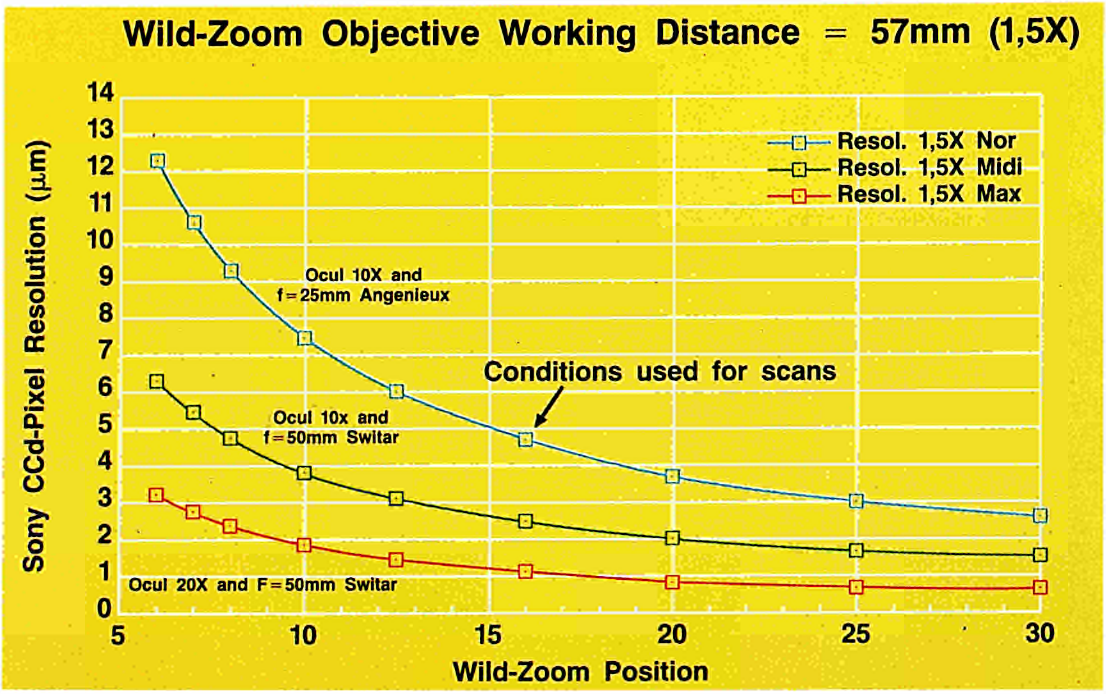

**Above:** *Isothermal, low cycle fatigue lives of MA 760 at 1050°C*

## d. Fatigue

The fatigue behaviour of the ODS alloy MA 760 was investigated at 1050°C in the framework of COST 501-II to generate a data base and to analyze the damage accumulation and failure mechanisms under isothermal-LCF and thermo-mechanical fatigue conditions, figure above.

Development continued of the computer vision system for the in-situ monitoring of the initiation and growth of microcracks on the surface of samples while being mechanically tested. The disks used for storage of the acquired images were upgraded to 200 MByte capacity, corresponding to 700 images. Since one scan represents 30 images, the storage capacity of a disk is enough for 23 scans, allowing one complete mechanical test to be monitored in a fully automated mode without any intervention by the operator. Software improvements were also made to the system.
The system is being used in a project investigating the thermo-mechanical fatigue behaviour of bare and coated single crystal nickel based alloys for aero-gas turbine applications.
An evaluation has therefore been made of its potential for detecting and measuring microcracks by comparing its accuracy with that achieved by the traditional replica method and direct optical observations. The comparison was made at the level of resolution used for the scan mode, see figure on next page. The computer vision system turned out to be the most accurate technique for monitoring microcracks.

A universal testing machine programmed for the uniaxial cyclic testing of alloys and ceramics at high temperature under C-O-S type corrosive environments has been equipped with an environmental chamber and with the necessary control and safety instrumentation. Commissioning is scheduled for early 1992. A new activity aimed at modelling the lifetime and the constitutive behaviour of single crystal nickel-base alloys was launched. Work in the preliminary phases of the project focused on familiarisation with the concepts of micro and macromechanics modelling.




**Above:** *Pixel resolution of the computer vision system vs. zoom position of objective lens.*

In addition to its direct involvement in the conduct of research within COST 501, the Institute also provides the Secretariat for this large collaborative programme. In COST 501-II 12 countries plus J.R.C.-I.A.M. are contributing more than 500 my effort with a value of about 42 MEcu over a three year period. One third of the 197 participants receive no additional financial assistance from national sources, taking part in the collaboration in order to profit from the larger effort which can then be directed towards the selected critical materials problems in power engineering components. The secretariat is responsible for handling the information flow to the Management Committee, and the 10 Work Packages to the Technical Committee - Materials and other bodies. It also provides technical editorial facilities for the publication and circulation of progress reports, study contract reports, etc., which are then handled by the relevant services in Brussels and/or Luxembourg.

Since Round II is planned to be completed by end 1992, the secretariat was deeply involved during 1991 in the formulation of the application to be made to the COST Committee of Senior Officials for a Round III, planned to start in 1993.

# Engineering Ceramics

The technology of processing engineering ceramics has made considerable progress, to the extent that ceramic materials are now seriously considered for thermomechanical applications and attempts are being made to design with ceramics. Such ceramic materials have to operate under severe conditions of temperature, corrosion and mechanical stress.

In the light of this industrial/technological relevance, the JRC ceramic materials effort is directed to studying the corrosion and mechanical properties of non-oxide ceramics at high temperatures. Special attention is devoted to the understanding of the degradation mechanisms by in depth microstructural investigations, particularly for failure analysis and component life-time prediction.

The engineering properties relating to ceramic/ ceramic joining, ceramics machining and non-destructive evaluation are also studied.

The research on mechanical properties has been focused on the characterisation of ceramic materials, the mechanisms of deformation and of damage and of failure in relation to microstructure. The work was carried out at temperatures up to 1300$^0$C in inert and corrosive gaseous environments on silicon nitride and carbide ceramics and composites.

The corrosion studies at temperatures up to 1300$^0$C on newly developed silicon nitride materials have been done with the purpose of elucidating the fundamental gas and hot corrosion mechanisms.

High quality ceramic joints have been developed for operation at high temperature, in corrosive environments and mechanical stress, simulating industrial environments.

Ceramics machining techniques have been studied to minimize damage effects in the materials and the relationship between mechanical properties and the machining technology and microstructure has been clarified. Reliable test specimens of ceramics and composites can now be prepared.

The developments in the field of non-destructive testing for ceramics and ceramic materials and composites have been studied and investigations made for introducing some new techniques in our research work.

The research activities are well integrated in the CEC-ceramics programme e.g. Science, or in direct collaboration with Universities, Research Centres and Industry.

The unique test facilities and the expertise available formed the nucleus of
- industrial contract research;
- support to the Commission Services;
- exploratory research activities.

The research and development work has spin-off's in several modern technologies i.e. Aerospace and Aeronautics, Automobile Industry, Energy Sector, Petrochemistry, Mechanical engineering etc.

The results of the ceramic project are reported per discipline:
- Interfacial Engineering,
- Corrosion Properties.
- Mechanical Properties,

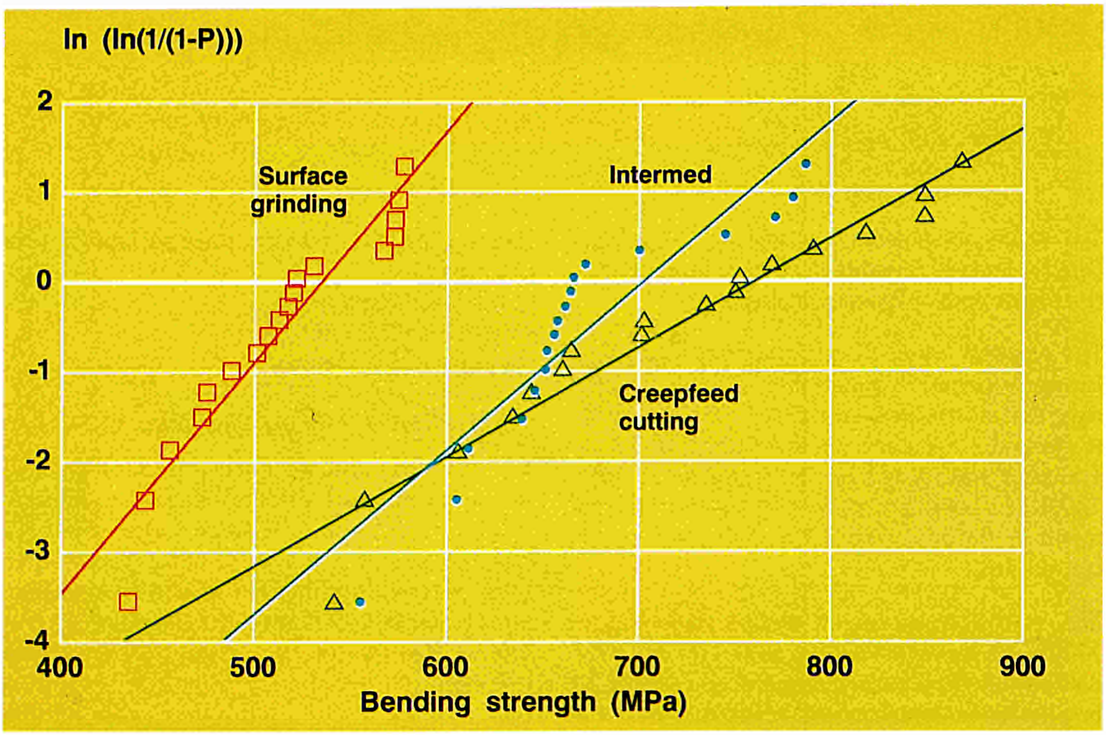
**Residual Stress in Ceramic Surfaces**

Following the establishment of a reliable technique for measuring residual stress states in machined ceramic surfaces using X-ray diffraction, a project has been conducted with the ECN (NL) laboratory to study the viability of the technique as a nondestructive tool for determining critical (life reducing) machining damage in hot pressed silicon nitride (HPSN).

HPSN samples were machined under 3 standard conditions of pendular grinding (small down feed, rapid table speed), creepfeed grinding (large down feed, slow table speed) and an intermediate condition, all giving the same material removal rate. A grinding parameter P, taking into account the various grinding factors, was calculated for each condition and related to final material bending strength, fracture probability and to residual stresses measured in the specimen. Stress tensors were calculated for the three machining conditions as shown in figure below.

**Below:** *The stress tensors are calculated to be:*

| | | | |
|---|---|---|---|
| | -152 | -110 | 14 |
| Creep feed cutting: | | -17 | 33 |
| | | | -39 |
| | -155 | -125 | 23 |
| Intermediate Mode: | | -72 | 27 |
| | | | -58 |
| | -359 | -269 | 27 |
| Pendular Grinding: | | -175 | 32 |
| | | | -124 |




**Above:** *Bending strength versus machining condition of the HPSN.*

The characteristic bending strength for each machining mode was determined applying the Weibull model to data from 18 bend tests per conditions (figure above), yielding values of characteristic strength as given in table below.

Figure on the next page shows a linear dependence of P, the grinding parameter, on the sum of the relevant bending stresses and on the Weibull bending strength. Clearly, the residual stresses reflect severity of grinding forces. However, surface stresses are compressive and should increase fracture strength. The observed systematic reduction in strength points to the tips of induced cracks lying in the zone of tensile stress underneath the compressive layer. Pre-fracture crack lengths in this study are estimated to be 30-55µm while the transition from the compressive to the tensile stress state occurs at about 25µm below the surface. The Weibull plots of the 3 machining conditions support this hypothesis. Whereas low-P grinding gives higher mean strengths, the range of fracture strengths is much greater, suggesting that the crack length distribution is much broader and still includes a number of cracks of dimensions similar to those obtained with high P grinding. Clearly, before residual stresses can be used to reliably predict the strength of ceramics, the distribution of fracture inducing flaws will need to be better known.

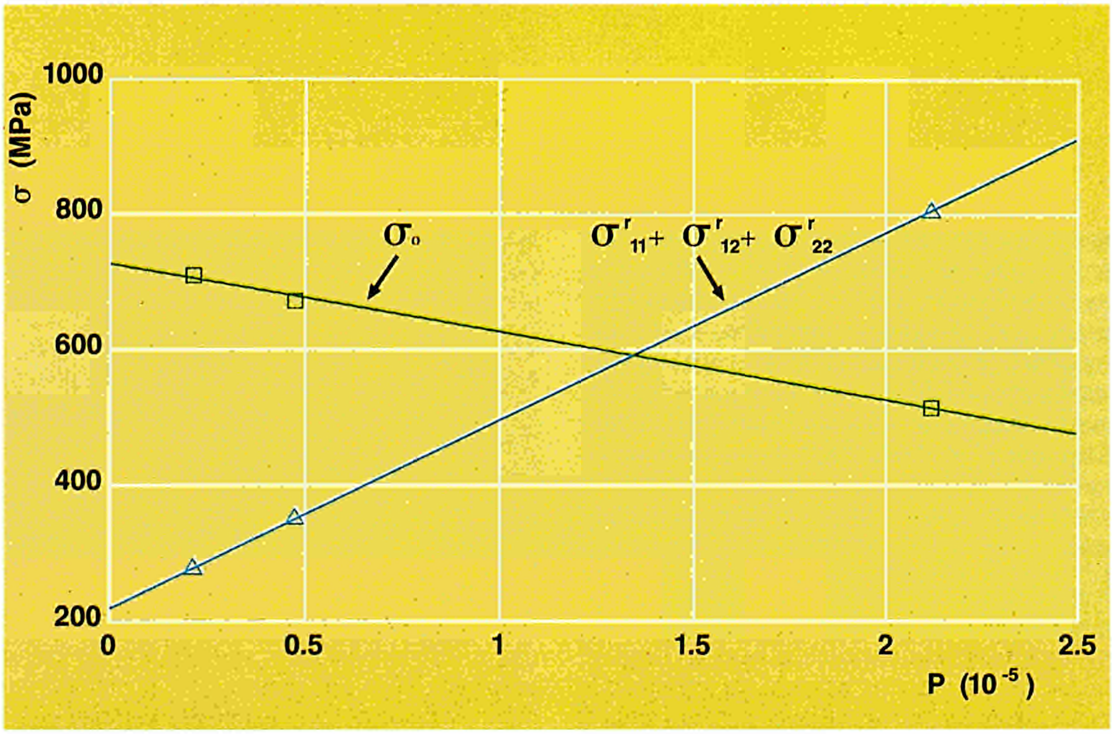
The characteristic (bending) strength for each machining mode is determined by applying the Weibull model to 18 bending strength measurements per condition. Figure above shows the Weibull plot and the linear regression lines through the data-points.

Table below shows the calculated values of the Weibull characteristic strength the Weibull modulus m and the mean strength $\sigma_m$, which is the weighted average of the measured bending strengths.

The relation between the characteristic strength, σ, from table below and the grinding parameter P from table on page 7 is shown in the figure on page 9. Also shown in this figure is the absolute sum of the three stresses working directly on cracks perpendicular to the surface: $|\sigma_{11}$, $\sigma_{12}$ and $\sigma_{22}|$.

**Table below:** *the calculated values for $\sigma_o$, m and $\sigma_m$.*

| Machining Mode | m | $\sigma_o$ MPa | $\sigma_m$ MPa | $\delta(\sigma_m)$ MPa |
|---|---|---|---|---|
| Creep Feed Cutting | 7.4 | 757.6 | 715.7 | 46.6 |
| Intermediate Mode | 9.2 | 691.4 | 672.6 | 29.5 |
| Pendular Grinding | 10.3 | 534.4 | 513.8 | 21.6 |




**Above:** *The relation between the machining parameter P end both the Weibull bending strength $\sigma_o$ and the measured stresses along the surface l $\sigma_{11} + \sigma_{22} + \sigma_{12}$ l.*

The other three stresses are all perpendicular to the direction of the bending stress, so it is assumed that these stresses have only a small influence on the strength of the material.

## Stress Gradients in Machined Ceramics

A parallel study into the definition of stress gradients in machined ceramic surfaces has been conducted using X-ray diffraction and foil bending techniques. Progressive increase in the angle of incidence of the X-rays produces a corresponding increase in the depth of the excitation zone and allows determination of residual stresses through the subsurface.

Similarly, the machining of a polished and fully annealed foil on one side only, induces compressive surface stresses on the machined side which bend the foil. A simple model based upon an exponential relationship between residual stress and depths from the surface correlates well with experimental data from both techniques.

## Ceramics Corrosion

High temperature corrosion studies of engineering ceramics is an on-going activity concentrating on non-oxide ceramics (silicon carbides and nitrides) and having as its prime objective the study and evaluation of the mechanisms and kinetics of gaseous corrosion in atmospheres of industrial importance. During 1991, activities have concentrated on further testing and evaluation of the thermodynamic model devised earlier to explain and predict the corrosion resistance behaviour of silicon nitrides in sulphur-containing atmospheres of low oxidation potential. Under these conditions, according to the model, the formation of a pseudo-stable protective layer of silica is possible in equilibrium with the environment. Dramatic improvements in the corrosion resistance are possible compared to atmospheres in which the oxygen partial pressure is below the limit for the formation of silica. This is illustrated by a comparison of the linear rate constants for both conditions (see table below).

**Table below:** *Comparison of corrosion rate constants (weight loss in µg $cm^{-2}$ $h^{-1}$) for two silicon nitrides in $H_2$-$H_2S$-$H_2O$ mixtures at 1300°C*

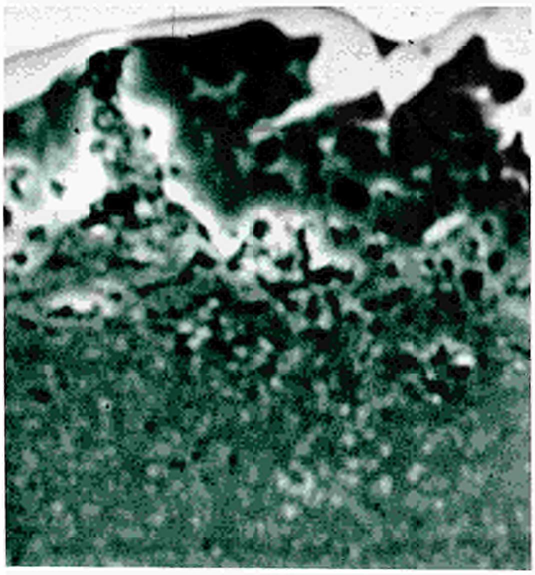
| | $H_2$ | $H_2$-$H_2O$* | $H_2$-$H_2S$** | $H_2$-$H_2S$-$H_2O$ |
|---|---|---|---|---|
| HPSN | 189 | 8 | 1350 | 11 |
| HIPSN | 166 | 5 | 2500 | 10 |

* 0.7 vol% $H_2O$ added ** 0.4 vol% $H_2S$

In the case of silicon nitrides, the 'protective layer' is usually not pure silica but rather a mixture of silica, silicates and a glass, the latter two formed by reaction of cations (e.g. Y, Al) derived from secondary, intergranular phases. These phases are present as a result of the addition of metallic oxides to aid densification of silicon nitride via a liquid phase. Stratified differentiation of the 'protective layer' takes place with time leading to a situation where the silica is concentrated at the boundary of the bulk ceramic (see figure below).

It would appear that little or no direct reaction occurs between this layer and sulphur, in the form of hydrogen sulphide, as sulphides have not been detected on the outer surface and are presumed to occur below the 'protective layer' only as a result of transport through cracks or along grain boundaries. In order to investigate this further, the protective film formed on high purity silicon carbides under the same conditions will be studied.

**Below:** *Cross-section of HPSN after 300h in $H_2$-0.4%$H_2S$-0.7%$H_2O$ at 1300$^0$C showing stratified nature of corrosion layer.*



In addition the behaviour in similar environments of pure silica and of thin silica films grown under oxidising conditions will be included.

The study launched at the end of 1990 to investigate the behaviour of typical intergranular phases of silicon nitrites in oxidising/suphidising environments has progressed to the stage of the preparation of a number of selected materials. Three phases from the Si-Y-O-N system commonly encountered in silicon nitrites densified with yttrium(III)oxide and their cerium analogues (H-phase, $M_{10}(SiO_4)_6N_2$, with apatite structure; J-phase, $M_4Si_2O_7N_2$; and K-phase, $MSiO_2N$, with $\alpha$-wollastonite structure) have been selected and are being prepared. Initial experiments have confirmed the rapid oxidation of these materials to simpler silicates at temperatures at which silicon nitride itself is relatively unaffected. During 1992, these studies will progress to sulphur-containing environments as well as establishing the kinetics of the oxidation/sulphidation reactions.

### Mechanical Properties of Monolithic Ceramics and Ceramic Matrix Composites

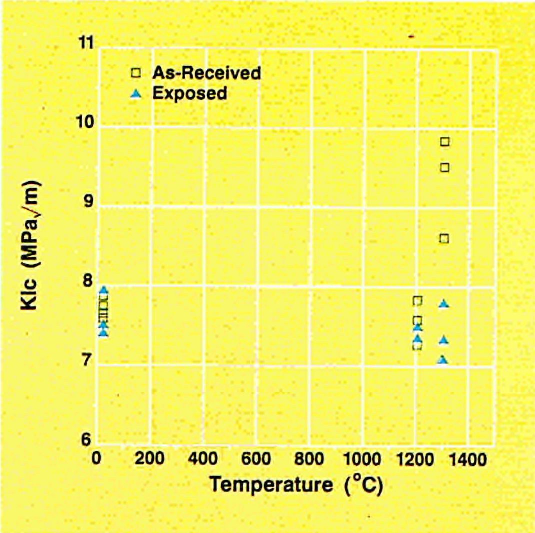
The research activities concentrate on the characterisation of the mechanical properties and on the study of the mechanisms of deformation, of damage and of failure in relation to the microstructure. Two material classes with potential of application as high temperature structural materials are studied, viz. monolithic ceramics and continuous fibre reinforced ceramic matrix composites.

The investigated monolithic ceramic is a hot-pressed silicon nitride which had been previously studied under uniaxial cyclic push-pull fatigue at 1200$^0$C in air.

The current work focuses on the toughness and subcritical crack growth behaviour after an exposure simulating the anticipated corrosion conditions in industrial coal gasification environments.

The residual strength, toughness and subcritical crack growth at room temperature, 1200$^0$C and 1300$^0$C have been characterised in flexure type tests after prior exposure at 1200$^0$C and at 1300$^0$C and compared to the properties of the as-received material.

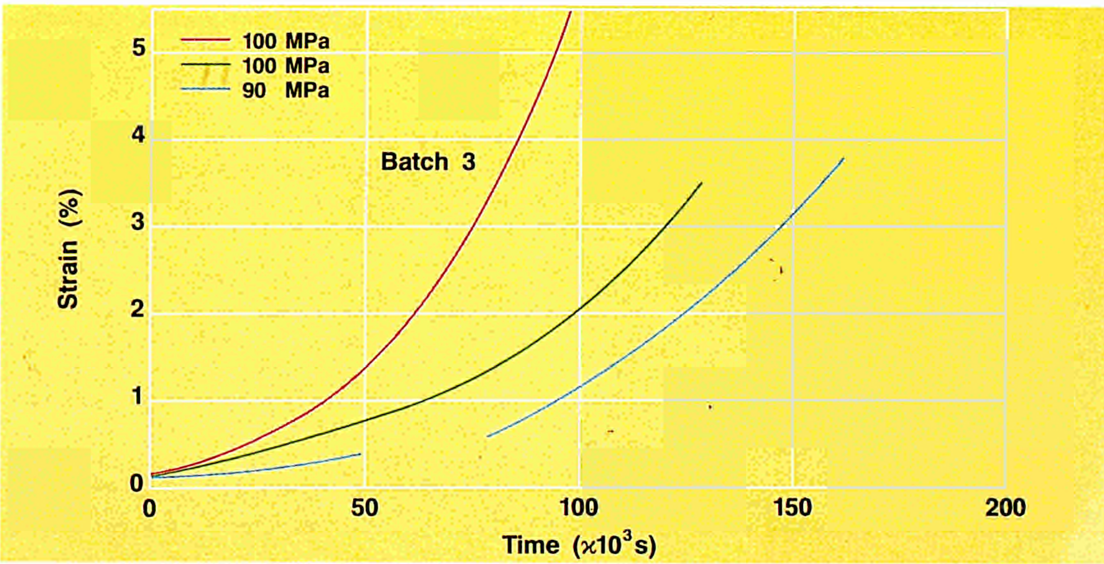
For some loading conditions drastic changes occur as observed e.g. for the temperature dependence




of the toughness, shown in the figure above. Preliminary microstructural investigations indicate that the observed changes in behaviour can be explained by a partial crystallization of the amorphous glassy phase at the grain boundaries.
The ceramic matrix composite consists of a bidirectional reinforcement of cross woven alumina fibres in a silicon carbide matrix obtained by chemical vapour infiltration. The most attractive feature of continuous fibre reinforced ceramic composites is their "ductile" failure, which only prevails for a limited range of interfacial properties. At high temperatures, oxidative attack modifies the interfacial characteristics and thus obstructs the study of the intrinsic mechanical behaviour. Within this research project, which focuses on the long term creep behaviour, this is avoided by testing under high vacuum with oxygen partial pressures below 1.E-9 mbar. Moreover, in order to facilitate the evaluation of the complex effects of time dependent stress distribution over the different constituents of the composite, the specimens are tested under a macroscopically homogeneous uniaxial stress state. Creep tests have been performed in the temperature range from 950ºC to 1100ºC at stress levels above the first matrix cracking stress. For all the tests, a three stage creep curve is observed, with rupture strains in some cases exceeding 5 percent (figure below). A model was constructed that describes and subsequently predicts the primary and secondary creep stages as a function of stress.
Uniaxial creep testing on the composite material will be complemented by a push-pull fatigue programme. During the reporting period, a vacuum chamber has been designed and partly installed on the hydraulic machine that will be used for this programme.

**Above:** *Change in the toughness with temperature for $Si_3N_4$ as received and exposed to coal gasification type corrosive environment*

**Below:** *Creep curves for the 2D ceramic Matrix composite $H_2O_3$/SiC at 1100ºC*



**Ceramic Components**

During 1991 a new project was initiated which aimed at studying the behaviour of ceramic components for advanced heat exchangers.
A literature survey pointed to the selection of silicon carbide as the ceramic with the most potential for application as a tubular heat exchanger material.
The same survey revealed that knowledge of both mechanical and thermal properties would be essential to the designer and operator of plant containing such components.

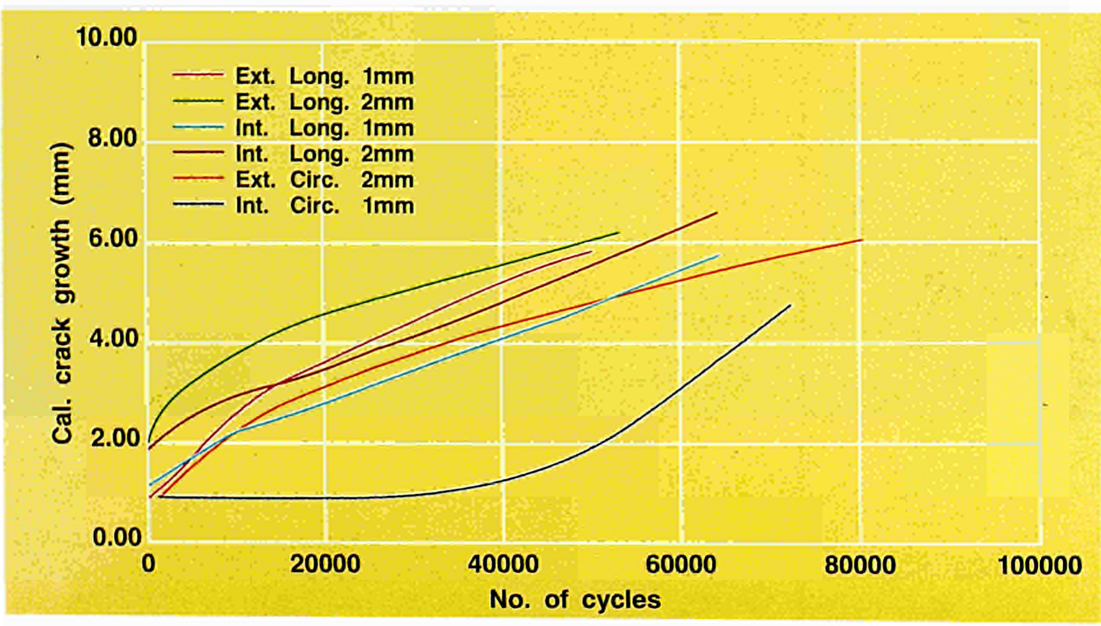
It was concluded that a test facility should be designed and constructed which allowed internal pressure and end loading of tubular test-pieces with the added possibility of thermal cycling. By the end of the year the existing test facilities have been upgraded to provide test temperatures up to 1400°C.
In order to design test-pieces which can retain pressure and allow the transmission of end loading work has started to develop suitable joints. This "joining" aspect forms not only an intrinsic part of the present project but must also be addressed for the successful application of the ceramic heat exchangers in high temperature plant.

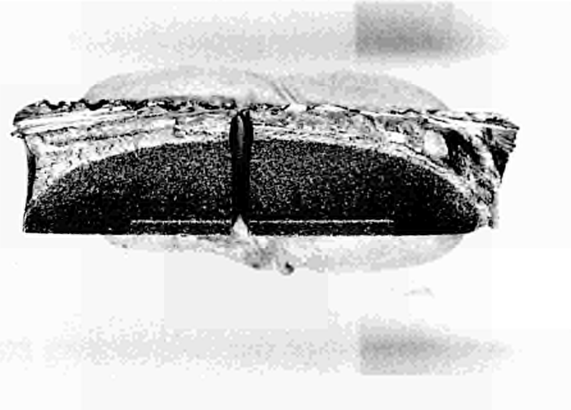
# Components and Thermal Fatigue

The project aims at the numerical modelling and experimental verification of the life spent in propagating a crack to failure in components exposed to cyclic thermal gradient fields. An irradiation damage effect is included in the study to simulate the conditions seen by the first wall of NET.
The out-of-pile rig for thermal cyclic testing has been extensively exploited. 316L stainless steel tubular components containing various notch geometries have been subjected to the 80°C/350°C thermal cycle typical of the first wall of NET for lives ranging from 7000 to 80.000 cycles.

Thermal fatigue crack growth from longitudinal and circumferential notches has been derived from continuous DC potential drop measurements using calibration curves based on actual measurements of failed sections beneath potential drop probes (figure below).

**Below:** *Calibrated crack growth rates for various notch geometries*





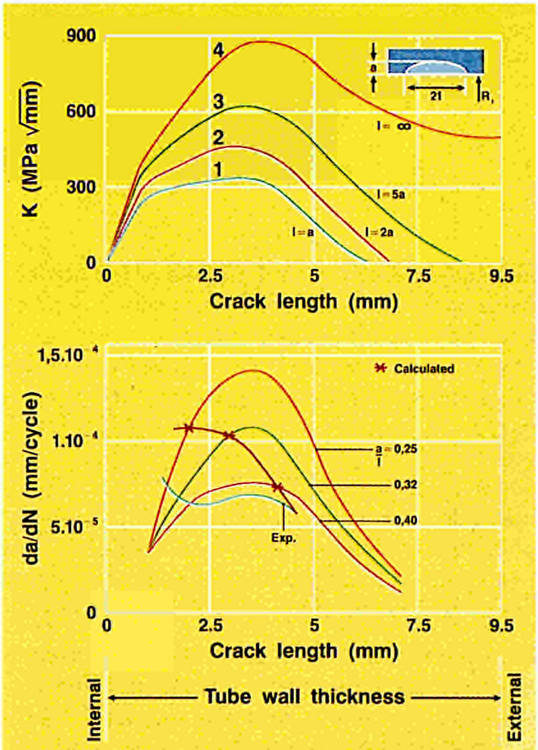
Initiation of crack growth takes place almost immediately for all but the internally circumferential notched tubes while the rates of crack growth are apparently little influenced by the starter notch geometry and depth, see figure above.

The computer code PREDI-N, developed in the reporting year, predicts the crack growth rate of longitudinal, semi-elliptical cracks of arbitrary shape in tubes subjected to cyclic thermal gradient fields. Temperature and stress-strain fields are calculated in time and in space by means of a finite element formulation, using temperature dependent material data. The stress-strain module is based on a combined isotropic-kinematic hardening rule which accounts for the Bauschinger effect. Generalized plane strain conditions are treated by a two pass solving technique. Stress intensity factors are evaluated by means of the code CRACK. The predictions are based on a Paris crack growth law. The shape of the stress intensity factors and of the crack growth rates of internal, longitudinal semi-elliptical cracks in a cyclic temperature field of 80⁰C-350⁰C were calculated, see figure below. Due to the severe loading the tube deforms plastically over distances of about 2 mm from the internal and the external surfaces. It is planned to also include an elasto-plastic formulation of the crack growth law in the code in future in order to enable prediction of crack growth rates in plastified regions. The large effect of the crack shape a/l on the growth rate is to be noted in the figure below. This emphasises the importance of measuring the growth rates at several locations along the crack front in the tube validation experiments. The crack shape a/l changes during the growth of the crack, evolving from a/l=0.25 to a/l=0.4 in the example of the figure below. If this is taken into account the prediction and the experimental data correlate quite well in the LEFM controlled region.

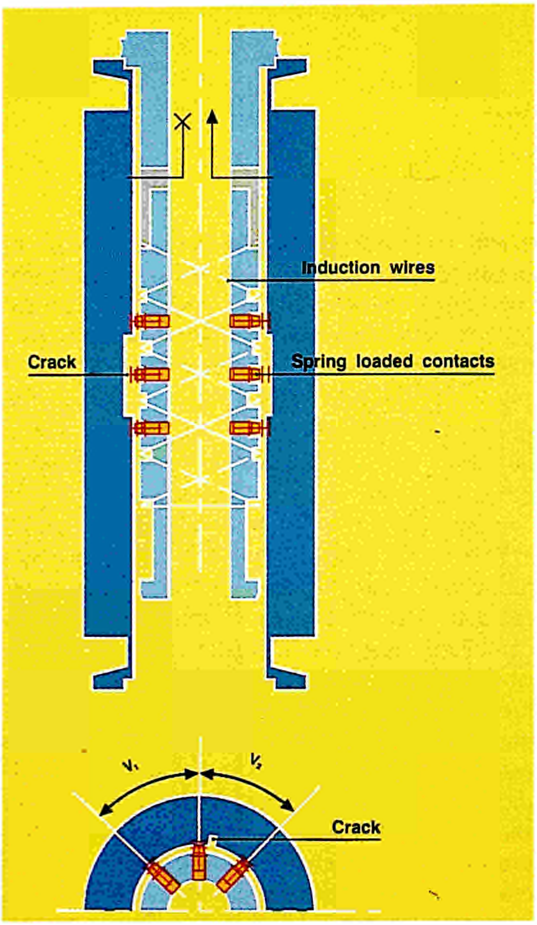
In the reporting year fracture mechanics experiments to determine the constants of the Paris crack growth law at different temperatures on the 316L SS material for NET continued, using CT samples. The effort on the in-pile rig concentrated on the selection and the assessment of the technique for the measurement of the crack growth rates under neutron irradiation. It was found that the DCPD technique was not suitable because of the high currents involved.

**Above:** *Shape of thermal fatigue crack growing from an internal spark eroded notch of 1 mm depth and 15 mm length.*

**Below:** *Stress intensity factors (top) and crack growth rates (bottom) as a function of crack length in a tube wall for internal, semi-elliptical, longitudinal cracks.*
*The bottom figure compares predicted growth rates for a crack with a changing a/l ratio with experimentally measured values.*



The AC potential drop technique was selected and the corresponding measuring equipment purchased. An ACPD probe for validating and calibrating the technique on the out-of-rig was designed and manufactured, see figure above.




**Above:** *ACPD probe design for the measurement of crack growth rates in tubes in a neutron irradiation environment.*

# Operational Defects in Materials and Lifetime Prediction

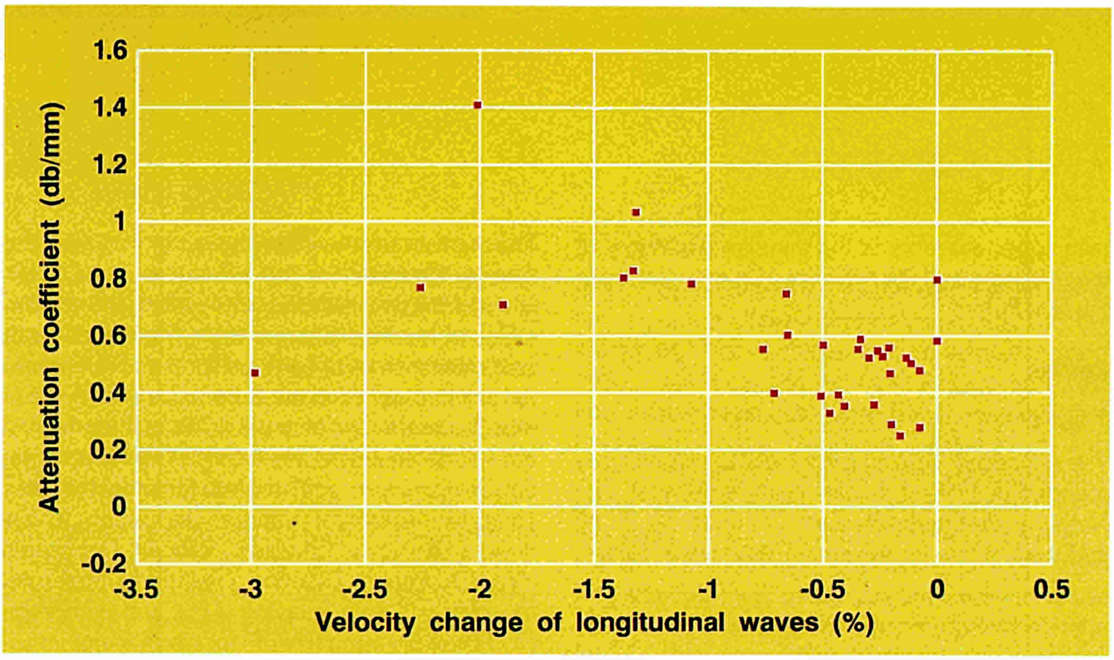
Remanent life assessment for pressurised components operating at elevated temperatures requires knowledge about the degradation of the material. The aim of this project is to improve the damage measurement methods for a quantitative characterisation of the internal defect state which determines the lifetime and performance of components under creep loading conditions. Ultrasonic (US-) methods backed up by quantitative metallographic studies and hardness measurements have been applied to different materials for a quantitative determination of the creep damage incurred.

Research efforts in 1991 concentrated upon completing the measurements (US-velocity, metallography) on the Mn-Cr austenitic steel AMCR 0033 and verifying the results obtained so far.

In addition, the applicability of ultrasonic attenuation measurements for the detection of creep damage has been investigated.

In this steel, creep damage in the form of intergranular microcracks is observed and a damage variable related to the density and size of the microcracks can be identified.

The relationship of the damage variable to non-destructive ultrasonic measurements and metallographic findings has been established so allowing the direct comparison of quantitative metallographic and ultrasonic results, which yields the conclusion that for this type of creep damage US-velocity measurements are very sensitive to microcrack formation whereas the attenuation measurements




show considerable scatter (see figure above). The consistency of the measurements with predictions from material models could be demonstrated. The results allow one to assess the applicability of ultrasonic measurements to other types of microstructural damage, i.e. cavity formation on grain boundaries and to draw conclusions concerning the advantages and limitations of metallographic and ultrasonic techniques for damage determination.
The investigations in other materials have been continued. Preliminary metallographic and ultrasonic studies were performed on a bar of MA 6000, as supplied by INCO Alloys.

**Above:** *Correlation between the attenuation coefficients (US-frequency = 10 MHz) and the longitudinal wave velocity changes in creep damaged AMCR 0033 specimens. Whereas velocity changes of more than 0.3% clearly indicate creep damage, considerable scatter is observed in the attenuation measurements.*

# Wear and Corrosion Resistant Coatings

### Introduction and Objectives

Lifetime and load capacity of tools and other structural components depend both on how the particular material withstands wear as well as on the environmental conditions the part is exposed to. For this reason, protection against wear and corrosion is of great economic importance.

The development of protective coatings has become an important branch of advanced material technology in recent years. So far, considerable progress has been made: wear resistant coatings on various tools lead to prolonged lifetimes combined with higher performance: corrosion resistant coatings are in many cases prerequisites for successful operation of parts in oxidising and corrosive environments at high temperatures.

The activity "Wear and Corrosion Resistant Coatings" has the objective of contributing to an improvement of existing coatings and to the development of new coatings. In particular, it is intended to combine the surface modification techniques of the laser/implanter foundry with the deposition techniques available at the thin film laboratory of the IAM Ispra and the Advanced Coating Centre of the IAM Petten.

## Results

### 1. Creation of the Advanced Coating Centre

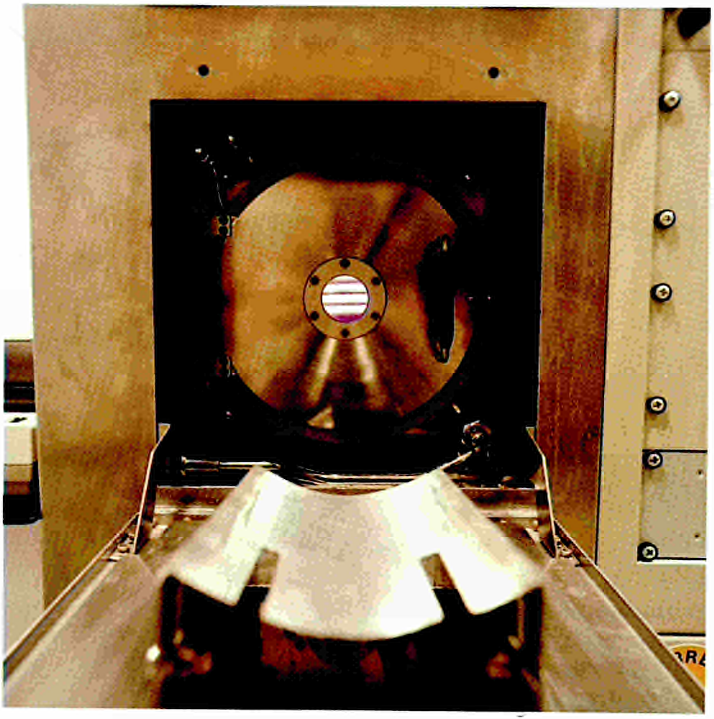
The Advanced Coating Centre (ACC) was officially founded in 1991 as a Joint Venture between The Netherlands Energy Research Foundation (ECN) and the Institute for Advanced Materials at Petten.

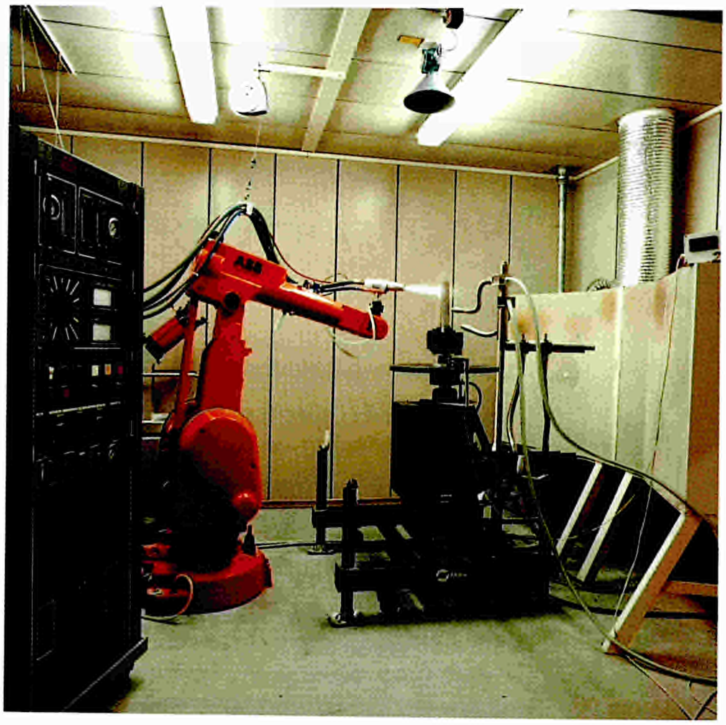
**Below left:** *Plasma Assisted Chemical Vapour Deposition equipment. This machine can be used to produce hard coatings in the 400 to 600°C temperature range, hence significantly lower than the temperatures needed for thermal processes.*

**Below right:** *High Velocity Oxygen Flame Spraying process uses the combustion flame of oxygen and propylene or acetylene to melt ceramic and metallic powders. The torch is designed in such a way that the velocity of the droplets reaches Mach 2, thereby producing high density coatings.*

The market targets where chosen to maximize the synergy between the pre-existing areas of excellence of the two institutes involved: clearly, the domains of mechanical application, wear and corrosion resistance were selected for their high potential development and the experience already accumulated in the two institutes. The coating techniques to be employed were chosen for their potential development and interest for industry. They comprise: Chemical Vapour Deposition (CVD), Plasma Assisted Chemical Vapour Deposition (PACVD), Plasma Diffusion Treatments (PDT), Atmospheric Plasma Spraying (APS), and High Velocity Flame Spraying (HVOF) for which major industrial developments are foreseen. It is also expected that an investment in Low Pressure Plasma Spraying (LPPS) or Controlled Atmosphere Plasma Spraying (CAPS) will have to be made in the coming years to support the R&D efforts in automotive and aircraft industries for which this technique is of strategic importance.

One of the focal points in the development of the ACC will be the in-situ diagnostics for Plasma Assisted Deposition Processes. For that purpose, a Langmuir Probe and Optical Emission Spectroscopy





for in-situ plasma diagnostics, as well as a quadruple to keep track of the concentration of the reactive species in the reactor during the chemical vapour deposition processes have been ordered. The ultimate aim is to relate the plasma characteristics to the final properties of the coatings (microstructure mechanical properties), and to develop, at a later stage, online intelligent process control of the CVD and Spraying processes.

The laboratory construction for the ACC started in 1991 and will be finished by the end of March 1992. Most of the machines were delivered in the second half of 1991, and the installation was started up.

First experiments with Plasma Assisted Chemical Vapour Deposition (figure left on page 24) of TiN and $Si_3N_4$ films were made in December 1991, leading to several modifications of the PACVD machine. The Hot Wall CVD equipment was started during the same period and preliminary TiN deposition tests have been made.

The PDT machine was delivered and assembled in November 1991. Tests of the Plasma Spraying installation and High Velocity Flame Spraying equipment (figure right on page 24) were performed in the last quarter of 1991. Spraying of WC, $Al_2O_3$, $ZrO_2$-$Y_2O_3$, NiCr has been performed and this equipment is now fully operational. The test equipment and in-situ diagnostics systems will be delivered before summer 1992.

The ACC is constituted as a business unit, and will work on R&D projects for the benefit of ECN and IAM, but also offers to industry research facilities and expertise to support the development of new coatings for materials operating under severe conditions.

The research programmes of ACC in 1992 are initially based on two internal R&D projects from ECN, one project from IAM, and one third party contract.

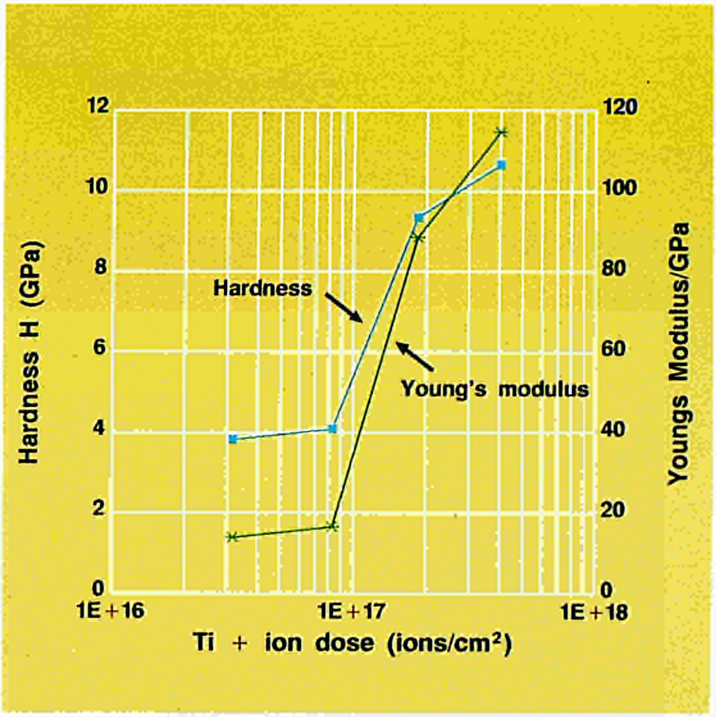
The development will take 3 to 4 years in order to develop basic knowledge on the processes as well as to take into account pre-industrial aspects of the development.

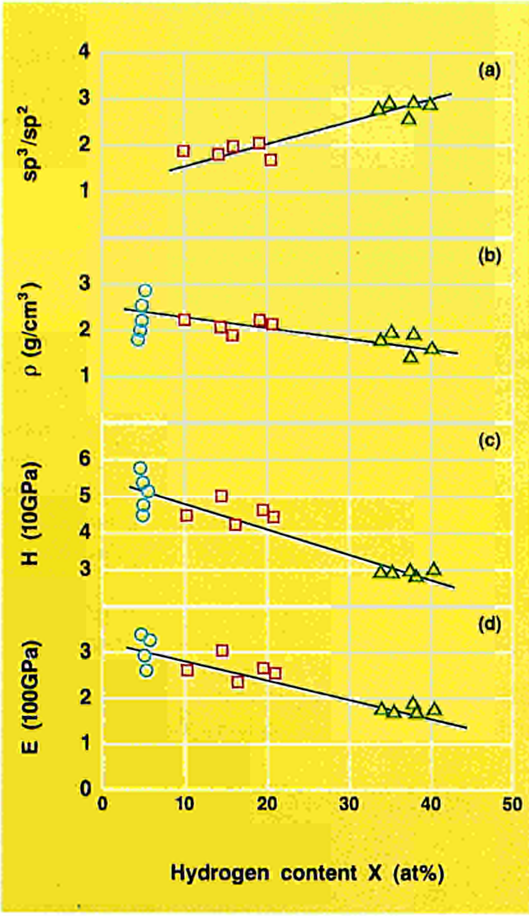
The various R&D programmes are oriented towards technical and industrial applications and use the synergy offered by the unique combination of techniques offered by ACC, such as CVD and PDT or CVD and Thermal Spraying.

## 2. Ti-B-N Films

In view of the promising results which were obtained by the multilayer interdiffusion (MI) and co-sputtering (CS) techniques (see Annual Report 1990), Ti-B-N coatings were also prepared by $Ti^+$ implantation into hexagonal BN coatings. Figure below shows the results of hardness, H, and Young's modulus, E, measurements by the nanoindenter method as function of the Ti ion dose, which was varied from $4.10^{16}$ to $6.10^{17}$. The $Ti^+$ ion energy was 200 KeV. Although a hardening of the BN coating was observed, only moderate hardness values of up to 12 GPa were obtained compared with the 50 GPa obtained by the MI method.

**Below:** *Hardness and Young's modulus of $Ti^+$ implanted hexagonal BN coatings as function of the ion dose.*






**Above:** *$sp^3/sp^2$ bond ratio (a), density (b), hardness (c) and Young's modulus (d) versus the atomic hydrogen content, x, of a-C:H coatings.*

## 3. Hf-B-N Films

Hf-B-N coatings were prepared by the MI and CS techniques. Due to their chemical similarity, we expected that the incorporation of hafnium atoms in the BN lattice would cause a similar hardening effect to titanium. Coatings with different (Hf)/(BN)ratios were prepared by varying the single layer thickness, $d_{Hf}$ and $d_{BN}$, of Hf and BN respectively, using the MI technique, and by varying the position of the substrate between the Hf and the BN targets when applying the CS technique. Under optimum conditions, Hf-B-N coatings reached maximum hardness values around 20 GPa which is considerably softer than Ti-B-N coatings.

## 4. Hard Carbon Films

Amorphous diamond-like carbon films were prepared in a dual ion beam facility, equipped with two Kaufmann sources.

Films were prepared:

i) by sputtering carbon atoms from a graphite target and assisting the deposition process with an $Ar^+$ and $H^+$ ion beam of variable ion-ratio and energy:

ii) by direct deposition using methane as the working gas and without assisting with a second ion beam. Hardness and Young's modulus of the films were determined using a depth-sensing ultralow-load nanoindentation method.

The atomic hydrogen content was determined by FTIR spectroscopy from the integrated absorbance of the C-H stretching modes. Absolute values were obtained by calibration against ERD measurements. The $sp^3/sp^2$ bond ration of hydrogen atoms was determined from an analysis of the individual components of the C-H stretching modes. The chemical composition of the films was determined by RBS. Film with an atomic hydrogen content, x=≤ 5% displayed hardness values of up to 57 GPa and Young's modulus up to 335 GPa. It was observed that the hydrogen content is the most significant parameter for hardness, Young's modules, density, and the $sp^3/sp^2$ ration, where as the energy, $E_a$, supplied to a condensing C atom plays a major role only for x=≤ 5%. Figure above shows the measured values of the $sp^3/sp^2$ bond ratio (a), density (b), hardness (c) and Young's modulus (d) versus the atomic hydrogen content, x.

# Properties, Performance, Characteristics and Innovation of FUNTIONAL MATERIALS

# Composite Materials Properties Improvements

The tensile properties of the plasma sprayed deposits of aluminium containing ceramic particulate dispersions (see Annual Report 1990, p.35) are summarized in Table 1. The results indicate that the strength level attained by the VPS materials in the as-sprayed condition is fairly low when compared with that of similar hot-compacted composites.
However, the possibility of achieving medium-level strength with a lower number of thermomechanical processes might be attractive for several kinds of non-structural materials as well as for coatings, where other properties such as wear resistance and frictional properties may become dominant.

With the aim of widening the applications of VPS particulate composites, an evaluation of the tensile strength of the above composites in the hot-compacted condition has been carried out in the JRC-Ispra laboratories.

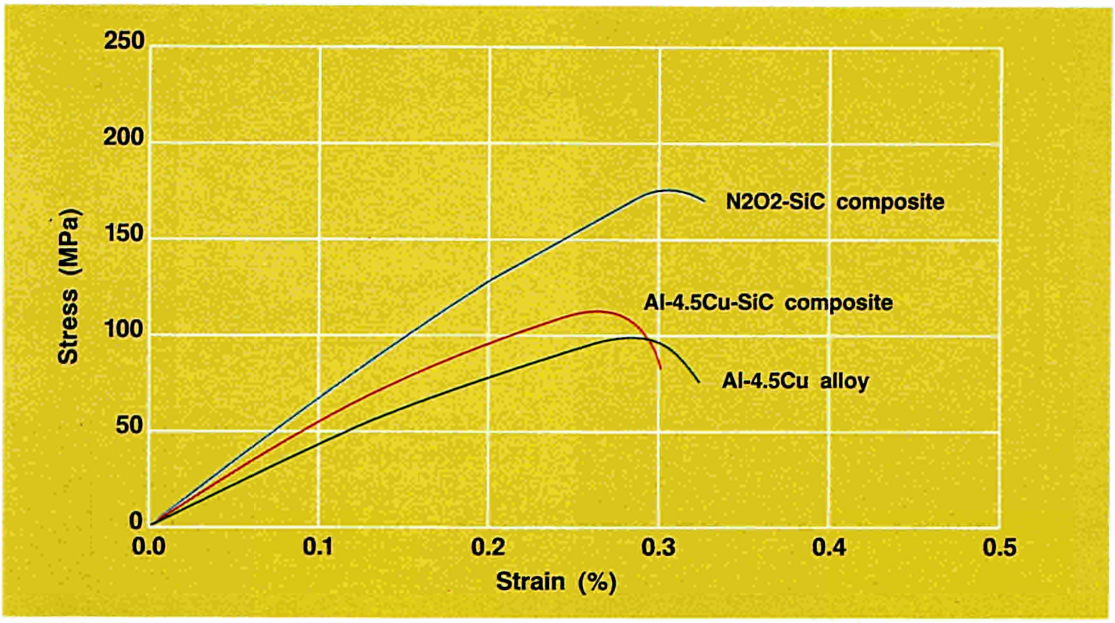
The Al+33%SiC composites were studied in both the as-sprayed condition and the annealed condition (400°C for 30 min) with the purpose of enhancing the bonding between the splatted droplets which form the deposit. By considering the tensile data, it is observed that the treatment did not alter significantly the tensile behaviour of the material, suggesting that there was already a sufficient chemical bonding between the particles.

A comparison between the Al-4.5Cu+33%SiC composite and the corresponding control alloy reveals that the former material shows an increase in the UTS value and in the stiffness properties, qualitatively assessed by the slope of the stress-strain curve, figure on page 29. Since the uniform elongation values of both are low, in spite of the higher value that could have been expected for the unreinforced alloy, it is believed that an additional embrittling factor arise during the heat treatment. Microstructural observations of the two materials showed a clear increase in the presence of blisters after heat treatment. The fact that the elongation of the unreinforced alloy elongation is as low as that of the composite shows that the effect of the blisters is more significant than the notch effect introduced by the SiC particles, in reducing the ductility of the VPS deposits. As far as the N202+SiC composite is concemed, it attains a remarkably high strength level though still maintaining low uniform elongation values. In addition, it is worth noting that the tensile data for the three age-hardened materials were affected by a relatively large scatter of values which again highlights the important role played by the defects in these composites.
On fracture surfaces of the broken specimens little secondary cracking or any other form of damage could be observed behind the fracture line apart from some small defects originally present in the material. The separation path was roughly perpendicular to the loading direction and only occasionally were the SiC particles exposed on the fracture surface.

**Table below:** *Tensile data of the VPS composites in the as-sprayed condition*

| Material | Treatment | Number of specimens | UTS (MPa) | | µm(%) |
|---|---|---|---|---|---|
| Al+33%SiC | as-sprayed | 2 | 96.4 | 95.4 | 2.99 |
| | annealed | 4 | 94.9 | | |
| Al-4.5Cu control alloy | T6 | 7 | 104.9 | | 0.36 |
| Al-4.5Cu+33%SiC | T6 | 7 | 118.4 | | 0.26 |
| N202+SiC | T6 | 5 | 175.6 | | 0.34 |

Notes: Treatment T6 = The heat treatment T6 consisted of 8 hours at 535°C plus 10 hours at 540°C, water quenching and artificial ageing at 160°C for a period of 8 hours.




**Above:** *Stress-strain curves of the Al-4.5Cu+33%SiC and N202+SiC composites and of the Al-4.5Cu control alloy.*

The SiC particles were sufficiently bonded to the matrix and evidence of decohesion was only noted in some of the ceramic phases close to the fracture surface. All the specimens failed by a brittle mechanism. Agglomerates of SiC or particularly large voids acted as crack nucleation sites. From a microscopic point of view, the matrix behaved in a ductile manner showing a dimpled fracture morphology. Occasionally, cavities and heterogeneous particles were noticed on the fracture surface.

**Conclusion**

The results of microstructural studies and tensile tests performed on Al-SiC particulate composites produced by a vacuum plasma spray co-deposition method are described.

It is reported that the tensile strength of the composites in the as-sprayed condition is of great interest considering that it can be further improved by suitable thermomechanical treatments when free-standing bulk materials are required.

Moreover, the materials are also attractive for application in the as-sprayed state as coatings.

# Chemical Sensors

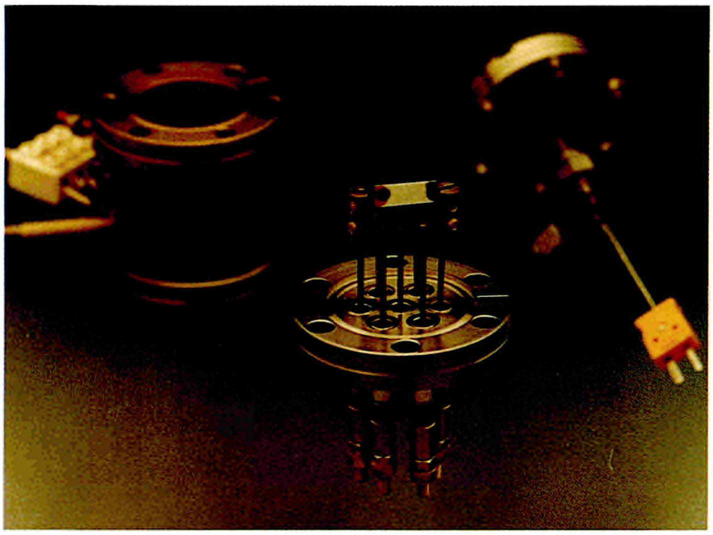
The study of Solid State Chemical Sensors has been continued in 1991, with special emphasis on the $No_x$ detection problem. Two main lines of research have been followed:

a) The development of a potentiometric sensor, of the type $Ag/AgNO_3//B^+Al_2O_3/Pt$ (cooperation with MILANO RICERCHE)
b) semiconductor sensing devices, in which the change in semiconducting properties of $SnO_2$ films due to adsorption of molecules in the surface has been studied in an $NO_x$/air mixture (cooperation with CSIC, Madrid and the University of Madrid).

**Potentiometric Sensors**

Preliminary results with the thermodynamic cell indicated above the in presence of $NO_2$ appear to be in fair agreement with the Nernst equation.

In the case of $NO/NO_2$ mixtures (currently called $NO_x$), the amount of NO in the mixture can be calculated from the reaction equilibrium, $NO+½\ O_2 = NO_2$ provided an independent measurement of the oxygen partial pressure, is performed simultaneously, e.g. by an oxygen sensor.



### Semi-conducting Sensors

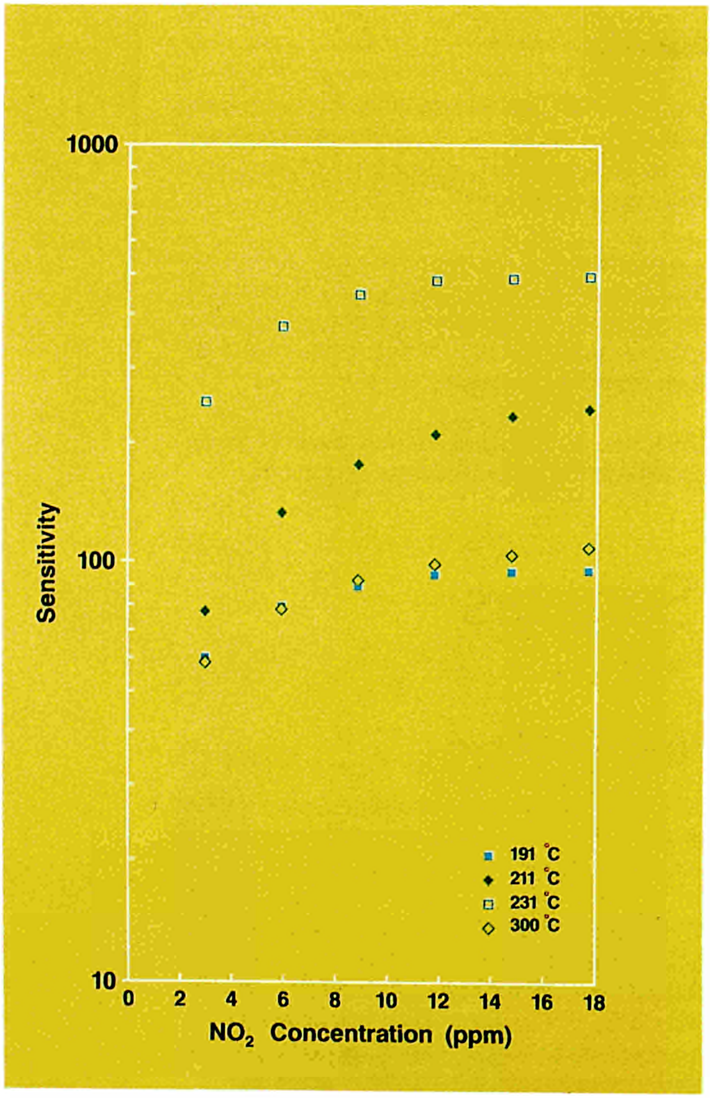
An automated system has been set-up, constructed during 1991, with the objective of preparing gas mixtures having an extremely precise composition (figure above).

Samples of undoped and transition metal doped $SnO_2$-films have been deposited on different substrates, at CSIC-Madrid. A sophisticated film deposition technique has been employed. This has unabled very uniform thicknesses to be obtained; the size of the sensor has been sufficiently miniaturized (e.g. 4 x 8 mm). Spray pyrolysis has also been employed as a deposition technique. In this way thinner layers have been achieved.

About 200 measurements have been carried out on both types of samples of undoped $SnO_2$ in inert gas and in air; the latest results show the possibility of detecting $NO_2$ in an amount as low as 0.2 ppm in the mixture, which is a lower detection limit than previously published (figure below).

These results are the subject of a publication. They show that these sensing surfaces are a reasonable basis not only for alert systems, as needed for monitoring nitrogen oxides in industrial fumes or car exhaust, but also for environmental control, where much lower concentration levels have to be measured.

Discussions about cooperation in the field of $NO_x$ sensors are being carried out with an automobile engineering research laboratory (TNO Delft).




**Above:** *The measuring cell*

**Below:** *Sensitivity R/Ro of an $SnO_2$ Sensor to varying $NO_2$ concentrations in air at four different temperatures (R = resistance in $NO_2$ containing mixtures; Ro = $NO_2$ free)*

# Modulation of SURFACE PROPERTIES

# Surface Treatments for Improved Performance

## Surface Modification Centre

### Equipment

Requests were received form several groups of IAM for the implantation of samples with aluminium. But the ion source in its original form did not allow the implantation of aluminium. Since the target was not sufficiently cooled. As a result the aluminium reached its melting point during operation. The source has now been modified. A new cooling system for the sputter target has been designed and has been mounted. It is now possible to implant elements from a target with a low melting point such as aluminium, magnesium etc.

Laser coating is one of the important tools for surface protection. In the case of the two step procedure, the surface is covered first with the alloying elements. The prepared surface is then remelted in a second step using a laser or an electron beam. In the one step procedure the alloying material is directly introduced into the laser beam. For this reason the laser has been equipped with a device which allows a wire to be fed into the beam.

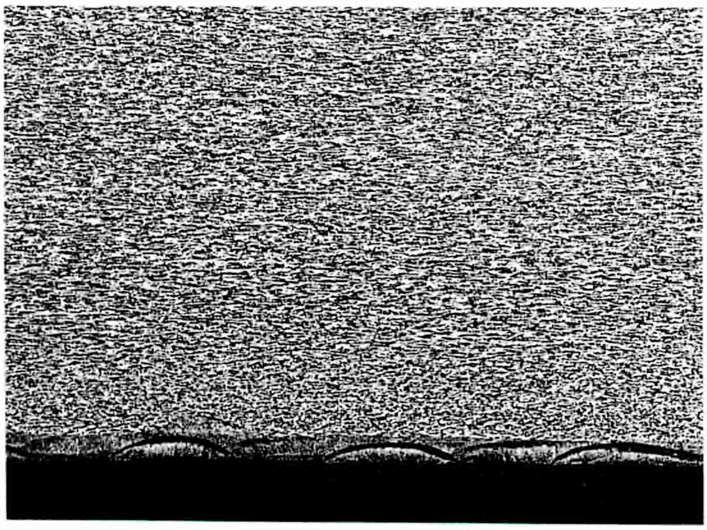
However in order to control the process the beam nozzle has to be modified. A first set of new nozzles has been designed and fabricated. They have given satisfactory results. Further development is underway.

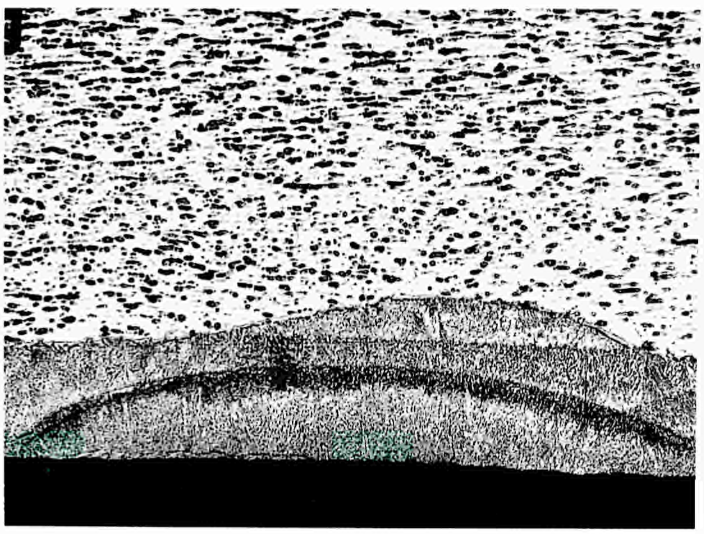
One of the goals of surface treatments is the improvement of wear behaviour. In collaboration with the JRC Institute for System Analysis a novel machine for wear testing has been developed and designed. The construction of the machine is underway. It will allow the measurement of wear on even very resistant surfaces, and it will offer the possibility of following the wear behaviour continuously during longer tests. This will allow changes in the wear process to be observed during the test, when parts of the material, which are below the surface become involved. It also will allow to obtain time dependent information on the wear process itself.

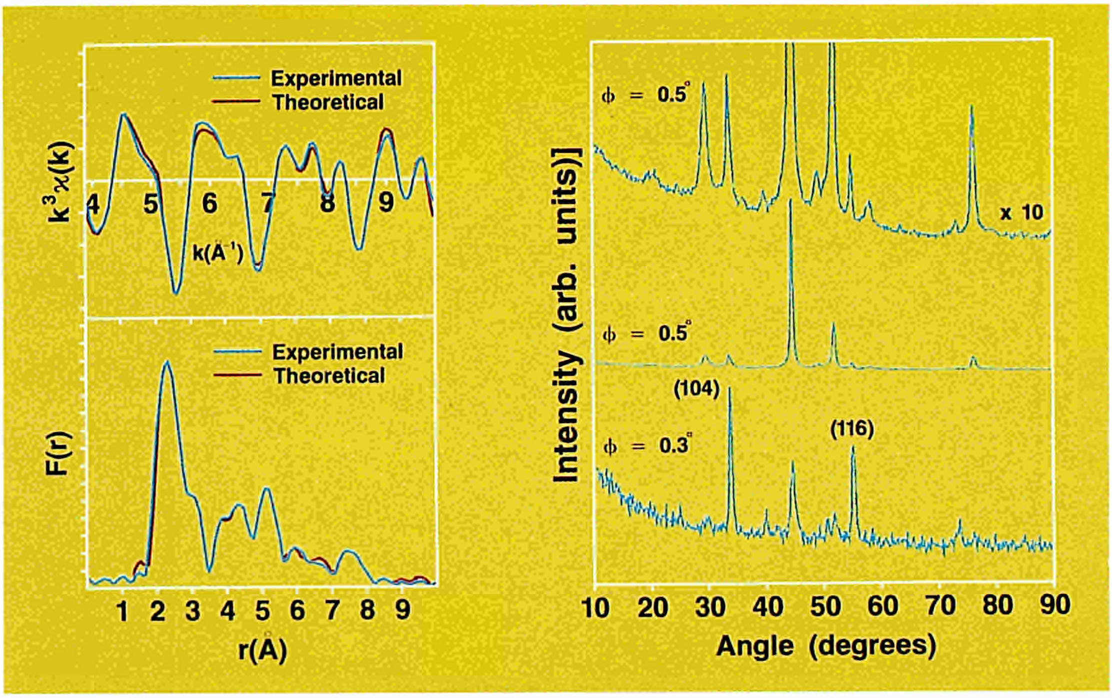
### Studies

1. A procedure for the surface melting of stainless steel by a laser beam has been established. Essential parameters for a good surface are the beam power and its velocity and also the type and velocity of the protection gas. The degree of overlap of the different melt paths is another important variable. By choosing the right parameters, it is possible to generate a rather homogeneous molten layer on the surface with a thickness between 50µm and 300µm (figure below).

**Below:** *Surface structure of laser treated 316 (50x and 200x)*








**Above left:** *Experimental and theoretical EXAFS data of a NiAl intermetallic formed on the surface of Al coated Ni-20Cr*

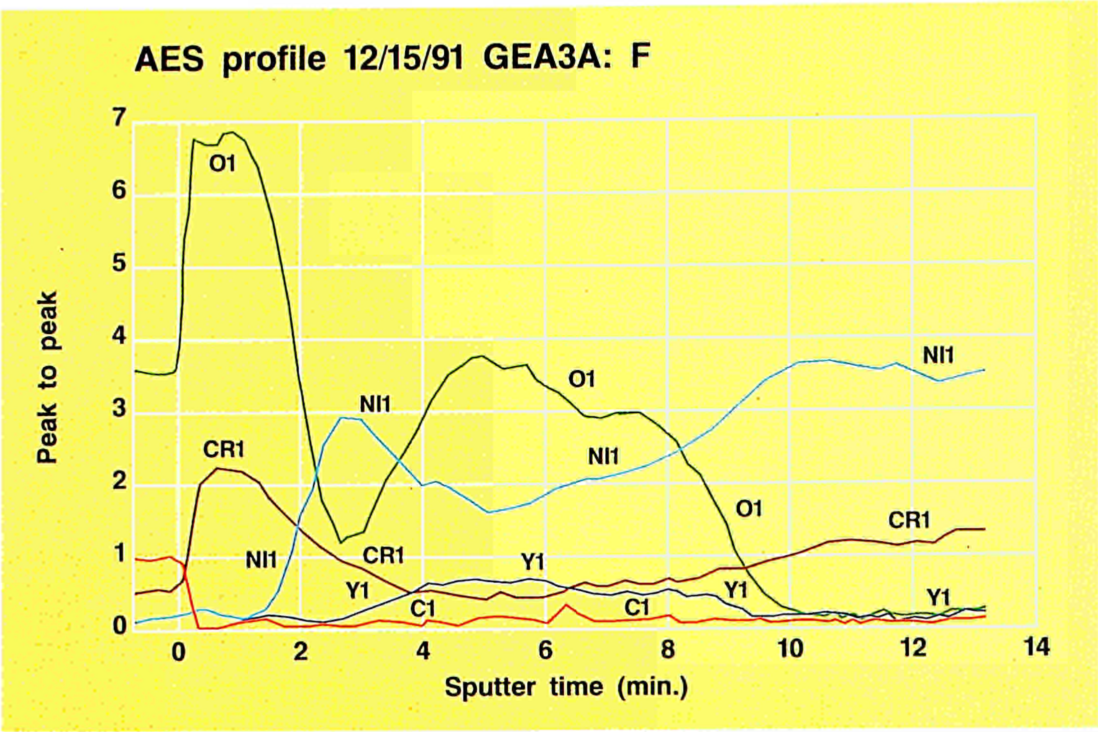
**Above right:** *GAXRD spectra of oxidised, yttrium implanted Ni-20Cr illustrating the depth profiling capability of the technique (Ø is the incident angle of the X-ray beam on the sample surface)*

The grain structure of the molten part is strongly modified. The much smaller grains, which are a consequence of the extremely rapid cooling of the liquid layer, will change those properties of the surface which depend on the grain size. The high cooling rate is also responsible for the formation of a rather fine distribution of second phase particles. A series of fatigue specimens have been produced with laser treated surfaces in order to determine the influence of the fine grain size at the surface on the fatigue behaviour.

2. For the ion-beam mixing of the system $Al_2O_3$/Al, samples of the pure $Al_2O_3$ substrate (GAXRD) were covered with a layer of polycrystalline Al of about 100 nm thickness. The implantation with 100 KeV $Ar^+$ ions was done at target temperatures below 200°C, in a vacuum of about $3.10^{-6}$mbar, and at fluences between $4.10^{17}$ and $10.10^{17}$ $Ar^+/cm^2$.

An AES concentration profile of the Al-layer of an unimplanted $Al_2O_3$/Al sample revealed that the thickness of the Al-layer was 120 nm, which is in good agreement with the desired 100 nm. From this AES-profile and from ESCA-MULT determination it can be seen that there is $Al_2O_3$ on the surface of the Al-layer up to 5 nm. After that distance the concentrations of oxygen and $Al^{+3}$ decrease rapidly with increasing distance from the surface, whereas the concentration of $Al^0$ increases, as expected.
The same type of sample, was implanted with $6.10^{17}Ar^+/cm^2$. An AES-profile revealed that the thickness of the Al-layer was 83 nm, which also is in good agreement with the desired 100 nm, further determinations indicated thicknesses of 110 and 87 nm.
For a fluence of $6.10^{17}Ar^+/cm^2$ the Al-layer should have completely disappeared either due to mixing or sputtering, or both. This is in contradiction with the experimentally obtained thickness of the Al-layer, and with the results of ESCA-MULT determinations. These show that between 50 and 80 nm there is $Al_2O_3$ and that the concentration of $Al^{+3}$ is decreasing, having a minimum just at 80 nm. After this point it starts increasing with increasing distance from the surface. This means that the existing layer contains $Al_2O_3$. Also exactly at 80 nm $Al^0$ starts to appear, decreasing to about zero at 260 nm. This implies that mixing has taken place. The oxygen concentration increases from 60 to 160 units between 50 and 260 nm, and is nearly parallel to that of $Al^{+3}$.

3. In a further ion-beam implantation study the main aim was to improve the corrosion resistance of nickel based alloys through implantation of active ions - in this case yttrium.




**Above:** *Auger depth profile of the Y implanted Ni-20Cr oxidised for 8 minutes at 700⁰C*

A Ni-20Cr alloy was implanted with Y in an attempt to promote growth of improved chromium oxide protective scales. Auger/ESCA spectra on these samples revealed the implantation profiles as well as the existence of Carbon contamination. Comparison with an oxide dispersion strengthened alloy (MA754) was made. In this system yttrium oxide is mechanically alloyed into the material during fabrication. Work is continued on the growth and improvement of alumina ($Al_2O_3$) protective layers with the implantation of Y into deposited Al films and duraluminium bulk samples, and with the separate implantation of both Al and Y into Ni-20Cr.
Various analysis methods have been used including glancing angle X-ray diffraction (GAXRD), grazing incidence X-ray reflection (GXRR) and X-ray absorption spectroscopy (XAS) at SERC Daresbury synchrotron radiation source (in this case the ReflEXAFS technique was employed).

Figure left on page 25 illustrates the Fourier filtered experimental ReflEXAGS spectrum (solid line) of Al doped Ni-20Cr after oxidation at 700⁰C for 4 minutes, together with the theoretical curve (dotted line) expected for NiAl intermetallic a nearly perfect fit is observed. Figure right on page 25 shows the GAXRD spectra of Y implanted Ni-20Cr oxidised for 8 minutes at 700⁰C taken at two different angles of incidence. Analysis of the spectra clearly indicated a layer of yttrium oxide (ca. 700Å) at the interface between the protective chromia and the alloy substrate. This is confirmed in figure above which shows the Auger depth profile of the Y implanted Ni-20Cr oxidised for 8 minutes at 700⁰C. Separate Yttrium oxide and Chromium oxide layers are evident (sputtering rate = 150 Å/min).

4. In the same context, Positron Annihilation doppler Spectroscopy, using an ordinary beta source (22Na) has been used for the investigation of defects and the characterization of ceramic coatings and of metallic substrate.

The table on page 27 shows typical results, obtained with TiN films (8.5µ) deposited on stainless steel in the Thin Film Laboratory. The parameter S, characterizing the defect density shows a better recovery, after annealing, in stoichiometric TiN (a) compared to non stoichiometric TiN (b). A much higher defect density is seen after one year aging. This method may allow a good quality characterization of samples, as well as a precise determination of their mass density.

5. At the end of 1991 work commenced on the ion beam mixing of Ti-BN multilayers to form hard thin films. GXRR and GAXRD are being used to directly measure the amount of mixing and to compare this with theoretical predictions from the TRIM and PROFILE computer codes.

| N° | Sample material and treatment | S x $10^5$ | W x $10^5$ | (W/S) x $10^5$ |
|---|---|---|---|---|
| 1 | SS (a) as received (mirror polished) | 50707 + 7 | 23502 + 7 | 46349 + 22 |
| 2 | SS (b) as received (mirror polished | 49843 + 12 | 24414 + 12 | 48982 + 32 |
| 3 | SS (a) annealed | 49531 + 13 | 24711 + 15 | 49891 + 36 |
| 4 | SS (b) annealed | 49578 + 20 | 24671 + 21 | 49763 + 61 |
| 5 | SS (a) annealed, sputtered, chem. cleaned | 49422 + 10 | 24845 + 9 | 50272 + 27 |
| 6 | SS (b) annealed, sputtered, chem. cleaned | 49419 + 16 | 24853 + 11 | 50290 + 33 |
| 7 | $TiN_x$ (a), x = 1 as deposited, on SS (a)-5 | 52355 + 10 | 21493 + 9 | 41052 + 23 |
| 8 | $TiN_x$(b), x = 1 as deposited, on SS (b)-6 | 52570 + 11 | 21470 + 8 | 40840 + 22 |
| 9 | $TiN_x$(a), x = 1 annealed | 52194 + 11 | 21661 + 10 | 41504 + 27 |
| 10 | $TiN_x$(b), x = 1 annealed | 52491 + 10 | 21429 + 9 | 40824 + 24 |
| 11 | $TiN_x$(a), x = 1 annealed, aged | 50795 + 18 | 23250 + 20 | 45772 + 51 |
| 12 | $TiN_x$(b), x = 1 annealed, aged | 51951 + 13 | 22307 + 10 | 42938 + 28 |

**Table above:** *PAS Parameters measured with $TiN_x$ coatings and SS substrates*

6. High temperature turbine blades are protected against gas corrosion by coatings of M CrAlY. Such coatings of different composition have been produced by low pressure plasma spray on a number of materials used for turbine blades.
Methods have been developed to improve the adherence, the density and their shock resistance. The coatings have been tested in a high temperature corrosion loop at temperatures around 1000°C.
These studies are related to work executed for a third party contract.

Data and Information Management
for ADVANCED MATERIALS

# High Temperature Materials Databank

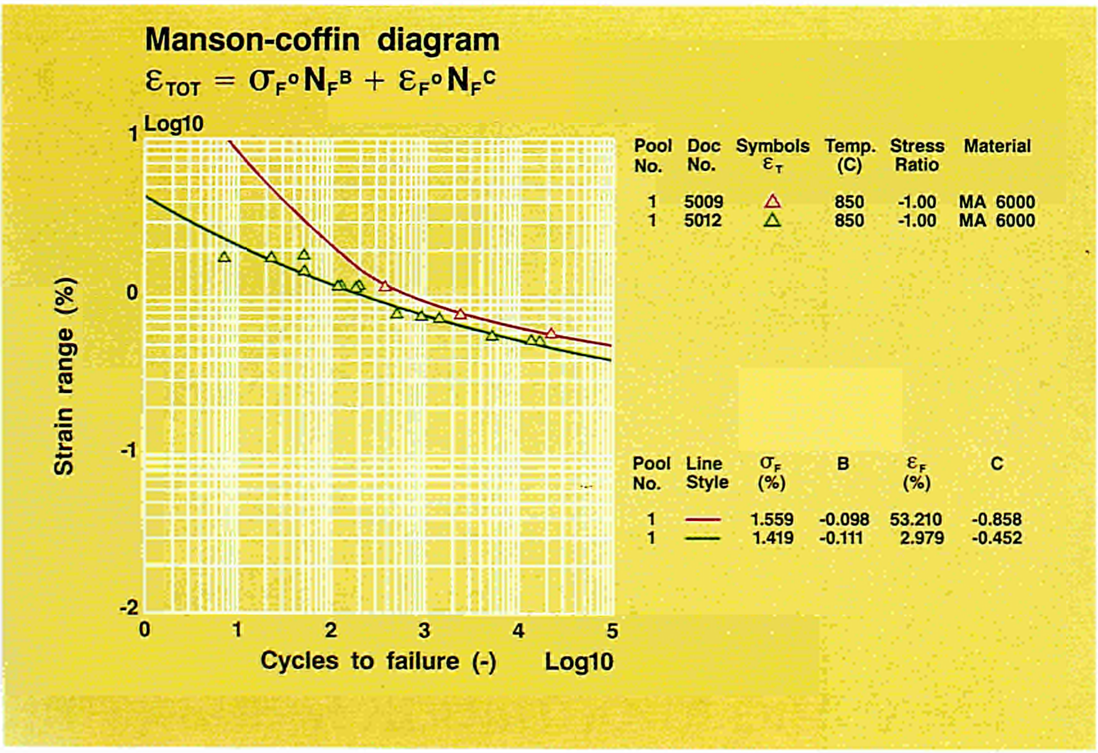
The High Temperature Materials Databank (HTM-DB) supports the Data and Information Management for Advanced Materials Project by providing computerised information on materials properties through the storage of mechanical test data in combination with a sophisticated modelling and evaluation system. It aims to cope with requirements for data management, evaluation and input for computer aided processing and information services. It further serves the dissemination of data between collaborating parties in joint projects.

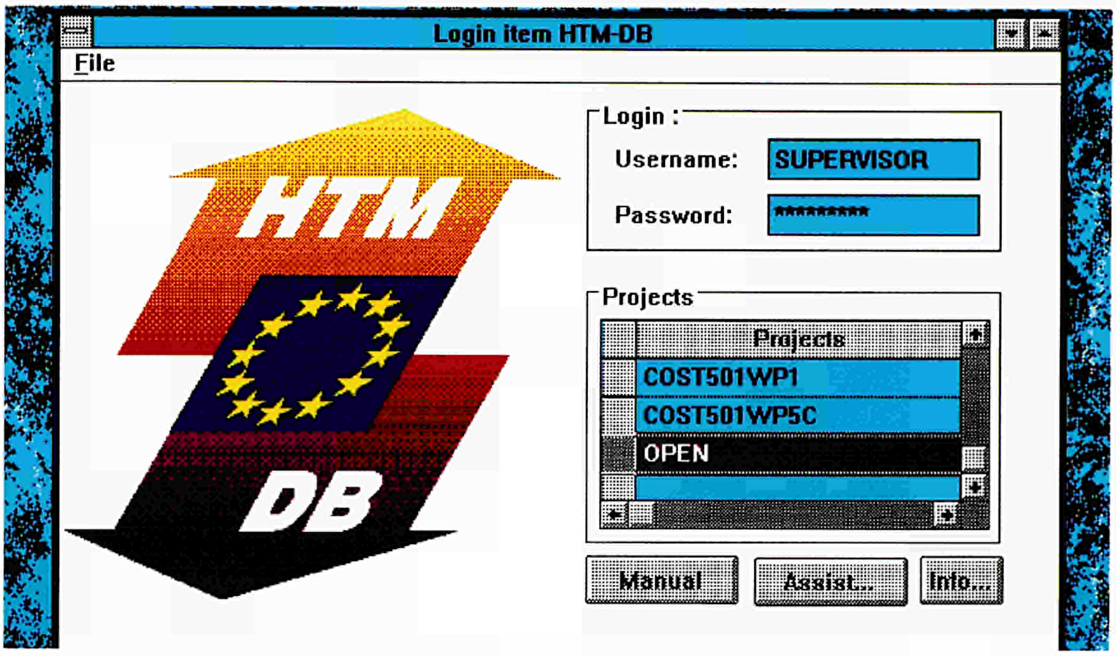
In 1991 a major effort has been devoted to further extension and improvement of the evaluation programs. Through them the databank offers project partners a common retrieval system and enables comparison of their data which gives them information on the scatter of their experimental test measurements.

The MANSON-COFFIN diagram (figure below) shows the data and calculation results for the same material batch, tested in different laboratories (sources 5009 and 5012). The HTM-DB is accepted as the centre for data acquisition and distribution for COST 501-II Work Package 5 projects which are related to new and service exposed ferritic steam pipe materials in European power plants. Evaluation programs like NORTON CREEP LAW and LARSON-MILLER are used to show the difference in the material potential between new and service exposed material. Most of the COST partners are connected to the HTM-DB and work with the PC-interface (see annual report 1990).

The HTM-DB is an online databank which is installed on the AMDAHL mainframe computer in Ispra with ADABAS as the databank management system.

**Below:** *MANSON-COFFIN diagram*

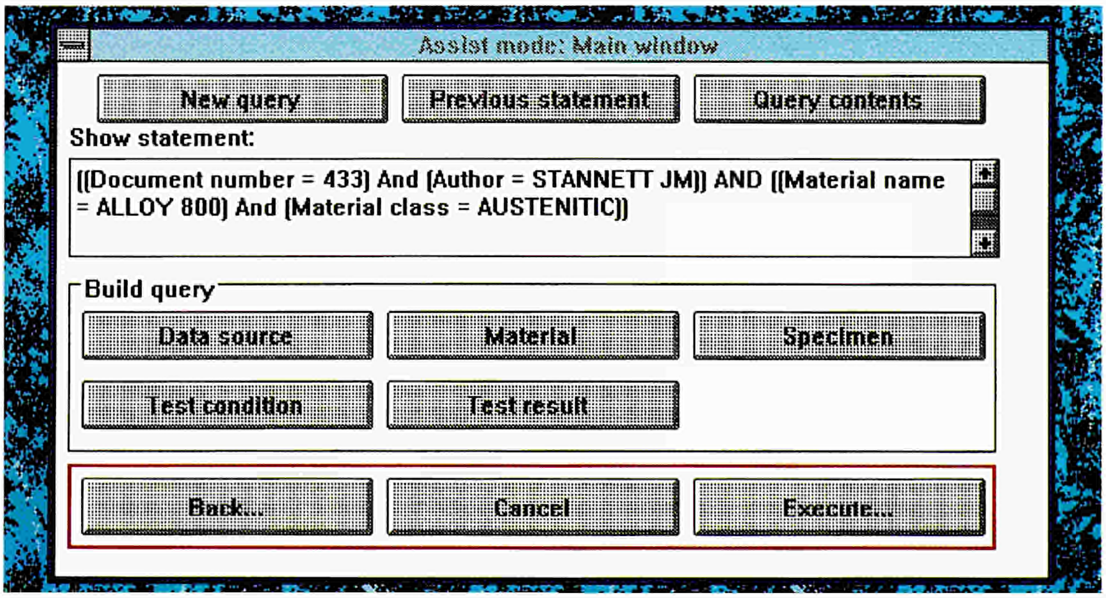





Due to the policy of the CEC to terminate the Amdahl mainframe service July 1st, 1992 the HTM-DB must be transferred to a local workstation. It was therefore necessary to reorganize the HTM-DB on a workstation using ORACLE as a relational DBMS and the evaluation programs must be rewritten under MS-DOS.
The user friendly data input and data retrieval interface is implemented on the PC side under Microsoft WINDOWS which interacts with the workstation via a network connection.
ORACLE provides facilities for Ethernet and/or asynchronous local and/or wide area network connections so that the online service to project partners (COST, BRITE, FUSION, etc.) can still be maintained. The data input and data retrieval interface is written in SQL windows in a very user-friendly manner. The figures above and below shows window screens of the new databank. Figure above shows the databank main menu with HTM-DB logo and the windows with an entered password and user identification. This information controls access to confidential data belonging to particular projects. Figure below shows the main menu of the ASSIST guided data retrieval mode with an built-up query which offers the user the most important fields for data retrieval.
The new databank structure in combination with the chosen soft- and hardware is very flexible for small changes. It can easily be adapted to the special needs of a company and can also be implemented on a PC. Therefore it seems to be attractive to companies as an 'in-house' solution especially with respect to preserving the confidentiality of their own data.

**Above:** *Window screen of the new Databank main menu with HTM-DB logo*

**Below:** *Window screen of the main menu of the ASSIST guide*



# Information Centre

The objective of the Information Centre is to provide an information bureau, a meeting forum and an instrument for cooperation, the promotion and dissemination of information on materials research in the Community and to act as continuous interface to industry.

In 1991, efforts have been focused on the following activities:

Within the frame of the Institute initiative for standardisation and pre-normative R&D of advanced ceramics a meeting of the CEN working group on standardisation of Ceramic Matrix Composites was organised.
The International Workshop: Towards Clinical Trials of Glioma with BNCT (September 1991) was co-organised with the CEC Concerted Action on Born Neutron Capture Therapy. The Workshop reviewed all available data required for starting clinical trials on patients and derived at a consensus for a strategy for clinical tests. 100 experts attended the Workshop (see also under Exploratory Research: BNCT, on page 66).

The second European Colloquium on Designing Ceramic Interfaces: Understanding and Tailoring Interfaces for Coating, Composite and Joining Applications was held in November 1991.
It focused on:

* identification of coating, composite, and joining developments where attainment of specified interfacial properties is vital,
* establishing the relevance and need for interfacial design in applications of advanced engineering ceramics,
* understanding the principles of interface fabrication and how these can be exploited to optimise properties and design,
* development of interface design principles by theoretical modelling and systematic experimentation,
* identification and initiation of technical interactions needed for the development of optimised interfaces.

12 invited lecturers and 25 poster papers were presented. 70 experts attended the Colloquium.

# 2. Contribution to the Specific Programme:

## REACTOR SAFETY

# Project on the Inspection of Steel Components (PISC)

**Introduction**

The Programme for the Inspection of Steel Components carried out since 1974 under the auspices of the CEC/JRC and the OECD/NEA is a major international effort (14 countries and 80 institutions) to better assess the capability and reliability of Non Destructive Inspection procedures on structural components.

Three phases were centred on the Ispra Joint Research Centre which, in its roles of Operating Agent and Reference Laboratory, managed the other programme and provided, with the other participants of EC countries, approximately half of the programme funding; the other half came via contributions in kind from the non-EC participating countries. OECD/NEA provided the Secretariat of the PISC Managing Board, consisting of representatives of the participating countries.

The programme is now in its third phase (PISC III project) and presently the activities concentrate on the validation of the PISC II results and on the extension of the PISC methodology to various important structural components made from a range of materials.

Although in the new JRC programme from 1992 to 1994 PISC has not been considered a high priority area in the Reactor Safety Programme of the JRC, its completion will nevertheless be supported in 1992-1993. The JRC management believes strongly that the output of PISC, principally its methodology, should be extended into broader fields, involving critical facilities in Europe in which structural integrity is of paramount importance. Moreover, the interest shown, directly and indirectly, by other EC Directorates-General, for example those responsible for Energy, Industrial Affairs, and External Relations, has led to the initiation of new actions which will be in support of Commission Services with the aim of extending the PISC approach to a broader industrial field.

Thus, in order to complete the PISC programme, the JRC will contribute an effort, during the period 1992-1993, of 1,5 MECU from a total budget of 4 MECU estimated for PISC during this period. It is strongly believed that this total effort will be necessary and sufficient to bring PISC to a successful conclusion, but all partners must commit themselves to provide the expected support for the testing of structural assemblies, destructive examination and the evaluation of results as proposed at the start-up of the programme and reiterated by some CSNI members on specific request of the PISC Management Board.

In the application of strict rules of quality verification of the PISC II results evaluation, the Management Board of the programme will not authorize publication of any PISC results before the end of 1992, even for the programme actions terminated in 1990 and for which the evaluation of results is nearly concluded. PISC reports in 1992 are thus only general status reports not containing diagrams of results or specific conclusions.

PISC III results will be published as they become available in 1993 and 1994.

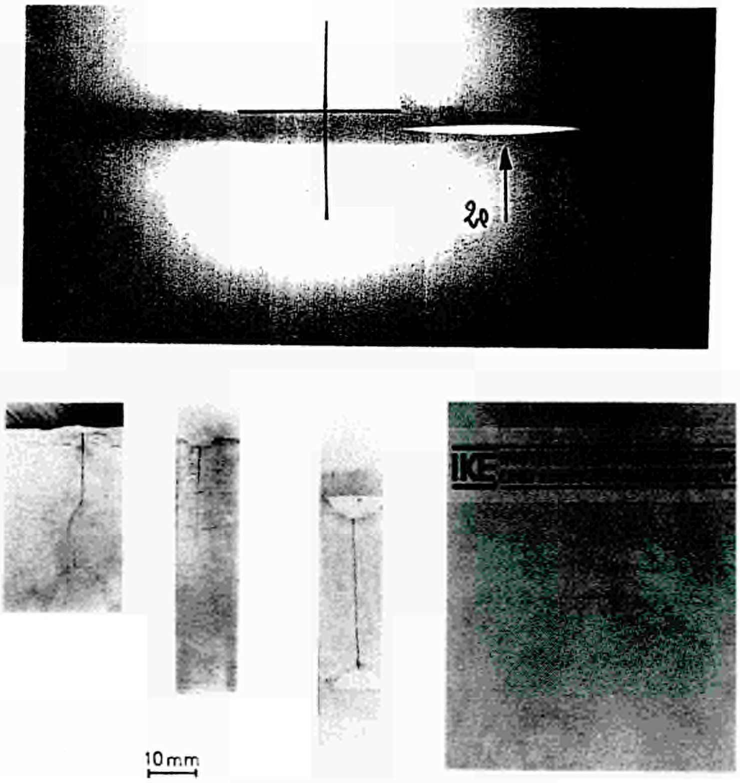
**PISC and Performance Demonstration**

There are two main types of engineering codes and standards, those which detail the methods and procedures to be used (prescriptive) and those that define the results which must be achieved (performance standards).

International organisations concerned with standardisation and codification now tend to prefer the performance type of standard to the prescriptive type for several reasons. First, the standard prescribing the means may put up technical barriers to trade; secondly it may impede later technological progress by freezing technology at a given level. On the other hand, performance standards avoid the problems that can arise when a prescriptive standard prescribes a suitable methodology but which will, not necessarily, achieve the required performance.

It is therefore believed that the standards concerning results (performance standards), will grow in importance; they allow competition between companies which use their well established technologies. They will render easier harmonization at the international level. Nevertheless it must be emphasised that performance standards must involve performance demonstration.

An important question is whether one can propose appropriate and affordable test pieces to be used in the procedural demonstration of the inspection




Smooth sharp planar defects (artificial defects).

**Above:** *Realistic flaws as artificially introduced in PISC blocks and mock-ups*

techniques. Such test pieces have to contain flaws which simulate both those which may commonly occur in real structures as well as those relating to limiting cases that could be imagined during actual plant inspections. One solution, which has been attempted several times, is to collect samples removed from actual structures containing real flaws. The objection to these samples however, is that they always represent particular cases which are possibly not the worst nor the most difficult that could arise in reality, moreover, these samples are contaminated. An alternative approach is to design and manufacture artificial flaws in realistic assemblies which produce the same response to the NDT technique used as those from real flaws. Some advantages of this latter approach are:

- the possibility of more certain characterisation;
- the possible parametrization of the tests, including the provision of limiting cases;
- the possibility of providing guidance to experimenters enabling improved understanding;
- the provision of non contaminated assemblies which can be more easily used for assessment;
- the cover of a more relevant selection of structural geometry and material.

To be convincing, however, this second approach must be supported by a demonstration and confirmation that there is a good correlation between the artificial "realistic" flaws and their natural counterparts.

The PISC II methodology has led to considerable development in this second approach where experience shows that the introduction of these artificial "realistic" flaws in blocks and structures provides affordable and reproducible demonstration test samples (figure above). Mathematical models, involving a better understanding of the physical phenomena, indicate which artificial flaws can be used to replace natural ones for the validation of NDT techniques and the training of inspection teams.

The concept of performance demonstration was supported by the PISC results and by the demonstration through this programme of the validity of validation assemblies.

In 1990, ASME significantly modified Section XI of its Code for Pressure Vessels and Boilers, by the introduction of a new appendix "Performance demonstration for Ultrasonic Examination Systems". Also in Europe, Performance Demonstration has been seen as the way to harmonize the ISI (In-Service Inspection) procedures without "technical dispute". JRC, IAM, Reference Laboratory of PISC, has developed, with a network of industries and laboratories (*) the know-how to introduce artificial realistic flaws, in preference to the real flaws that some have recommended.

**PISC III Programme Status**

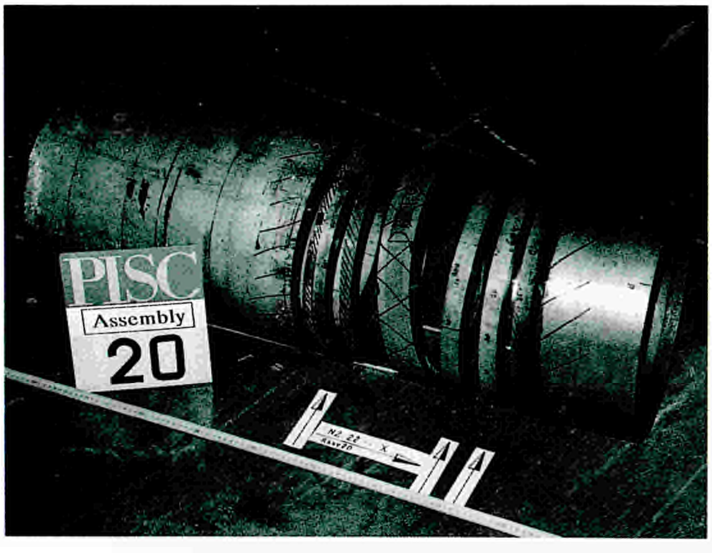
PISC III, the third phase of the PISC series requires the demonstration of the capability of the methodology to deal with assemblies of real geometry containing realistic defects. The PISC methodology is also extended to all major parts of the primary circuit of the LWR reactors. Moreover, the work done on the austenitic steel testing is of considerable value for the inspection of LMFBR components. Eight programme Actions have been established as identified in the Organization Scheme.

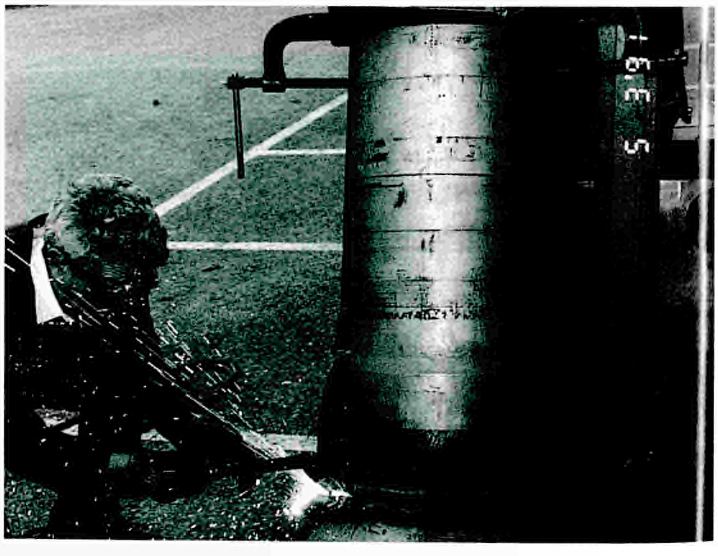
The present stage of PISC III work concerns the Round Robin Testing activities which have been, or are presently being performed in several of the PISC III Actions. The final work to be undertaken in all the eight Actions has been defined and most of the necessary test samples and other resources have been obtained.

The objectives of each of these eight Actions of PISC III and the status of work are as follows:

Action No. 1 (Real Contaminated Structures) seeks to collect results from specific investigations on real service induced defects in materials and structures of the primary circuit of light water reactors (LWRs). The hot cell facilities at JRC Ispra were fully equipped for non destructive and destructive work on a collaborative basis.
An important objective of this Action 1 was the study of real defects from an NDT response point of view with the hope to establish a correlation between real and artificial "realistic" defects to be used for test assemblies.

**Below:** *Destructive examination of assembly No. 20, Safe-ends parts (Safe-ends 21 and 22)*



(*) Present major contributors of PISC assemblies: ENSA (Spain), ANSALDO (Italy); CEA (France); EDF (France); ENEL (Italy); IVC (United Kingdom); JAPEIC (Japan); MITSUBISHI (Japan); NUCLEAR ELECTRIC (United Kingdom); UNIV OF STUTTGART/MPA (Germany); BATTELLE with NRC (USA)

Action No. 2 (Full Scale Vessel Tests) validates the results obtained by procedures in the PISC II exercise but using more realistic inspection conditions: the installation funded by BMFT (German Ministry for Research and Technology) and installed at the Staatliche Materialprüfungsanstalt (MPA), Universität Stuttgart, Germany is being used. It is made of a BWR full scale vessel to which have been added modular full scale PWR components. Nine teams participated from 1988 to 1990 in Phase 1 concerning the sizing of selected defects.
Eight organizations participated in Phase 2. This phase was aimed at the validation of ASME type procedures by an international team using an ISI automatic scanner offered by RWE and MAN to PISC for the period of the exercise (September 1989 to April 1990). A large amount of support was given by the Federal Republic of Germany to enable the work on Action 2.

**Below left:** *Assembly No. 51: wrought to cast welds (contribution of EDF and JRC)*

**Below right:** *Assembly No. 43 (contribution of Japan).*

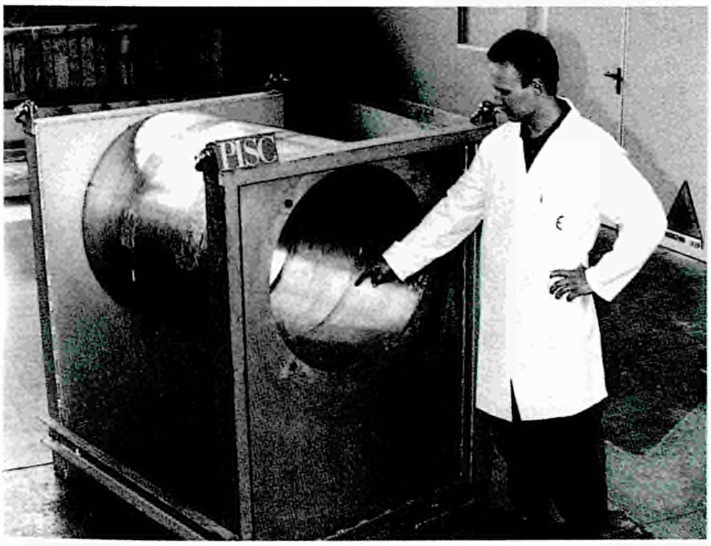
Action No. 3 (Nozzles and Dissimilar Metal Welds) completed in March 1991 the round robin tests of 4 safe-ends representing some of the most difficult technical aspects of In-Service-Inspection.

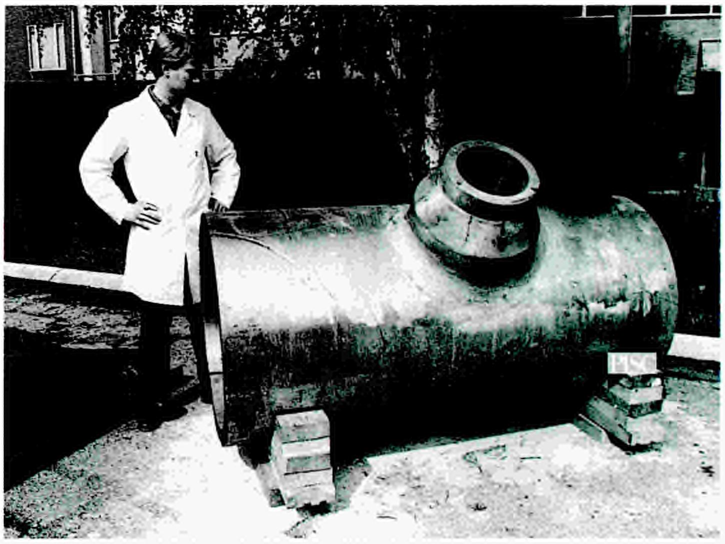
In this work, a Japanese-Italian BWR assembly of nozzle plus safe-end, an American BWR assembly with two nozzles and safe-ends and a Spanish PWR safe-end were inspected in 13 countries. These assemblies are now being destructively examined (figures on page 36).

The results so far evaluated by the PISC data analysis group show that the capability exists for safe inspection of the configurations considered.

Action No. 4 (Austenitic Steel Testing) applies the PISC II methodology to the primary circuit piping of LWRs. Round robin tests for the capability assessment and parametric studies started in 1990.

Wrought steel pipe samples were offered by the USA, Japan and France; moreover, large cast steel samples have been manufactured in Spain (figures below).
Twenty five teams have registered their intent to participate in one or more phases that will extend up to 1992.



Action No. 5 (Steam Generator Tubes Testing) involves in its present phase Round Robin Tests (RRT) of individual tubes of steam generators containing real and artificial defects. Thirty teams from 11 countries participate in the RRT which is planned until 1993. Eight boxes of tubes are in circulation, (figure below). The validation of artificially introduced defects has involved experts from Belgium, France, Germany, Italy, Japan, Spain, United Kingdom and United States of America. The Reference Laboratory (JRC, IAM/NDE) has prepared many artificial defects in tubes. Orders have been placed by the Operating Agent to get realistic corrosion defects by CEA, MITSUBISHI, CEGB, KEMA. Emphasis has been put on corrosion defects (IGA, SWSCC, PWSCC).

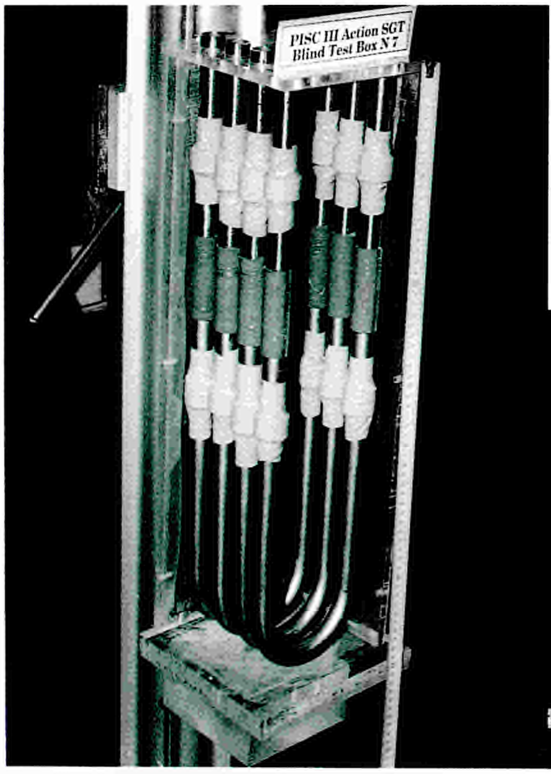
Action No. 6 (Mathematical Modelling on NDE) has the objective to validate experimentally derived mathematical models and perform parametric studies in order to assess the importance of defect characteristics. Sixteen organizations in eight countries have registered their intent to participate in studies to assess mathematical models of ultrasonic inspection by validating the models with experiment and assessing the utility of these models in terms of limits of valid application, satisfactory and efficient computer performance and accuracy. Fifteen models have been offered for possible study. An important objective is to promote the practical application of mathematical models as an aid to more effective and efficient inspection procedures and interpretation of results.

Action No. 7 (Human Reliability Studies) seeks to evaluate the influence of human interpretation of inspection results, equipment malfunctions and human interaction in the overall inspection procedure.

Perhaps the most important result obtained in the recent reappraisal of the PISC II results was that Ultrasonic Techniques effectiveness was dominated by human reliability aspects.

Action No. 8 (Support to Code and Standard (C/S) Organizations) proposed by the Management Board and approved by OECD-NEA and CEC-JRC gives direct support through the PISC group of experts to C/S organizations. Such a proposal has been supported by all PISC members and involves three aspects:

**Below:** *Blind test box containing bent tubes with flaws*




- Informing the relevant Codes and Standards Technical Committees of PISC results and PISC related programme results.
- Critical review by PISC members of technical documents on request from national and international technical groups.
- Preparation of technical reports by PISC members related to Codes and Standards problems, for the benefit of National, CEN, ISO and IIW technical groups which elaborate standards.

The organization and support of Action 8, involves mainly the Operating Agent.

**Ageing of Plants**

Many studies are under way in the energy production industry to maintain high plant capability and availability factors in face of ageing phenomena of most of the plant structural, mechanical and electrical components. Due to the difficulty of identifying new sites in several countries and costs of building new plants, replacement of aged components becomes a growing technology in nuclear and non-nuclear plants. When such replacements are difficult or impossible (e.g. of a pressure vessel) annealing and repair programmes are to be considered in view of the extension of the life of some components. Replacement or special treatment of components are thus motivated by economic reasons but together with the need to permit safe operation up to the end of the projected life time and beyond. Many different ageing phenomena can contribute to failures that lead to extended plant outage or even threaten to shorten the projected lifetime of the plant. Typical failure mechanisms include:

- corrosion, e.g. stress corrosion cracking;
- embrittlement (plastic, rubber);
- vibration, thermal cycling, fatigue;
- wear, erosion;
- deposit effects;
- creep in fast breeder reactors and in non-nuclear plant components.

These phenomena have to be studied and understood. In several cases, adequate understanding of the mechanisms can only be gained in the course of supporting laboratory test research programmes and inspection campaigns.

A plant life management programme includes the following tasks:

. Identification of components of importance to plant life;
. Identification of life threatening factors;
. Understanding and description of ageing phenomena;
. Evaluation of the replaceability of components;
. Study of annealing and repair methods for non-replaceable components;
. Evaluation of residual life of repaired components.

R and D institutions are obviously capable of important contributions to the understanding of ageing/degradation mechanisms of structural materials. However, related to most of the points above two priority items are often not clearly identified.

a. The research and development of monitoring techniques for online monitoring of the degradations mentioned above in order to verify the fitness for purpose of the structural material:
   - irradiation and thermal embrittlement,
   - corrosion cracking, fatigue cracking, deposit induced degradation....
b. The improvement and better use of periodic inspection procedures.

Besides these essential activities for which IAM has specific expertise and capabilities, studies of verification or simulation are also necessary as well as methods for restoration of materials properties such as annealing of embrittled material.

This requires detailed trials on the effectiveness and stability of such methods already in use for the most critical plant components like pressure vessels.

Concerning inspection techniques, PISC and related studies have quantified the performance of vessel weld inspections, particularly as part of the PISC III programme, covering the effectiveness of austenitic weld inspection (safe-end and pipe work) as well as the inspection of steam generator tubes. Several challenging inspection problems have still to be solved and, to complement the results of PISC, two other study groups are necessary:

- The identification of all the RPV regions and components which are to be inspected for safety assessment purposes, and the ranking of these components in safety relevance (New Management of ISI).
- The identification of an inspection procedure suitable for each region or component of the primary circuit (Challenging Inspection Problems).

# 3. Contribution to the Specific Programme:

## RADIO-ACTIVE WASTE MANAGEMENT

# Safety of Final Storage in Geological Formation: Materials Research Aspects

Tests have been conducted on the corrosion of mild steel to be used as a container for highly active wastes in connection with their disposal in sea bed sediments. In most cases these sediments are rich in calcium carbonate. It was realized that, due to the thermal gradient caused by the heat released by the wastes, a thick layer of calcium carbonate would by formed around the mild steel samples. As a consequence, the corrosion is strongly reduced.

For a land based repository a backfilling composed by pressed bentonite is usually foreseen. A series of tests has been conducted in order to verify whether an addition of powdered calcium carbonate to the bentonite allows a protective layer to form. Around the sample two conditions were chosen. In the first, tests were performed at constant temperature, 80°C, and in the second under a thermal gradient of 1°C/cm. Two different compositions of the bentonite-calcite mixture was chosen.

In both cases, it appeared that a thick layer was formed at the surface of the sample, containing a large proportion of magnetite. In the test performed under a thermal gradient, the surface layer appears to be more compact.

Using the data obtained previously, a final analysis of the penetration of hydrogen into the glass has been performed. In fact hydrogen can enter into the glass by three different mechanisms. Firstly, it can enter by ionic exchange with alkali ions, diffusing then into the glass body. It can be present at the surface due to the formation of silicon hydroxide and finally can diffuse into the solid body as a water molecule. The data obtained by nuclear reaction has clarified that water diffusion is found mainly at high temperature, while around room temperature, the hydrogen present in the glass interior has to be attributed to ion exchange and subsequent diffusion as $H^+$.

# 4. Contribution to the Specific Programme:

## FUSION TECHNOLOGY and SAFETY

# Materials Integrity

## Behaviour of Mechanical Properties under Irradiation

*HFR Irradiations*

The irradiation of tensile samples of AISI 316L welds up to 15 dpa at 250, 300, 350, 400 and 450°C (FRUST experiment) has been completed. The specimens unloaded from the SIENA irradiation capsule, were transported to JRC Ispra for post-irradiation analysis. The welds were of the electron beam (EB) type and Submerged Metal Arc (SMAW) type. The post irradiation tensile tests will be completed in 1992.

In the same period tensile specimens of Cr-Mn steel (AMCR type) weldments irradiated up to 10 dpa at 250 and 450°C and Cu-Cr-Zr alloy for divertor applications, irradiated up to 10 dpa at 150 and 250°C, were ready for post-irradiation testing.

New optimized Cr-Mn steels (IF type) are under irradiation in the SIENA capsule at 250 and 450°C, up to neutron fluences corresponding to 10 and 15 dpa. The composition of these steels, produced in collaboration with UNIREC (France), was optimized to improve the mechanical properties and satisfy the low activation requirements.

Neutron Irradiation Hardening - Evaluation of the results of the Oak Ridge Test Matrix [1]

In 1983 a number of low-temperature neutron irradiations started under an IEA implementing agreement in the "Oak Ridge Test Matrix" (ORTM). The materials came from the various national fusion technology programmes: SA 316L from the EC, 25% CWPCA and 20% CW316 from the USA and SAPCA from Japan. The irradiations were performed in the HFR reactor at Petten, the similar R2 reactor at Studsvik and in the HFIR reactor at Oak Ridge; the latter to obtain data at higher helium appm to dpa ratios.

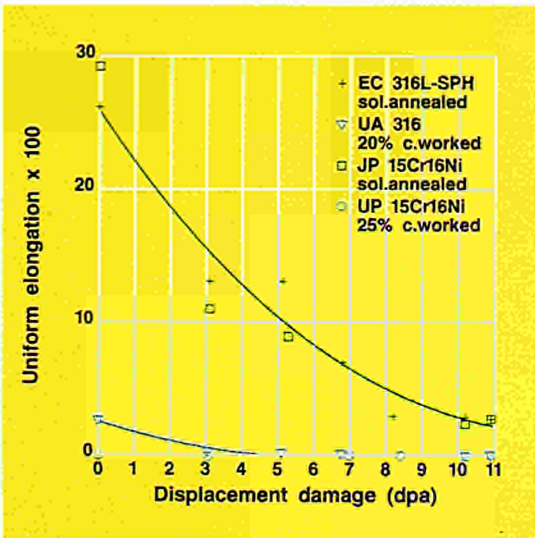
The four austenitic stainless steels were irradiated at 250°C from 3.1 to 10.9 dpa. Irradiation hardening was measured by tensile tests at the same temperature at an initial strain rate of $4.6 \times 10^{-4} s^{-1}$. The tensile curves were reviewed and analyzed in terms of true stress-true strain to assess remaining homogeneous strain hardening capability.

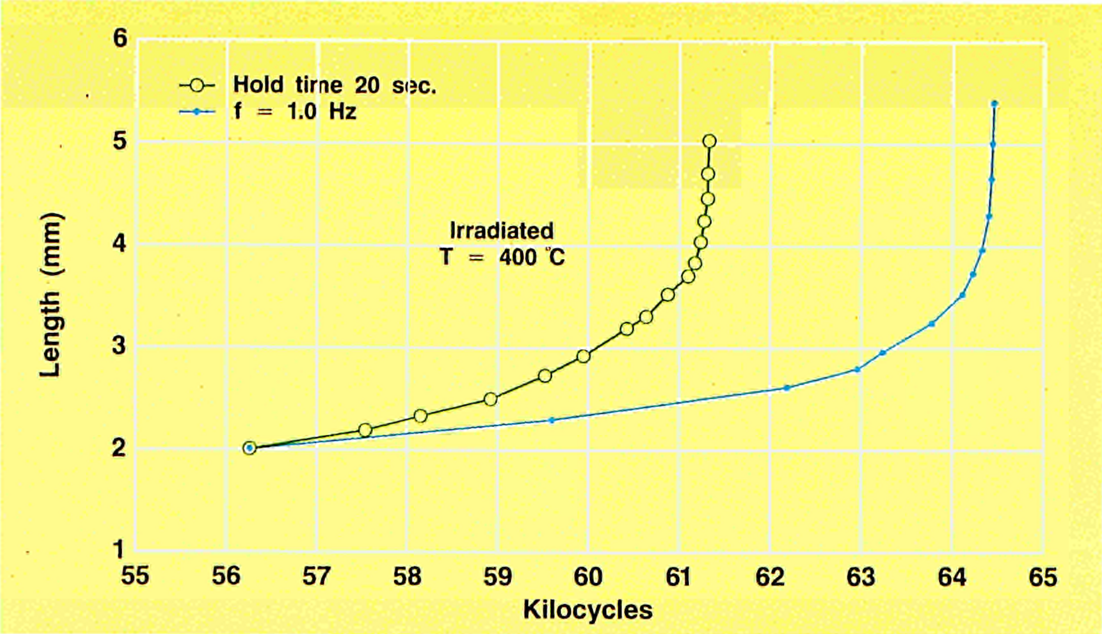
Radiation hardening of solution annealed material saturates at 820 MPa after about 6 dpa; that of cold-worked material at 1040 Mpa. Material condition (annealed or cold worked) dominates over the small difference in Cr-Ni content.
There was no influence of damage (from $1.6 \times 10^{-7}$ to $11 \times 10^{-6}$ dpa $s^{-1}$) and helium appm/dpa ratio (ranging from 6 to 40). The uniform elongation of solution annealed materials gradually decreased to about 3% at 10.9 dpa, while disappearing in the cold-worked material after irradiation to 3.2 dpa (figure below). The trend of hardening by prior cold work and/or irradiation suggests a stress limit of about 850 Mpa for homogeneous deformation.

## Fatigue Crack Growth under Light Ion Irradiation

Following NET requirements, in-beam fatigue crack growth was measured under irradiation in AISI 316L type stainless steel with hold times of 20 and 40 s. The energy of the protons was 18 MeV, the damage rate was of the order of $6.10^{-7}$dpa $s^{-1}$ and the irradiation temperatures were 200 and 400°C.

**Below:** *Residual uniform elongation in function of displacement damage*






**Above:** *Influence of hold time on fatigue life under irradiation at 400°C*

The data obtained have been compared to those measured in similar irradiation experiments performed under cyclic tensile stress [2] and data from experiments without irradiation. The results obtained from the irradiation experiments are:

- A dependence of crack propagation on temperature, already observed in tests without irradiation.
- A notable increase in the resistance of the material to crack growth and an extension of fatigue life. These effects are more evident during crack initiation and in the first stage of crack propagation due to hardening by defect agglomerates produced by displacements. Microhardness measurements and fractographic analysis support this interpretation.
- For experiments with hold times creep-fatigue interaction is present at 400°C; with a hold time of 20s a reduction of fatigue life has been observed, in contrast with tests without irradiation (figure above).

**In-beam Creep Fatigue Interaction**

The calculations for NET indicate that the first wall fatigue cycle is characterized by compressive stresses during the plasma burn period and the reversal of these stresses during the off-burn period, in strain controlled conditions with a non-zero mean strain. Such a loading cycle is prohibitive for uniaxial fatigue testing under light ion irradiation, since this type of irradiation requires the use of miniaturized specimens with a thickness typically smaller than 200 µm. The situation is different when the torsional stress mode is used. In this case stress reversal in miniature specimens can be accomplished easily and stress reversed creep fatigue tests can be performed during irradiation with light ions.
Strain controlled creep fatigue tests with zero mean strain were performed on 316 L stainless steel specimens in the 20% cold worked condition, at a temperature of 400°C during irradiation with 19 MeV deuterons, imposing a holdtime at the minimum shear stress in the load time cycle. In addition to the number of cycles to failure (Nf), stress strain curves were measured to determine the creep induced stress changes in the loading cycle. These measurements show that, for tests in which the imposed total shear strain range $\Delta\gamma$ was smaller than 1.3% (tensile equivalent $\Delta\varepsilon$ = .75%), the stress relaxation induced by the irradiation creep process leads to an increase of the minimum stress (where the hold time is imposed) and the maximum stress in the cycle and thus, to a shifting of the hysteresis loop on the stress axis. Increasing values of T cause a drastic reduction of the number of cycles to failure by more than a factor to ten.

No reduction of Nf was observed for $\Delta\gamma$ = 2.45% ($\Delta\varepsilon$ = 3.2%); the stress strain curve stayed symmetric for the duration of the test.
The experiments further show that the influence of irradiation creep on the fatigue behaviour of the specimen depends essentially on three parameters: the total strain range, the mean strain and the load time relation.

When the results are applied to the wall loading cycle, which is not symmetric but contains a mean strain, one can exclude an increase of the absolute stress values in the loading cycle such that a reduction in Nf will occur.

**Radiation Creep of AISI 316L**

Irradiation creep data were obtained for stainless steel alloys in the HFR at Petten during the last ten years.

The main important technological result is that the creep rates are almost temperature independent and so small that stainless steel of the 316 and the AMCR-type could be used for the first wall in a fusion device from the point of irradiation creep.

The following results were obtained:

- The stress exponent is about 1 for deformed materials and about 1.8 for annealed materials.
- The secondary creep rates are almost temperature independent.
- The total elongations obtained in the HFR at Petten for 5 dpa correspond approximately to those obtained for 316 at ORR- and at EBRII-reactors. For USPCA (316) and AMCR 0033 the creep elongations at 5 dpa are about a factor two smaller than those observed for US 316 though the creep rates are about equal for all steels at 5 dpa.

The smaller creep elongation is due to negative creep at the beginning of the irradiation due to microstructural changes.
In cold worked USPCA and AMCR 003 the negative creep elongation observed at the beginning of an irradiation is only seen for stresses below 100 MPa and increases with decreasing applied stress and irradiation temperature.

The critical temperature for negative creep elongation corresponds to the transition temperature $T_t$ for interstitial cluster formation in stainless steels.

**Radiation Damage Studies in Ni Alloys**

Radiation damage rate measurements were performed in nickel between -90 and +30°C. Precipitates are formed even at temperatures as low as -90°C. The build up of interstitials and vacancies followed the formation of precipitates. The activation migration energy of interstitials decreased to 0.90 eV for a radiation flux corresponding to $10^{-10}$dpa $s^{-1}$.

Nickel-10% chromium alloys were irradiated between 150 and 500°C with high energy protons.
The radiation enhanced diffusion rate is much larger between 150 and 250°C than between 300 and 400°C. It is assumed that the diffusion rate is determined by an interstitial diffusion mechanism in the low temperature regime and that with increasing irradiation temperature the concentration of interstitials decreases through recombination with thermally activated vacancies. At higher irradiation temperatures, where vacancies determine the diffusion rate, their concentration decreases with respect to that in thermodynamic equilibrium so that at 500°C the thermal diffusion coefficient is much larger than the radiation enhanced diffusion coefficient.

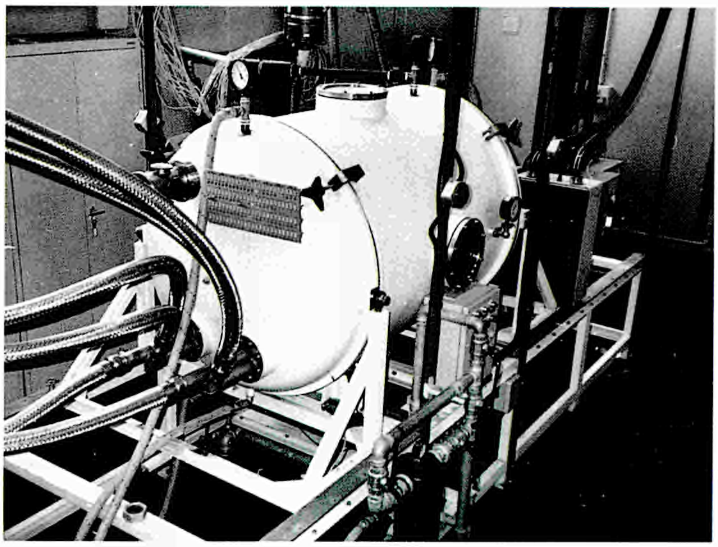
**Influence of Pb-17Li on the Properties of Blanket Materials**

Production of Corrosion Barriers

Production of a $LiMn_2O_4$ type oxide layer [by preferential oxidation of the steel in a controlled $H_2/H_2O$ atmosphere (ratio 1000:1) prior to reaction in Pb-17Li] on the surface of 316L stainless steel has been completed. Tests in a Pb-17Li filled thermal convection loop have shown that after 500h at 600°C the effect of the coating is to reduce, by a factor of 4, the rate of corrosion of the steel by liquid Pb-17Li. Optimization of the coating thickness, together with hydrogen permeation measurements, will be carried out in the near future.

Mechanical Properties

Tensile tests at $1.1x10^{-4}s^{-1}$ and $2.8x10^{-7}s^{-1}$ strain rates have been carried out on DIN 1.4914 martensitic steel under vacuum, Pb-17Li, hydrogen and a combination of hydrogen and Pb-17Li.



**Above:** *The thermal fatigue test facility*

Results showed that hydrogen did not severely affect the tensile properties of the steel, with a ductile fracture mode being observed in each case. However, for tests at 400⁰C, the addition of hydrogen to Pb-17Li reduced the increase in plasticity normally associated with tests in Pb-17Li.
The effect of constant uniaxial load on the corrosion rate of AMCR 0033 (260 MPa) and DIN 1.4914 (250 and 277 MPa) has been investigated in three Pb-17Li filled thermal convection loops operated at 450⁰C for 20,000h (the currently envisaged Pb-17Li blanket lifetime). The applied load did not adversely affect the rate of corrosion of the specimens. The AMCR specimens showed a preferential dissolution of manganese to produce a ferritic corrosion layer of 35μm, whereas the 1.4914 specimens showed no corrosion layer due to the corrosion proceeding as a uniform dissolution process.

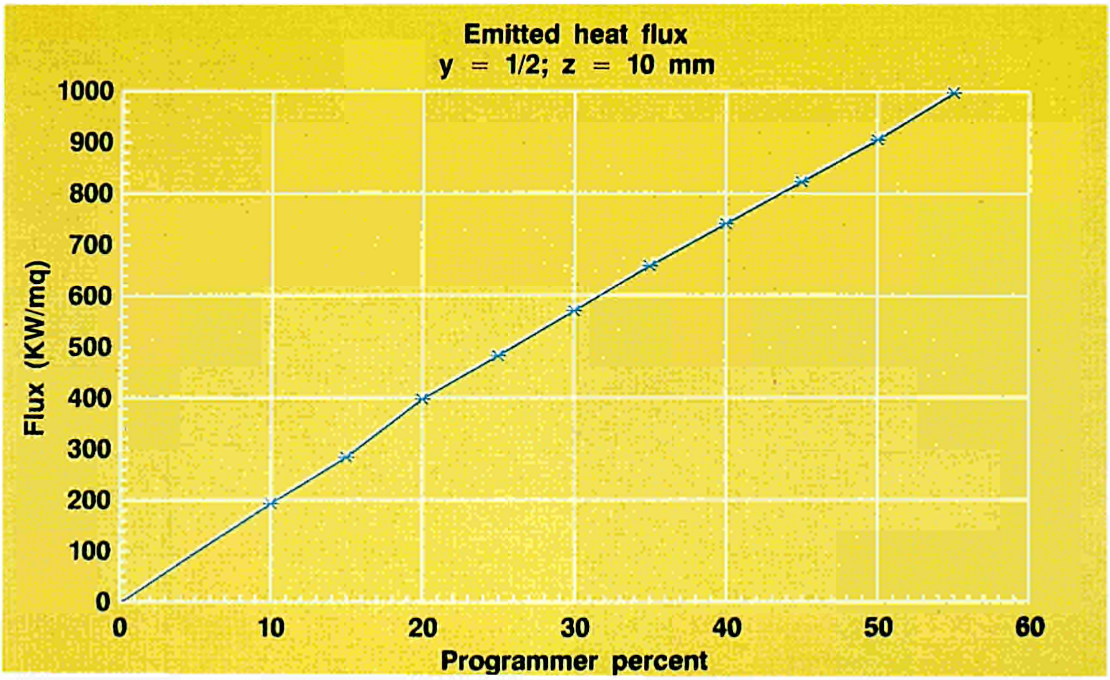
## The Lithium-Lead-Bismuth System

Preliminary results of the study of the solubility of bismuth in Pb-17Li, using electrical resistivity measurements, indicate a solubility limit of 1000 wppm. This result indicates that contrary to our initial expectation it is not possible to remove bismuth from Pb-17Li, whether originally present or produced by nuclear transmutation of Pb-17Li in a fusion reactor (in Europe industrial lead contains 20 wppm of bismuth).

**Below:** *Emitted heat flux as a function of the electrical power supplied to the heating elements*

## Functional Validation of First Wall Components Under Thermal Fatigue

The problems addressed by this project are the evaluation of the thermal fatigue lifetime of First Wall (FW) mock-ups and the assessment of the thermal fatigue growth rate of typical flaws. The approach to these problems is multidisciplinary, combining theoretical analysis and experimentation. The theoretical analysis is based on numerical modelling by a Finite Element Method (FEM) of the FW thermal-mechanical behaviour under cyclic heating loads. The experiments are performed in a thermal fatigue test facility, shown in figure above, designed and constructed for that particular purpose. The facility was provided with a closed loop water cooling circuit and new control instrumentation. The figure below shows the results of measurements of the heat flux by calibrated calorimeters.



The heat flux emitted by the heating elements varies linearly with the electrical power supplied to the heating elements.

The testing capacity was doubled by adding a second independent testing line. The test sections and two sets of infra-red heating elements which simulate the thermal cycle the FW will be exposed to, are contained in a cylindrical vacuum chamber. The closed loop water cooling circuit (up to 1 MPa pressure and 40 $m^3h^{-1}$ water flow) provides heat removal. A computerized data acquisition system allows control of the experiment and recording and analysis of the relevant data from the facility/components instrumentation. Simple geometry, small dimension components, without metallurgical modifications like welded or brazed joints, serve for FEM lifetime analysis validation. This activity is focused on the IAEA benchmark exercise at present under way with the participation of NET Team-Garching, KfK- Karlsruhe, IVKI-Moscow, JAERI-Naka and IAM-Ispra. Testing of three test pieces, B1, B2 and B3 came to an end after 27000, 52500 and 32500 cycles, respectively, with a peak heat flux of 0.5 $MW/m^2$.

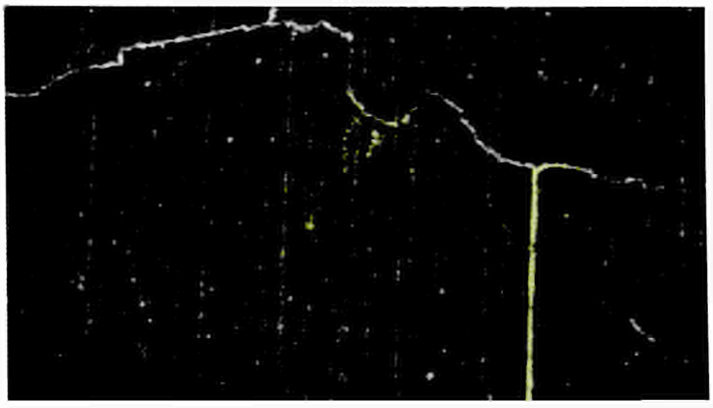
Post mortem analysis and comparison with numerical lifetime predictions are under way.

Figure below displays a typical thermal fatigue crack on the surface of the B1 component.

A second IAEA benchmark exercise based on the thermal fatigue crack growth experiments is being defined. Its aim is to study crack growth behaviour under cyclic thermal stress. The experiment will be carried out on test pieces with artificial flaws and plasma disruption induced surface cracks.

The experimental conditions for the plasma disruption simulation were defined and five test pieces manufactured. A statistical evaluation of the initial crack size distribution was performed by optical and SEM metallography.

**Below:** *Thermal fatigue crack on the surface of the IAEA benchmark component*



**Advanced Low Activation Materials**

C-fibre and SiC-fibre reinforced SiC matrices, so called C/SiC and SiC/SiC ceramic composites, offer both highly appreciable low activation characteristics and quasi-ductile mechanical behaviour provided by the fibre/matrix interaction. This new class of materials is under investigation as a structural material for application in nuclear fusion devices. In the current period a larger batch of unidirectionally reinforced SiC/SiC was received from European industry. The potential of this commercial SiC/SiC in terms of low activation was studied by a detailed calculation (ANITA code and REAC-ECNS library). This non-elementally tailored ceramic matrix composite (CMC) fulfilled all current low activation limits of interest in Europe. The study was based on a quantitative analysis of critical trace elements by means of proton and alpha particle activation. Mechanical testing was refined towards a clear distinction between the fracture regimes for interlaminar failure and true bending. As all low-Z materials, SiC will produce substantial quantities of He under fast neutron irradiations. Therefore, the first experiments will investigate the effects of He implantation on the stability and mechanical properties of SiC/SiC composites. The equipment for He implantation at the Ispra cyclotron was modified so that both bending and tensile specimens can be irradiated.

**Fusion Materials Databank**

The usefulness of the HTM-DB for the fusion structural materials databank and evaluation has been recognized by the European Laboratories/Associations which are responsible for the mechanical testing of structural candidate materials for the design of the NET/ITER reactor. in this context, IAM Petten has been charged with the task of collection and evaluation of the experimental data concerning such materials, their compilation in a format suitable for direct use in structural design and analysis and making them available from a computerized database as a subset of the HTM-DB. For this purpose the Associations have been supplied with the necessary software and in collaboration with the HTM-DB staff are in the process of inputting the data into the new database. This activity is to be continued in the context of the new Multiannual Fusion Technology Programme.

# 5. Contribution to the Specific Programme:

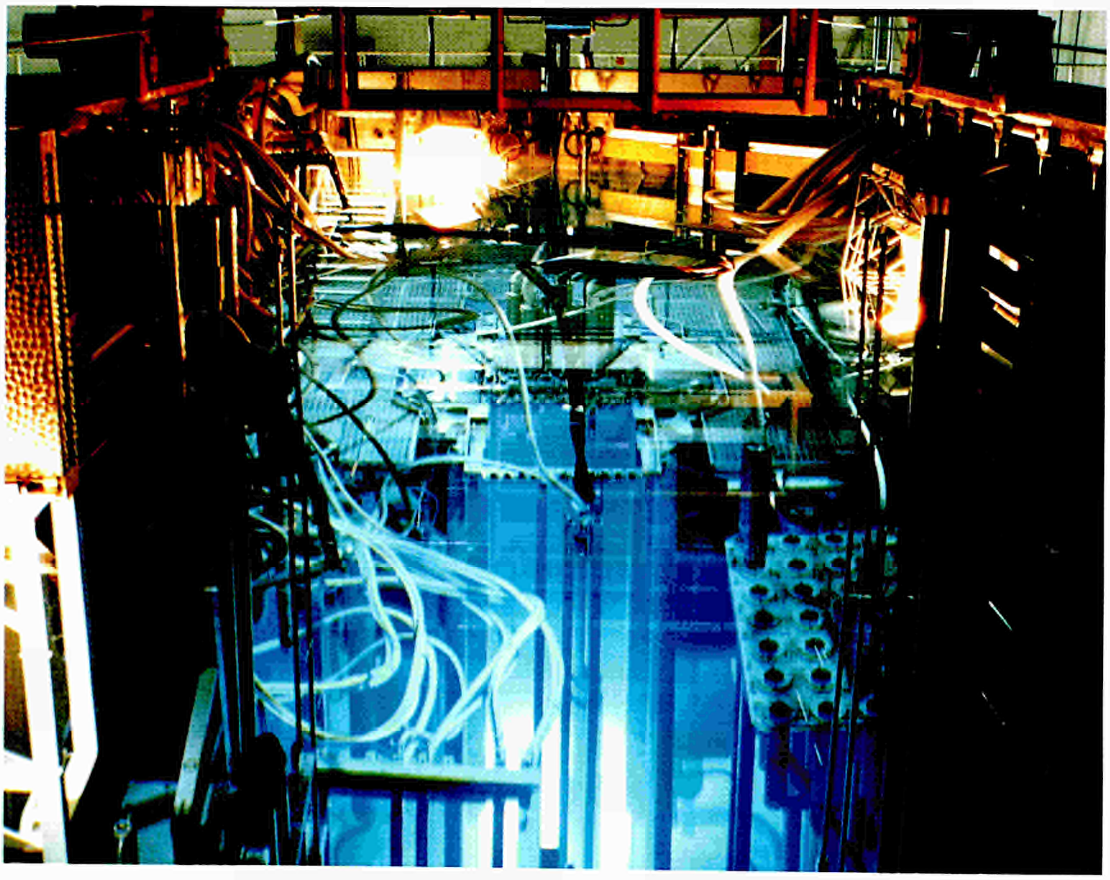
## NUCLEAR FUELS and ACTINIDE RESEARCH

# Irradiation Experiments in the High Flux Reactor

In recent years, as part of the programme of the Institute for Transuranium Elements of the Joint Research Centre in Karlsruhe on the development and improvement of Advanced Fast Breeder Reactor fuels, a series of experiments have been performed in the High Flux Reactor. In the last 2 years, 3 new experiments have been prepared for irradiations in 1992. The first two experiments NILOC 3 and 4 (consisting of 6 fresh, mixed nitride fuel pins) are extensions of the irradiation series NILOC (NItride fuel, Low in Oxygen and Carbon) first proposed in 1987. The 6 irradiation capsules were manufactured and completed at Petten. The transport of the fuel pins to Petten took place in summer 1991. At the end of the year, the 6 fuel pins were loaded into the 6 capsules, sodium filled and sealed. The irradiations (2 HFR cycles for NILOC 3, and 1 HFR cycle for NILOC 4) are planned to begin in HFR cycle 92.04 (May).

The experiment POMPEI (Pellets Oxyde Mixte, PEtten Irradiation), which will study the behaviour of mixed nitride fuel at high burn-up, have been further delayed due to manufacturing difficulties with such specific fuel. The irradiation is now planned for the end of 1992.

**Below:** *View on irradiation experiments in the HFR*



# 6. Supplementary Programme:

## OPERATION OF THE HIGH FLUX REACTOR

# Operation of the High Flux Reactor

The Supplementary Programme on the operation of the High Flux Reactor (HFR) is funded by the Federal Republic of Germany and The Netherlands. Under the terms of the programme, German and Dutch institutions are offered irradiation space and staff services free of charge.

The German contribution is mainly managed via Forschungszentrum Jülich (KFA) and Kernforschungszentrum Karlsruhe (KfK). Most of the projects are related to the German nuclear energy programmes.

Light water reactor fuel testing programmes focused at pre-irradiated fuel rod segments from commercial reactors, were continued. A pre-irradiated BWR fuel rod which had been re-instrumented with pressure transducers at the Petten hot cells, was successfully irradiated for an additional burnup of 10.5 GWd/t(U). This project is part of a study on fission gas release. In support of an investigation of iodine solubility and degassing from a fuel rod after a LOCA scenario, two more tests were successfully performed with pre-irradiated PWR fuel rods. For testing of high burnup PWR fuel rods in the range 150-350 W/cm linear heat generation rate, a new irradiation device providing typical fuel rod surface temperatures already at the lower power level, was developed and commissioned. A power cycling programme using high burnup PWR fuel rods using this new device has been started. A performance test on a new type of fuel for a pressurized heavy water reactor was completed with a power transient test after reaching 15 GWd/t(M) burnup.

The irradiation programmes related to the high temperature gas cooled reactor, comprising graphite and fuel irradiations, is being re-oriented because of declining industrial interest in this development. On-going projects have however been continued to schedule. The "fundamental properties graphite programme" which contributes to the data base on irradiation effects on graphite was continued; 8 individual irradiations are either under evaluation, in the reactor or under preparation. In the graphite creep programme only 1 irradiation was performed with intermittent measurement of dimensional changes of the samples. A second experiment was started, a third is under preparation.

The reference tests of spherical HTR fuel elements for the German HTR programme were continued in 1991. In these tests the operating conditions of the so-called "HTR module" are simulated. The objectives are the confirmation of low coated particle failure at operating conditions and the confirmation of low free heavy metal contamination of the fuel element. The essential information is mainly obtained from on-line measurement of short, medium and long lived volatile fission products.

Fast breeder reactor fuel irradiations in support of the European Fast Reactor project were continued with a short transient test and the continuation of the two long term irradiation tests for fuel and cladding axial displacement investigations.

Development of future SUPER-KAKADU and HYPER-KAKADU experiments was continued. The objective of these ambitious tests is to study fuel performance under power cycling conditions with amongst others, application of noise analysis techniques.

Further progress was made on the redesign of the alpha-tight EUROS cell, which will be used for encapsulating longer fuel pins.

Fusion related investigations comprised the irradiation of prospective constituent materials for fusion devices with particular emphasis on first wall materials including damage studies on metals as well as welded metal joints and brazings, graphite base first wall coating materials, and molybdenum and molybdenum alloys to be used as divertor materials, materials for super conducting magnets, and the investigation of tritium release kinetics from different ceramic lithium compounds under irradiation.

As a new project under the German contribution to the supplementary programme, a "Healthy Tissue Tolerance Study" in preparation of patient treatments with boron neutron capture therapy (BNCT) has been started in co-operation with the University of Bremen at the Petten-BNCT facility at beam tube 11.

The Netherlands contribution to the HFR supplementary programme addresses two different areas, namely contributions to the European Fast Reactor (EFR) and fusion research programmes on the one hand, and use of the beam tubes for solid state and materials research on the other.

All the Netherlands projects are managed via Energieonderzoek Centrum Nederland (ECN).

For the EFR programme, radiation damage studies on structural materials are performed. The studies comprise crack propagation investigations on compact tension specimens and irradiation of samples for post-irradiation creep fatigue testing. Different stainless steels are under study.

Within the fusion programme, martensitic steels and vanadium alloys are irradiated at different temperature and fluence levels. Special attention is given to irradiation induced changes of fracture mechanics properties. This programme includes amongst others electron beam and plasma welds. The programme on irradiation testing of tritium breeding ceramic blanket materials, where ECN is co-operating with a number of European research centres, has made good progress. The main objective is to obtain data on tritium residence times in a variety of different zirconates, aluminates and silicates at different irradiation temperatures and lithium burnups. Special attention is given to the dependence of tritium release kinetics on purge gas chemistry.

Five beam tubes, HB1, 3, 4, 5 and 9 at which dedicated neutrons spectrometers are installed, are in permanent use for condensed state physics and materials science applications. The topics addressed by the present programmes are the following, with the method applied indicated in parentheses:

- crystal and magnetic structures of organic and inorganic substances (neutron diffraction);
- magnetic and structural phase transitions, phase diagrams (neutron diffraction and critical scattering);
- structure, order and disorder in solid, liquid and amorphous alloys (neutron diffraction and diffuse scattering);
- phonons, magnons, crystal-field excitations in crystals (neutron inelastic scattering);
- residual stresses in materials (high resolution neutron diffraction);
- texture determinations (neutron diffraction);
- disperse systems, colloids, polymers, precipitations, void formation, porosity (small-angle neutron scattering).

In the course of modifying and upgrading the neutron scattering facilities, a new spectrometer, dedicated to residual stress measurements, was taken into operation; first tests demonstrated its high potential in this technically highly relevant application.

Another beam tube, HB7, is applied for ECN's contribution to the BNCT programme. The equipment installed at this beam tube has successfully been applied for prompt gamma determination of boron concentrations in the blood which is one of the most important pharmacological parameters in BNCT.

As in preceding years, JRC has made efforts to prepare the HFR for future neutron applications. At the neutron radiography installation, in co-operation with ECN, images from aircraft and space components were taken, and the potential for the detection of corrosion in components and structures of aging aircrafts were investigated. Again in co-operation with ECN, neutron transmutation doping of silicon was performed on an experimental level, and a feasibility study started on a large scale production facility. Progress was made on the development of an in-pile rig to study the influence of reactor irradiation on thermal fatigue of fusion materials. With the BNCT facility at HB11, first full power tests were achieved, and the facility was used for phantom, cell culture and large animal model studies. This facility is now ready for being exploited under the supplementary programme (see above);
it is mentioned that the BNCT project was supported from the JRC exploratory research fund.

The reactor itself was operated smoothly with the high availability of 266 operation days corresponding to about 100% of the scheduled operation time; the high reliability of the facility is demonstrated by the fact that only five unscheduled reactor shut-downs occurred. The regular three annual in-service inspection of the reactor vessel gave no indication of any unexpected detrimental effect.

More detailed information on the HFR programme, operation as well as utilization, is given in the Annual Report, ref. /1/.

**Reference**

1. Annual Report 1991
   Operation of the High Flux Reactor,
   EUR 14416 EN

# 7. S/T Support to the Services of the Commission

# Standards for Advanced Ceramics

The general objectives of this activity are support to and stimulation of European standards and prestandards and the execution of research and development within European standardisation activities. The project supports the Directorate General "Internal Market and Industrial Affairs" section: "Standardization and Certification". The following results were achieved during the reporting period.

**Standardisation Activities:**

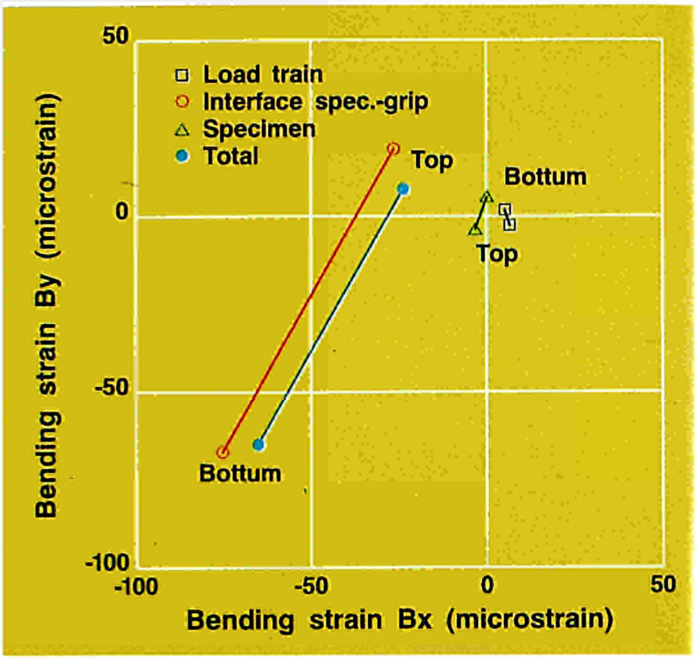
The European Standard Organisation CEN executes under mandate of the Commission a programme for the development of 42 standards and prestandards. The technical committee (TC) 184 "Standards for Advanced Technical Ceramics" set-up to coordinate the activities operates five working groups (WG) to which Institute scientists contribute in four cases. For the development of a classification standard a tri-lingual terminology is under preparation.
Working groups submitted testing standards on composites (tensile, compressive, flexure and shear at room temperature), and a draft standard on flexural testing of monolithic ceramics at room temperature.

**Pre-Normative R&D:**

- *Ceramic Corrosion*

A world-wide survey aiming to establish the level and types of activity in ceramics corrosion research, corrosion test methodologies used, attitudes towards standards and the willingness to collaborate in a standards programme was completed.
The survey revealed an active programme, in Japan, on test methodology development.
Under the auspices of JIS (Japanese Industrial Standards), the Japanese have published standards for a number of physical and mechanical property test techniques as well as one for high temperature oxidation in air (JIS-R-1609).
No other oxidation/corrosion standards for engineering ceramics are known to exist and no other prestandards work is known to be in progress.

The next step will be with the involvement of representatives from research and industry to establish the critical components of a programme to produce the appropriate standards.




**Above:** *Contributions of different origin to the total bending strain*

**Mechanical Testing of Advanced Ceramics**

In the project on uniaxial testing of monolithic ceramics and ceramic matrix composites at high temperatures, the different contributions of load train, grip system and specimen geometry to the total misalignment have been separated and quantified.

For a system based on a rigid load train, these contributions at the top and bottom end of the gauge length are shown in figure above.

For well-machined specimens the largest contribution by far is caused by the clamping between grip and specimen. The temperature distribution along the gauge length of a CMC specimen heated by induction through a susceptor has been optimised.

The performances of a non-contacting extensometer based on the laser-Doppler effect have been critically assessed.

**Flaw Sizing in Ceramics, by means of Ultrasonics**

*Sizing of surface breaking-cracks in reaction-bonded silicon nitride.*

Radial cracks resulting from Vickers indentations in reaction bonded silicon nitride have been examined by means of ultrasonics at a frequency of 30 MHz.
The sizes of the radial cracks varied between 100 and 600 microns.
Surface waves allowed to detect all radial cracks whereas $45^0$ shear waves did not allow to detect the smallest radial cracks of 100 microns.

The limits mentioned above are determined by the frequency of 30 MHz used.

Further investigations will examined whether increasing the frequency will allow to detect and size smaller cracks.

*Development of a mathematical model to optimize sizing and detection procedures of surface-breaking cracks.*

Surface waves generated by a focalised ultrasonic beam are a means to detect and size small surface-breaking cracks. A model has been developed which allows to calculate the normalised amplitude of the echo of these surface waves by a surface-breaking crack as a function of the position of the probe (the echo dynamics curve).
The shape of the echo-dynamics curve is well reproduced, especially the shift of the maximum amplitude of the echo on the crack (due to surfacewaves) with respect to the position of the crack as well as the exponential increase. The model will be used to optimise the detection and sizing of surface breaking cracks by designing special transducers.
Future activities will be focused on the calculation of the influence of the orientation and the finite dimensions of the surface-breaking cracks on the detection and sizing procedures.

# Standards and Codes for Non-Destructive Testing of Pressure Vessels and Weldments

The objective of this new activity is to support the development of European (pre)-standards and codes mandated by the Directorate General: Internal Market and Industrial Affairs (DG III) and to stimulate and coordinate Round Robin Actions.

At the end of 1990, the NDE Unit has taken the co-leadership with Siemens and KWU of a PISC Working Group for direct support to Codes and Standards (C/S). This action has the following objectives:

- Informing the relevant C/S Technical Committees of PISC results and PISC related programme results.
- Critically reviewing by PISC members of technical documents made by national, international technical groups on request.
- Preparation of technical reports related to Codes and Standards problems, for the benefit of National, CEN, ISO, IIW technical groups which elaborate standards.

Present support is given to pre-normative documents:

- handbook on the inspection of cladded pressure components,
- handbook on the validation of welds inspection techniques.

These documents are elaborated by groups of experts now managed by the NDE Unit in view of their use by the CEN Technical Committees, in particular TC 121 (Welding) and TC 138 (NDT).

During the reporting period the NDE Unit contributed to the following committees:

- CEN TC 121, coordinating and discussing the work of all the working groups and the elaboration of a general document involving all main NDE techniques: Design and Non-Destructive Examination of Welds.
- CEN TC 121 WG2, (Ultrasonic NDE) elaborating the following documents:
  - Ultrasonic Examination of Welded Joints,
  - Acceptance Levels for Ultrasonics Examination of Welds.
- CEN TC 138, coordinating and discussing the work of all the working groups and the elaboration of the document "Certification of NDT personnel".
- CEN TC 138 WG2, (Ultrasonics) elaborating of the following documents:
  - "General document" subdivided in: General principles; Sensitivity and Range setting; Transmission technique; Transfer correction; Geometrical conditions; Tandem examination; Characterization and Sizing of discontinuities.
  - "Characterization of the UT Equipment"

Specific actions are prepared and to be sponsored by the PISC programme in Support to Codes and Standards which will aim at Performance Demonstration Exercises for the support of Perform ance Standards for the inspection of pressure components welds.

# Materials Science and Technology for Aeronautics Application

The objection of this new activity is to produce an analysis of the European position with respect to construction materials employed in the aeronautic industries and to elaborate a strategic plan for materials science and technology in support of European industrial competitivity.
In the frame of the Directorate General: International Market and Industrial affairs (DG III), the Institute contributes actively in the field of technology and advanced materials to the development of the 'European Competitive Strategy in the Aeronautics Field'. During the year, our role was focused on providing expertise and advice to DG III on Aeronautics technology and materials implications. An important aspect of the work was related to the preparation of state of the art reports on materials in the Aeronautics Technology and in pinpointing future materials needs. The Institute participated in various meetings with European Aeronautics Industrialists to evaluate the future strategic technology lines. Actions are being taken to prepare the "materials strategic programmes".

# Standardization of Quality Control Protocols for Radio-Pharmaceutics

At the cyclotron laboratory further studies on the development of a $Ge^{68}$-$Ga^{68}$ generator in support of DG XI have been performed. This work relates to the Council Directives on the radiation protection of the general public and workers and of persons undergoing medical examination and treatment.
The separation method for $Ge^{68}$ by liquid-liquid extraction as described in the literature has an efficiency of only 70%, involving several repetitive steps and the risk of evaporation of the highly volatile $GeCl_4$ from concentrated HCl solution. A new separation method, based on cation exchange, has been developed. The efficiency is better than 90%, repetitive steps are not required and $GeCl_4$ evaporation omitted.
As a result, a lower initial activity is needed for a certain generator (reducing the irradiation time, the costs and the radiation danger for the workers), and the risks of ionizing radiation is reduced.
The commercially available generators showed a reduced elution efficiency due to entry of air into the generator. A better design of the generator and of the loading and elution process has facilitated stable elution efficiency over a much longer period (until now 4 months), leading to an decrease of the required activity loaded onto the generator. This work, to be finished in 1992, has an important social as well as commercial value, since it may lead to a widespread adoption of the $Ga^{68}$ radioisotope in medical diagnostics.
In support to the same Council Directives a course in collaboration with and sponsored by DG XI in the framework of the EUROCOURSES is prepared. The three day course addressing cyclotron production, quality control and utilization of medical radionuclides is scheduled for July 1992 and intended for technicians and paramedics working in this field. The underlying idea is that a better understanding of all the different steps involved in the production of radionuclides and radioactive pharmaceutical and in the use in nuclear medicine, reduces the hazards of ionizing radiation.
The course comprises theoretical lessons with emphasis on radiation protection, practical participation in isotope production at the cyclotron, visits to a hospital, using new diagnostic instruments and to a pharmaceutical firm, committed to the production of radioactive pharmaceutical.

# The Rôle of Materials in Environmental Problems Arising from Power Station

This wide subject was reviewed and presented in a report to the Directorate General: Environment (DG XI).
The effects on materials of limiting pollution from power engineering plant were described and requirements for new materials delineated. In line with the priorities of DG XI/A/3 the report concentrated on the gas turbine cycle but also introduced the analogue developments in the steam cycle and in the growing "Clean Coal" technology.
In the gas turbine cycle, the use of "rich burn" plus quench or lean bum technology leads to a reduction in air availability for cooling purposes in the combustion chamber, static and rotating blades.
Thus the demands for higher temperature capability are increased.
To meet these demands new materials and production processes will be required. As an example large blades produced by advanced single crystal blade technology will be needed as will thick thermal barrier coatings of assured quality.
Such coatings will also become design features in combustion chambers.
This report was presented to Gas Turbine emission specialists, representing producers and many utility users in Europe and was included in their discussion which centred on draft proposals for limits in $NO_x$, $SO_2$ and CO.
Attention in 1992 will move towards "Clean Coal" technology for gas and steam turbine power production and similar assistance with materials consequences will be given.

# Ceramic Catalyst Carrier

This project supports the Directorate-General "Environment", and has as its general objective to investigate the degradation mechanism of ceramic catalyst carriers, applied in exhaust systems of automotives.

These catalyst supports are manufactured as highly porous, thin-walled honeycomb structures to give the large surface area required for efficient catalytic operation. Ageing of this material leads to failure at higher mileages and occurs by:

* corrosion during thermal overloading in the aggressive exhaust gas;
* poisoning by Pb, Zn, P, S from fuel and additives;
* dynamic fatigue from thermo-mechanical stresses including temperature gradients under non-stationary operating conditions.

A state-of-the-art review concerning the application and integrity of automotive catalysts systems has been prepared, along with a corresponding test programme, which includes both, steady-state corrosion tests as well as burner rig tests in simulated exhaust gas atmospheres. The mechanical integrity of the catalyst carrier will be tested by thermal shock and dynamic fatigue measurements.

Microstructural examination after pre-determined exposure times will be used for the material characterization.
The material chosen is cordierite, a magnesium aluminasilicate ($2MgO+2Al_2O_3+5SiO_2$), currently the only ceramic catalyst support in use.

The preparation of specimens is underway and the first tests will commence early in 1992.

# Materials Data Systems and Standards

The increasing computerization of engineering activities had in the early 1980's stimulated the emergence of advanced factual data systems on materials property data. Although a considerable number of such systems were in operation in the EC, they were far from being mature and adequate for integration with engineering software such as CAD/CAM and CIM systems. The Commission has therefore taken initiatives to advance the integration of operational European data systems into a pilot network for materials information services. This "Materials Databanks Demonstrator Programme" was implemented by DG XIII/B in close cooperation with JRC Petten, thus combining information market expertise with appropriate materials science competences.

The evaluation of the Demonstrator Programme in 1990 has shown that there are still problems due to inadequacies of hardware, operating systems and their standardization, but that these are of minor importance compared with two main deficiencies:
- the lack of establishment and perception of the value of materials information,
- the lack of harmonized international standards for materials information.

Considerable effort has therefore been expended in devising a new programme for materials information for the proposed IMPACT 2 programme of DG XIII for 1992-1995 which has taken off with the following new objectives for the materials field:

- to make European information on engineering materials more accessible to more sectors of the manufacturing economy and the research community;
- to establish a European identity for that information by improvements to quality assurance, harmonisation and standardization in order to increase the impact of the world market;
- to create an industrial structure for the materials information sector;
- to enable large manufacturers to use materials information more efficiently;
- to encourage small-to-medium enterprises (SMEs) to make better use of the information which is already available.

The main method of realising these objectives will be by establishing demonstration-type and co-operative projects between producers of information, producers of computerized information systems and users of information in the manufacturing sector.

There have been several actions in support of the forthcoming IMPACT 2 Programme to the effect that scientific and technical information receives more recognition as a strategic industrial resource for the EC.

Most effort has been expended to assist the development of the materials resource model within the ISO development of a Standard for the Exchange of Product Data (STEP).

This is a major international effort involving many hundreds of engineers and computer specialists, strongly supported by major manufacturing companies.

The materials resource model is being developed on the principle that an engineering material is a product of a process and its numerical "properties" are the outcome of a particular measurement procedure in a specific set of circumstances. The combination of the measurement procedure and the circumstances is the data environment. The process of development involves identifying the relationships between the entities and their attributes which derive from these combinations of concepts and writing the relationships in EXPRESS. The relationships are also described in a diagrammatical form.

It has become clear in the last few months that the international cooperation on the materials model has not led to the adequate representation of European concepts, despite the fact that much of the intellectual basis is European in origin.

A recommendation has therefore been made to CEN to establish a development of the materials model as a work item for a draft European Standard derived from the ISO document.

This will all be combined with the development of Application Protocols within areas of specific interest to European industry which would have the effects of refining the basic resource model and identifying further requirements and so increasing the European influence of the resource model. Furthermore there is a need to have the materials model regarded as part of the considerable CEC effort to support research into CIM through the ESPRIT programme. The testing of the model by populating it with high temperature materials test data is well within the scope of the Institute and possibly within the scope of a contribution to VAMAS.

Both information market policy and marketing promotion of materials data systems require the accurate and meaningful description of databanks which has turned out to be an essentially unsolved task. This problem was studied with the aim to develop a standard recommendation. The results of the research showed that a new approach to this problem could be adopted based on the mathematics of sets. A practical demonstration of this approach was devised by using hypertext methods and demonstrated.

The activities of this sector have produced a significant basis of prenormative achievements in cooperation with national standards institutions and international bodies, in particular VAMAS and CODATA. In cooperation with industry they have produced analysis of the requirements to develop standards interfaces for systems integrating materials information into computer aided engineering systems where the main objective is data interchange. De-facto standards for sectorial material information are expected to result from industrial associations such as ALUSELECT and CAMPUS. In order to strengthen the prenormative R&D support to the European standards organizations in all sectors relevant to industrial technology and the integration of the European market in 1992, the creation of a new European organization for the coordination of prenormative research is under special investigation.

# Valorisation of Research Results

The objective of this activity aims to support the Directorate General: Telecommunications, Information Industries and Innovation (DGXIII) with the licensing of JRC patents.

## Spontaneous Downward Heat Transport

The objective of the activity is to demonstrate to industry that the new heat transport system is efficient and reliable under the severest conditions, and that new applications of solar thermal energy become possible.

After the completion of the campaign of comparison tests, the spontaneous system has been kept in operation for the whole year, exhibiting continuously full reliability.

The prototypes for snow melting and hot water production installed by two mountain refuges, at 3650 and 2100 m, were always in operation and have been continuously monitored.
They operated throughout the winter season without any trouble. In spite of a mean ambient temperature during the winter of −9°C and a minimum of −30°C, liquid water was always available, with a mean temperature of 22°C.

Another plant has been designed, built and installed in a refuge at 2500 m. In this case solar thermal energy is used for heating the digester of a plant treating the sewage of the refuge.
In this way the temperature of a anaerobic digester will be kept between 20 and 30°C, facilitating a relatively fast biological decomposition processes.

An Italian company has signed the second licensing contract for the commercialisation of this system in Italy. Contacts have been strengthened with Greek Industry.

## Ultrasonic Reference Transducer

The Reference Transducer (now called Standard Acoustic Source SAS 01) is a high precision, stable transducer, equipped with its own electrical excitation source, for the creation of standardized ultrasonic waves for transducers and hydrophones calibration.

The development of the system and the realisation of the first prototype are the results of the collaboration between INSA (Lyon), DASSAULT Aviation (St. Cloud) and our NDE laboratories.

During 1991, the information required for the licensing of the SAS 01 was prepared and communicated to the firm IMASONIC.
The main conditions of the contract were defined together with the DGXIII Direction C, the NDE laboratories were charged to prepare the technical annex.

Such an annex will contain drawings, electronics schemes, description of the software part and instructions for the fabrication and mounting of the SAS 01.

Two industrial prototypes are to be constructed and delivered in 1992: the first one to the "Centro Italiano per la Validazione delle Apparecchiature Biomediche" (Trieste, Italy); the second one is requested by the firm DORNIER (Germany).

## Oxygen Sensor

The $CeO_{2-x}/Pt$, cermet electrode for an electrochemical oxygen or lambda-type sensor has been proven to yield correct thermodynamical response in a reducing as well as in an oxidizing atmosphere.

In a reducing atmosphere, in fact, the $CeO_{2-x}/Pt$ component of the cermet is prevalently acting as the measuring compound, by its fast equilibration process; in an oxidizing atmosphere, on the other hand, Pt is the acting electrode whereas stoichiometric $CeO_2$ is inert.

The advantage is the coverage of an oxygen partial pressure interval ranging from $10^{-30}$ atm to 1 atm by a single electrode.

The problems arising in reducing atmospheres for the classical porous Pt electrode of conventional lambda probes is, due to the stability of $CeO_{2-x}$ and the dispersion of Pt in the cermet, not met in this case.
The cermet electrode shows therefore a very long lifetime.

In addition to the $CeO_{2-x}$/Pt cermet, a cermet $CeO_{2-x}$/Pt/$PrO_{2+x}$ has been investigated as an electrode, where both Pt and $PrO_{2+x}$ equilibrate in oxidizing mixtures. $PrO_{2+x}$ shows a very fast response. Pt, in this case, acts as the measuring element in the rather difficult regime of oxygen partial pressures, ranging from highly reducing to highly oxidizing atmospheres.

# 8. Exploratory Research

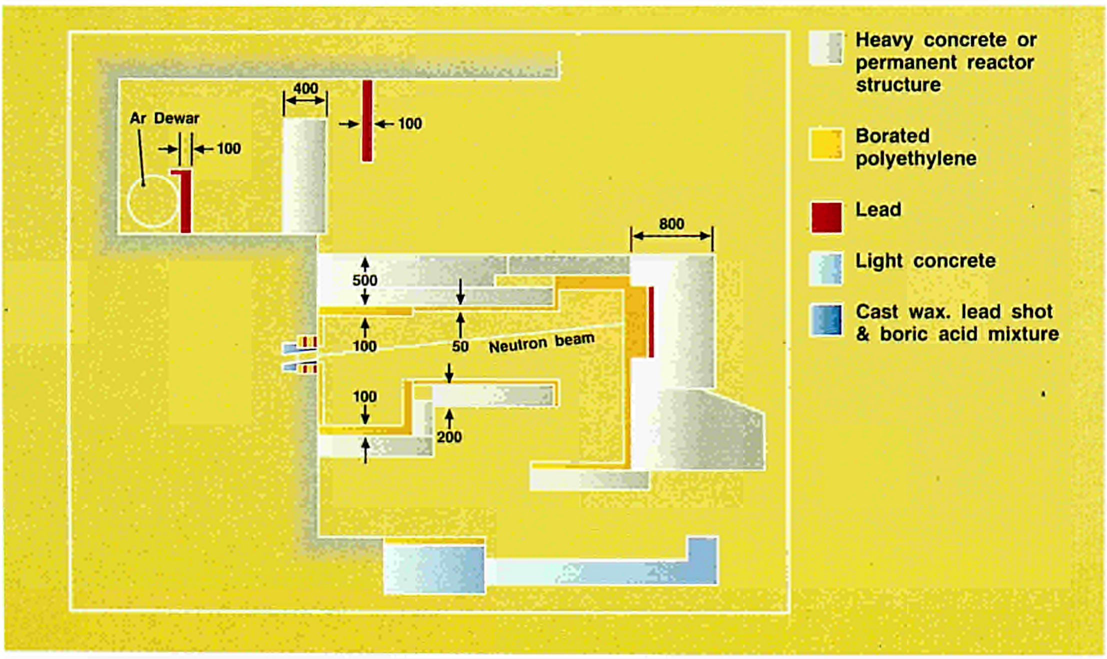
# Boron Neutron Capture Therapy (BNCT)

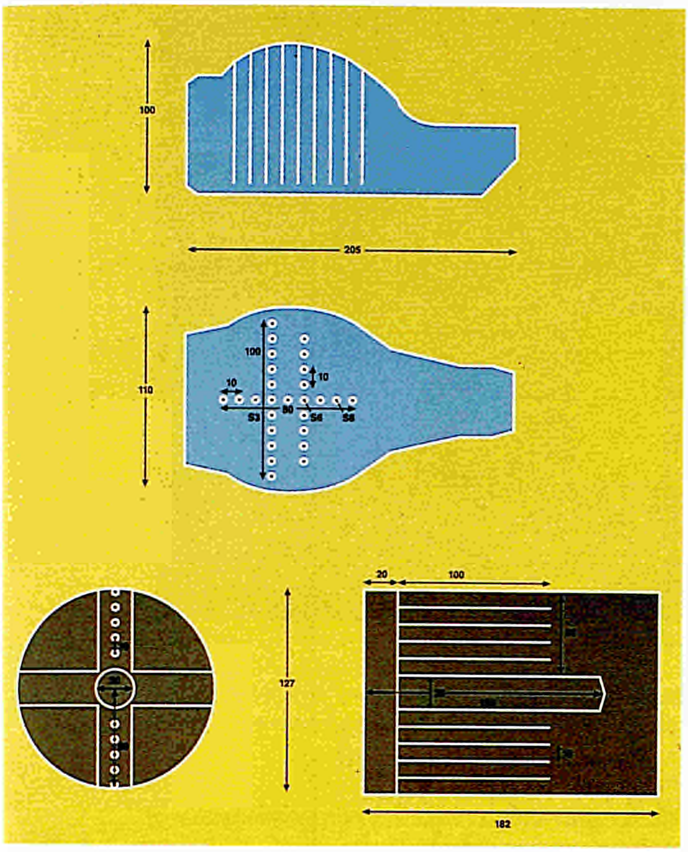
During 1991, the BNCT project at Petten has progressed further towards its intended goal to treat glioblastoma (brain tumours) patients at the High Flux Reactor (HFR).

In the early part of the year, it was necessary, due to stringent reactor safety requirements, to modify certain components of the facility that were potential sources of unacceptably high background radiation. Also, many of the safety and operating components had to be duplicated before the reactor safety committee was satisfied that the facility could be operated in a safe manner. It was finally possible, therefore, to open the facility for the first time at full reactor power in June 1991.

The present configuration of the irradiation room is shown in figure below. Over 40 tonnes of heavy shielding material has been installed to complete a fully- shielded room, in which all the experiments prior to ciinical trials may be performed. In June/July, the first nuclear measurements were performed to determine the neutron and gamma properties of the beam. Measurements were carried out at low reactor power using proton recoil spectrometry techniques in collaboration with colleagues from AEA Harwell UK, and at full reactor power using activation foil sets and thermoluminescence detectors, in collaboration with colleagues from INEL, Idaho, USA and ECN Petten. The results of the measurements determined the flux rates and spectra of the neutron and gamma fields of the beam, both in free air and in lucite (acrylic plastic) phantoms. Figure on page 00 above shows the geometry of the phantoms, indicating where finely drilled holes are distributed, in which activation foils and wires, and thermoluminescence detectors can be placed to measure the 3D-isodose distributions. The results of the measurements were compared with calculations performed at JRC Petten, using the MCNP code, and JRC Ispra, using the DORT code. Both sets of calculations agreed within acceptable limits. However, both overpredicted the results for the flux density, compared with the experimental measurements.

**Below:** *Horizontal cross-section through BNCT facility (present configuration), indicating treatment area (irradiation area) and lay-out of shielding*





**Above:** *The shapes and dimensions (in mm) of the beagle-head and cylindrical phantoms, showing the fine-drilled holes for placing foils and TLD's*

At both Ispra and Petten this was later found to be due to inadvertently neglecting the relatively high thermal neutron capture property of $^{36}Ar$ that is present to only 0.34% in natural argon.

The argon, in a liquified state is one of the major filter components in the beam tube. Following revised calculations, both sets of results agreed with measurements.

With agreement reached, it was then possible to perform some initial radiobiology experiments at the open beam. Cell culture irradiations were performed in tissue equivalent phantoms. Cells at different depths in the phantom and containing different concentrations of boron were used. The aim of these experiments is to determine the "killing" effect of the beam as a function of boron and depth.

The first 2 canine experiments out of a total of 45 experiments were performed. These experiments, which are the most important of the whole experimental programme, aim to determine the healthy tissue tolerance of the canine brain to the neutron beam. This information is necessary before treatment of human patients with glioma may be carried out. The healthy tissue tolerance study determines the limiting dose at which the patient may be irradiated without causing any physical or neurological damage to the healthy brain.

Following the reactor summer stop in August, the facility has undergone major modifications. These modifications will improve the reliability and operation of the system, which will not require any further modifications when patient treatments begin.

Computer modelling continued with the implementation at JRC Petten of the latest available version of the MCNP code. Work at JRC Ispra has been towards the upgrade of DORT to TORT, a 3-dimensional code. The Petten work is aimed towards the development of a treatment planning code that will be used by the radiotherapists at the hospitals where the patients will be pre-treated prior to BNCT. The code will be verified by comparison of results from phantom experiments. Computer modelling at Ispra continues towards, amongst other tasks, seeking possible alternative filter configurations that could improve the quality of the neutron beam even further.

Work at the JRC Ispra Cyclotron has continued towards the development of positron emitting radioisotopes to be used in P.E.T. diagnostic systems which could assist in investigations of the distribution of boron in the brain.

# Joining of Ceramics to Metals

Joining is one of the enabling technologies to permit the use of engineering ceramics in todays advanced and complex structures. This project has been studying in depth ceramic bonding, guided by interfacial principles and assisted by surface engineering, with a view to high temperature (HT) structural applications.

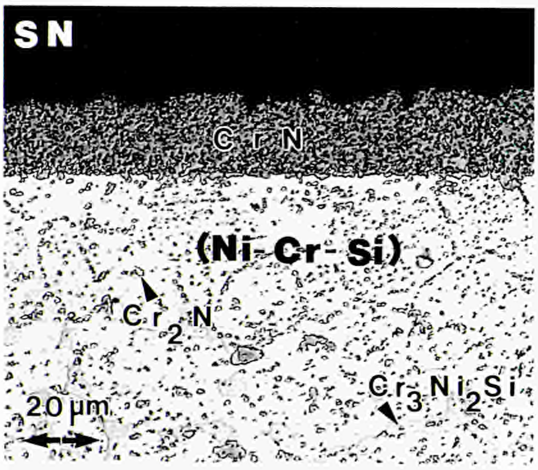
1. Joining of Silicon Nitride Ceramics

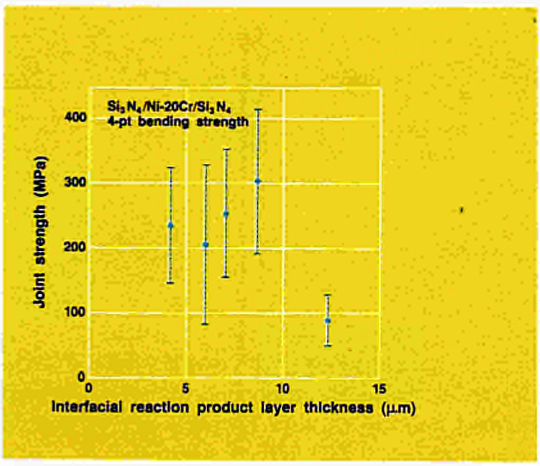
The interfacial microstructure and microchemistry, as well as the bond evolution and kinetics in $Si_3N_4$/Ni, Cr, Ni-Cr and Ni-Cr-Si alloys has been determined for a wide range of joining conditions namely, 900-1300$^0$C in different environments (vacuum, Ar, $N_2$).
Various reaction products, such as Cr-nitrides and metal silicides are formed depending on the processing conditions (figure below left).
In the absence of interfacial melting, caused by Si ingress and low temperature Ni-Si and Ni-Cr-Si eutectics, the reactions are dominated by the interdiffusion of the system constituents. The reactivity depends, most notably, on the Cr-content of the alloy and the $N_2$ partial pressure in the environment, which in turn control the dissociation, and subsequent dissolution of the ceramics.

From measurements of the kinetics of bond formation the long term thermal stability of the joints can be predicted, while the joint design, e.g. with respect to the interlayer thickness, can be optimized.
A certain reaction product layer thickness (5-10µm) is found to be the optimal for the joint strength (figure below right), while the bonding environment and pressure affect mostly the joint reliability. Preliminary studies have shown that $Si_3N_4$ can be directly bonded to Ni- superalloys via diffusion bonding. However, the use of ductile metal (Ni)/low expansion metal (W) as bonding interlayers results in sounder joints.

**Below left:** *Cross-sectional view of a $Si_3Ni_4$/Ni-20Cr joint showing the interfacial microchemistry (joining conditions: 1100$^0$C, Ar, 25h, 50MPa. SN: Silicon Nitride).*

**Below right:** *Flexural strength of $Si_3N_4$ joints as a function of the interfacial reaction layer thickness.*




2. Ion Implantation into $Si_3N_4$ and Ion Beam Mixing of $Si_3N_4$/Metal Systems

Ion beam mixing has been observed in the $Cr/Si/Si_3N_4$, $Si/Si_3N_4$, $Cr_2O_3/Si_3N_4$ systems and a limited, ballistic only, mixing in the $Cr/Si_3N_4$ system. Ion implantation induced subsurface amorphization of the ceramic results in a significant surface softening (25%) and surface crack blunting, thus improving wear properties and flexural strength distribution.

**Above:** *XTEM image of cross-section of $Si_3N_4$ implanted with $7x10^{15}$ $Si^+/cm^2$ of 1 MeV at 100K, showing a subsurface amorphous layer*

# Micro-Hydrodynamics of Laser Melted Pools

A well known, costly phenomenon, flux line erosion, has for years bedeviled the pyrometallurgical and glass making industries. It consists in the scouring or erosion of the refractory crucible in zones adjacent to where the interface between the metal and the slag (or the glass and its layer of flux) meet the refractory. Numerous patterns of erosion have been reported, such as waist formation, pin-holes, necking and cut formation.

We considered first the case of buoyancy convection alone in a steel/slag system, assuming that density and thermal conductivity are 7200 $kgm^{-3}$, 15.48 W/(mK) for fluid A and 2850 $kgm^{-3}$, 10.46 W/(Mk) for fluid B respectively.

Figure on page 00 shows the solute concentration gradients in both phases and the corresponding flow patterns.

As expected, the direction of motion is counter clockwise in both phases, hence creating opposing flow fields at the interface. This gives rise to a particular flow configuration and an erosion profile consisting of a relatively shallow feature along the refractory wall with the exception of a groove in the vicinity of the interface between phases A and B. This represents the typical erosion profile found for a wide range of buoyancy convection values. We note that this is not the characteristic shape found in practice in pyrometallurgical or glass making furnaces.

Another idealized case is that of Marangoni flow in the absence of buoyancy flow. We assume that the dissolving species diffuse to the phase interface and produce either a positive or negative concentration gradient of absorbed species with distance from the vertical wall.

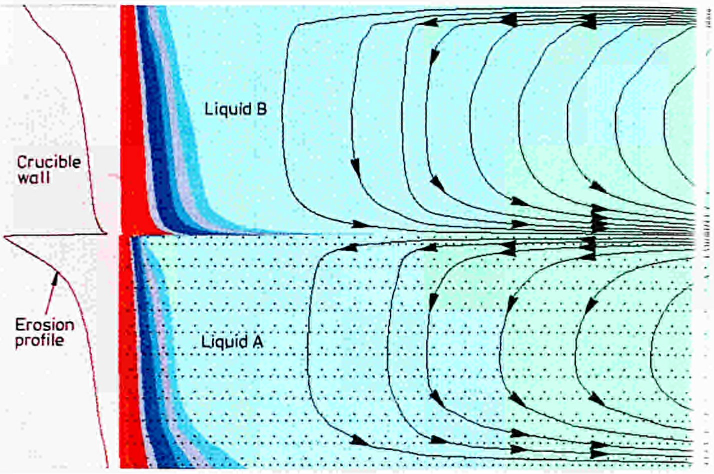
The latter event is seemingly the most likely. However, a positive adsorption concentration gradient at the interface from dissolving species may arise, in physical terms, in the following manner: surface active solute diffuses from the refractory wall to the interface at some distance from the wall. Here, the surface tension is reduced locally and if the adsorbing molecules transfer to the adjacent liquid phase, this can be easily replenished by diffusing matter coming from the interior and aided by upwelling flow streams. On the other hand, closer to the wall, imbalances may occur because the dissolving substance may sink through gravity and there is no equivalent reservoir for replenishing the adsorbate if it moves into the adjacent fluid phase. This results in a negative interfacial tension gradient, that is, the locality closer to the wall has the higher surface tension. Alternatively, such a gradient could arise in the case of a strongly adsorbing impurity species which may provoke a desorption of a weakly adsorbing species.
For example, figure below depicts the expected behaviour for a negative capillarity gradient, that

is $\frac{\vartheta\gamma}{\vartheta\chi} < 0$, for which $\frac{\vartheta\gamma}{\vartheta c} > 0$ where x is the

distance from the vertical wall, and c is the concentration of diffused species.

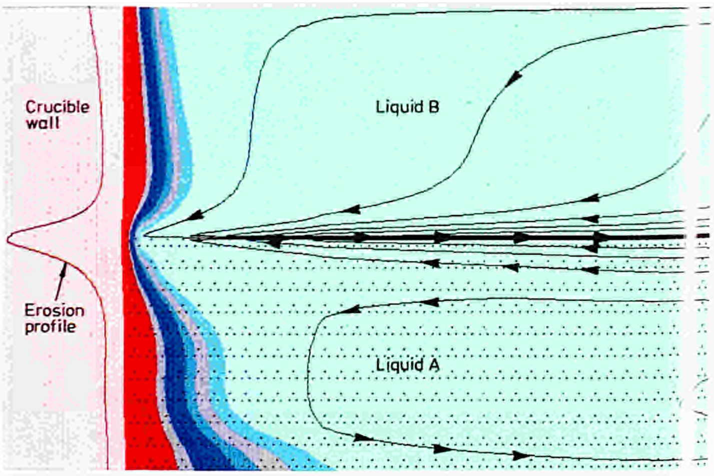
In our experiments chemical agitation effects were neglected. The problem is formulated by considering the Navier-Stokes equations coupled with the energy and diffusion equations. The relative contributions due to interfacial tension and buoyancy flows in determining the shape and rate of the erosion profile were studied. It was demonstrated that the erosion profile is influenced by the local increase of surface tension resulting from the dissolution of the wall material (refractory).

Two different cases have been investigated. In the case of buoyancy convection alone, figure above, the direction of motion is anticlockwise in both the slag and the molten phases, hence creating a shear field at the interface. The corresponding erosion profile shows a characteristic cut in the refractory which is not found in practice.



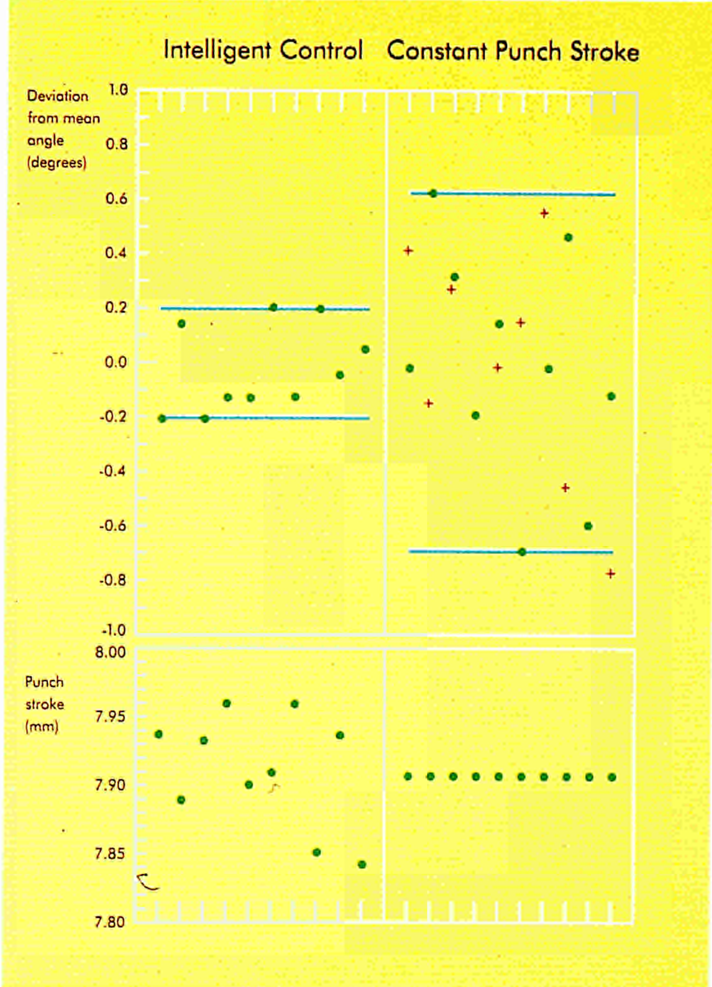

**Above:** *The solute concentration gradients and the flow patterns in both slag and molten phases, for the case of pure buoyancy convection. The corresponding erosion profile shows a characteristic cut in the refractory which is not found in practice. Concentration ranges from that of the refractory wall 0.08 (rose) to 0.96 (light blue)*

**Below:** *Similar distributions as in fig. above for Marangoni interfacial convective flow. The flow pattern in the slag has been completely reversed and the corresponding erosion profile is more symmetrical around the interface*



# Development of Intelligent Processing for Sub-Micron Ceramic Structures

An experimental study on the application of intelligent processing (IP) was undertaken to gain practical experience with computer control and modelling in the IP context and to explore the practical benefits. As the objective was to gain generic expertise it was decided to study a conveniently implementable process for which a process model and suitable sensors were already available, e.g. brakeforming. Brakeforming is a commonly used method for bending sheet metal with a hydraulic press by forcing the sheet into a concave die using a punch. The process is simple and cheap but is inaccurate and therefore offers scope for improvement through better control. By adding sensors and a computer the application of IP has upgraded a simple hydraulic press into a combined materials characterization and processing rig. Real-time, insitu characterization of the physical properties of each workpiece while it was being bent enabled the punch displacement to be adjusted of each workpiece to compensate for variations in elastic spring-back when the load was removed. The benefit of IP to brakeforming was demonstrated by the reduction in the scatter in bend angle by a factor of 3 over the standard process (see fig. above). Poor reproducibility in the standard process was traced to variations in elastic modulus of the workpieces, even for nominally identical material from the same batch. The lessons learnt from this study were that a) the physical implementation of IP is relatively easy provided that suitable hardware is available but b) the development of a suitable process model can be very time consuming.




**Above:** *Comparison of scatter in bend angle for intelligent control of punch stroke and for fixed punch stroke. Results for a 90 degree bend on 4mm thick 316 stainless steel.*

# Ceramic Fibres for High Temperature Composites

An exploratory project has been launched to study the problems of stabilisation of high strength ceramic fibres in ceramic- matrix composites, (currently the most promising next-generation materials for structural applications at extreme high temperatures). In-house competence for ceramic composite fabrication by slip casting and hot (1900$^0$C) densification directed the choice of composite and processing route to slurry-infiltrated carbon fibre.

A literature review combined with surveys of industrial needs has led to the selection of PAN or pitch-based carbon fibres in a silicon nitride matrix, as the system to be investigated.

The fibres need to be coated in order to protect them from chemical attack by matrix and environment and to accommodate interphase mechanical stresses induced by differences in thermal expansion co-efficient. Development of fibre coatings, therefore, forms an important part of the project.

A study of the thermodynamic relationships of the composite materials with a number of candidate coatings led to the selection of the following coating materials: SiC-C(pyr), C(pyr)-SiC-C(pyr) multilayers and BN for first investigations.

Initial experimental studies have concentrated on matrix slurry rheology and fibre wetting, and on the development of fibre array geometry.

This R&D activity is coupled with the JRC-IAM-launched EUREKA-CEFIR project, which is a multi-disciplinary industrial collaboration, including the major potential users and producers of HT ceramic fibres and composites in Europe.

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# IV Meetings/ Conferences

# Meetings/Conferences

| Date | Venue | Subject Area | Type of Meeting | Co-organiser/Co-Sponsor |
|---|---|---|---|---|
| 15-1-1991 | Ispra | Mechanics of Materials | Seminar | University of Pisa |
| 27-2-1991 | Milano | Solid State Chemical Sensor for Gases | Expert Meeting | Milano-Ricerche |
| 6/7-3-1991 | Petten | Standards for Ceramic Matrix Composites | Expert Meeting | CEN-TC184 |
| 18/20-9-1991 | Petten | Clinical Trials of Glioma with BNCT | International Workshop | CEC/DG XII - Concerted Action |
| 11/13-11-1991 | Petten | Designing Ceramic Interfaces | European Symposium | ------ |
| 27-11-1991 | Petten | Pollution Emission form Gas Turbines | Expert Meeting | CEC/DG XI |

# Ⅴ Glossary

# Glossary

| | |
|---|---|
| ABAQUS | Finite Element Code |
| ACC | Advanced Coatings Centre |
| AES | Auger Electron Spectroscopy |
| AMCR | Acier Mangan Chrome (Low Activation Material) |
| ANITA | Analysis of Neutron Induced Transmutation and Activation |
| APS | Atmospheric Plasma Spraying |
| ARTIC | Expert System |
| ASME | American Society for Mechanical Engineers |
| ASTM | American Society for Testing and Materials |
| BCR | Bureau Communautaire de Référence |
| BMFT | Bundesministerium für Forschung und Technologie |
| BNCT | Boron Neutron Capture Therapy |
| BRITE | Basic Research in Industrial Technologies for Europe |
| BUMMEL | Bubble Mobility Measurement Level |
| BWR | Boiling Water Reactor |
| C/S | Code and Standard |
| CAD | Computer Aided Design |
| CAE | Computer Aided Engineering |
| CAM | Computer Aided Manufacturing |
| CAPS | Controlled Atmosphere Plasma Spaying |
| CCG | Creep Crack Growth |
| CEA | Commissariat a l'Energie Atomique |
| CEC | Commission of the European Communities |
| CEGB | Central Electricity Generating Board |
| CEN | Comité Europeen de Normalisation |
| CIM | Computer Integrated Manufacturing |
| CISE | Centro Informazioni Studi Esperienze |
| CMC | Ceramic Matrix Composite |
| CNR | Centro Nationale di Ricerche |
| CODATA | Committee for Data on Science and Technology |
| COS | Carburation-Oxidation-Sulphidation |
| COST | European Cooperation in the Field of Science and Technical Research |
| COST 501 | Advanced Materials for Power Engineering |
| CRKPRO | Crack Propagation |
| CS | Co-Sputtering |
| CSIC | Consell Superior d'Investigaciones Cientifiques |
| CSNI | Committee for the Safety of Nuclear Installations (OECD-NEA) |
| CT | Compact Tension |
| CVD | Chemical Vapor Deposition |
| DAQ | Decentralized Data Acquisition |
| DB | Data Bank |
| DBMS | Databank Management System |
| DG | Directorate General |
| DOT | Deterministic Code |
| EB | Electron Beam |
| EBRII | Experimental Breeding Reactor II |
| EC | European Communities |
| ECN | Energieonderzoek Centrum Nederland |
| EDI | Electronic Data Interchange |
| EDS | Energy Dispersive System |

| | |
|---|---|
| EELS | Electron Energy Loss Spectroscopy |
| EFR | European Fast Reactor |
| EGSI | European Group for Structural Intermetallics |
| EMARC | European Materials Research Consortium |
| ENEA | Ente Nazionale Energie Alternative |
| ENEL | Ente Nazionale di Energia Electrica |
| ERD | Elastic Recoil Detection |
| ESCA | Electron Spectroscopy for Chemical Analysis |
| ESPRIT | European Strategic Programme for Research and Development in Information Technology |
| ETL | Environmental Testing Laboratory |
| EURAM | European Research on Advanced Materials |
| EUREKA | European Research Coordination Agency |
| EUROS | European Remote encapsulation Operating System |
| EXAFS | Extended X-ray Absorption Fine Structure |
| FBC | Fluidised Bed Combustion |
| FBR | Fast Breeder Reactor |
| FE | Finite Element |
| FEM | Finite Element Method |
| FRUST | Fusion Reactor Utilisation of Stainless Steel |
| FTIR | Fourier Transformed Infra-Red |
| FW | First Wall |
| GAXRD | Glancing Angle X-ray diffraction |
| GXRR | Grazing incidence X-ray reflection |
| HFIR | High Flux Isotope Reactor |
| HFR | High Flux Reactor |
| HPSN | Hot-Pressed Silicon Nitride |
| HSS | High Speed Steel |
| HT | High Temperature |
| HTM | High Temperature Materials |
| HTMDB | High Temperature Materials - Data Bank |
| HTR | High Temperature Reactor |
| HVOF | High Velocity Flame Spraying |
| IAEA | International Atomic Energy Agency |
| IAM | Institute for Advanced Materials |
| ICP-AES | Inductively Coupled Plasma-Atomic Emission Spectroscopy |
| IEA | International Energy Agency |
| IGA | Intergranular Attack |
| IIW | International Institute of Welding |
| IMPACT | Community Programme: Internal Information Service Market |
| IP | Intelligent Processing |
| IR | Infra Red |
| ISE | Institute for System Engineering (JRC Ispra) |
| ISI | In-Service Inspection |
| ISO | International Organization for Standardization |
| JOULE | Joint Opportunities for Unconventional or Long-term Energy Supply (Community R&D Programme) |
| JRC | Joint Research Centre |
| KAKADU | Kamin Kasel-Duo (Twin capsules for fuel pin irradiation |
| KECU | Kilo European Currency Units |

| | |
|---|---|
| KFA | Kernforschungsanlage Jülich |
| KFK | Kernforschungsanlage Karlsruhe |
| LCF | Low Cycle Fatigue |
| LEFM | Linear Elastic Fracture Mechanics |
| LFR | Low Flux Reactor |
| LMFBR | Liquid Metal Fast Breeder Reactor |
| LOCA | Loss of Cooling Accident |
| LPPS | Low Pressure Plasma Spray |
| LT | Low Temperature |
| LWR | Light Water Reactor |
| MAN | Machienenfabriek Augsburg-Nürnberg |
| MCNP | Monte Carlo Neutron and Photon code |
| MECU | Million European Currency Units |
| MFE | Margin to Failure Evaluation |
| MI | Multilayer Interdiffusion |
| MOX | Mixed Oxide |
| MPA | Staatlich Materialprüfungsanstalt (Stuttgart) |
| MPI | Max-Planck-Institut |
| MPR | Materials Performance en Reliability |
| NDE | Non Destructive Evaluation |
| NDT | Non Destructive Testing |
| NEA | Nuclear Energy Agency |
| NEL | National Engineering Laboratory |
| NET | Next European Torus |
| Nf | Number of Cycles to failure |
| NILOC | Nitride Fuel, Low Oxygen and Carbon |
| NPL | National Physical Laboratory |
| ODS | Oxide Dispersion Strengthened |
| OECD | Organization for Economic Cooperation and Development |
| ORR | Oak Ridge Research |
| ORTM | Oak Ridge Test Matrix |
| PACVD | Plasma Assisted Chemical Vapour Deposition |
| PAS | Positron Annihilation Spectroscopy |
| PD | Potential Drop |
| PDT | Plasma Diffusion Treatments |
| PET | Positron Emission Tomography |
| PISC | Project for the Integrity of Steel Components |
| PIXE | Proton Induced X-Ray Emission |
| PNA | Proton Nuclear Activation Analysis |
| PNRS | Petten Neutron Radiography Services |
| POMPEI | Pellets Oxide Mixte, Petten Irradiation |
| PVD | Physical Vapor Depositions |
| PVSCC | Pressure Vessel Stress Corrosion Cracking |
| PWR | Pressurized Water Reactor |
| PWSCC | Primary Water Stress Corrosion Cracking |
| R&D | Research and Development |
| RBS | Rutherford Backscatting |
| RBSN | Reaction Bonded Silicon Nitride |
| REFLEXAFS | Extended X-ray Absorption Fine Structure in Reflexion Mode |
| RF | Radio Frequency |

| | |
|---|---|
| RLE | Residual Life Evaluation |
| RPV | Reactor Pressure Vessel |
| RRT | Round Robin Test |
| RT | Radiographic Techniques |
| RWE | Rheinisch Westphälische Elektrizitätswerke |
| S/T | Science and Technology |
| SAM | Scanning Anger Microscope |
| SAS | Standard Acoustic Source |
| SEM | Scanning Electron Microscopy |
| SIENA | Steel Irradiation in Enhanced Neutron Arrangement |
| SIF | Stress Intensity Factors |
| SIMS | Secondary Ion Mass Spectrometry |
| SMAW | Submerged Metal Arc Welding |
| SMC | Surface Modification Centre |
| SME | Small to Medium Enterprises |
| SOC | Sulphidizing/Oxidizing/Carburizing |
| SOXAFS | Surface Oriented X-ray Absorption Fine Structure |
| SS | Stainless Steel |
| STEP | Science and Technology for Environmental Protection |
| STEP | Standard for the Exchange of Product Data |
| SWSCC | Secondary Water Stress Corrosion Cracking |
| TC | Technical Committee |
| TEM | Transmission Electron Microscopy |
| TMF | Thermo-Mechanical Fatigue |
| TNO | Toegepast Natuurwetenschappelijk Onderzoek |
| UKAEA | UK Atomic Energy Authority |
| US | Ultrasonic |
| UT | Ultrasonic Techniques |
| UTS | Ultimate Tensile Strength |
| VAMAS | Versailles Agreement on Advanced Materials and Standards |
| VPS | Vacuum Plasma Spray |
| WG | Working Group |
| WP | Work Package |
| XANES | X-ray Absorption Near Edge Structures |
| XAS | X-ray Absorption Spectroscopy |
| XPS | X-ray Photo-emission Spectroscopy |
| XRD | X-ray Diffraction |
| XTEM | Cross-section Transmission Electron Microscopy |

# VI List of Authors

# List of Authors

**I INTRODUCTION:** E.D. Hondros (Director).

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- Operational Defects in Materials and Lifetime Predictions: H. Stamm, F. Lakestani, A.M. Morrissey, U. Von Estorff.
- Wear and Corrosion Resistant Coatings: W. Gissler, E. Lang, J. Haupt.
- Composite Materials Properties Improvements: G. Piatti, D. Boerman, F. Dos Santos Marques, B. Looman, M. Vedani.
- Chemical Sensors: L. Manes, G.B. Barbi, R. Feduzi, M.D. Giardina, A. Manara.
- Surface Treatments for Improved Performance: P. Schiller, F. Brossa, F. Coen, T.A. Crabb, M. Forte, O. Gautsch, F. Geiger, P.N. Gibson, A. Manara, E. McAlpin, R. Stroosnijder.
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- Standards and Codes for Non-Destructive Testing of Pressure Vessels and Weldments: E. Borloo.
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