ANNUAL·REPORT·90

Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

EUR 13801 EN

ANNUAL · REPORT · 90

Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

Published by the
COMMISSION OF THE EUROPEAN COMMUNITIES

Directorate-General
Telecommunications, Information Industries and Innovation

L-2920 LUXEMBOURG

Cataloguing data can be found at the end of this publication

Luxembourg: Office for Official Publications of the European Communities, 1991

Catalogue number: CD - NA - 13801 - EN - C

Printed in the Netherlands

Table of Contents

I Introduction

Introduction

Here we report on progress in the work of the Institute covering the third year of this present four year JRC Programme. In the previous report, we described the reorganisation of the Joint Research Centre into a set of Institutes, in which the present Institute was structured to embrace most of the materials R & D of the former Joint Research Centre, in particular that in Petten and in the Ispra sites. This bimodal structure confers the benefit of size and also of a wider range of expertise which can be exploited in a complementary manner. Thus, close coordination across the two geographical sites has continued with increasing R & D cooperation even at the individual project level.

The report emphasises essentially this continuing process of consolidation in research activities and in evolving a distinctive identity. We report on progress in research in the individual projects, in new initiatives with the European materials community as well as in the planning of an Institute strategy.

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:

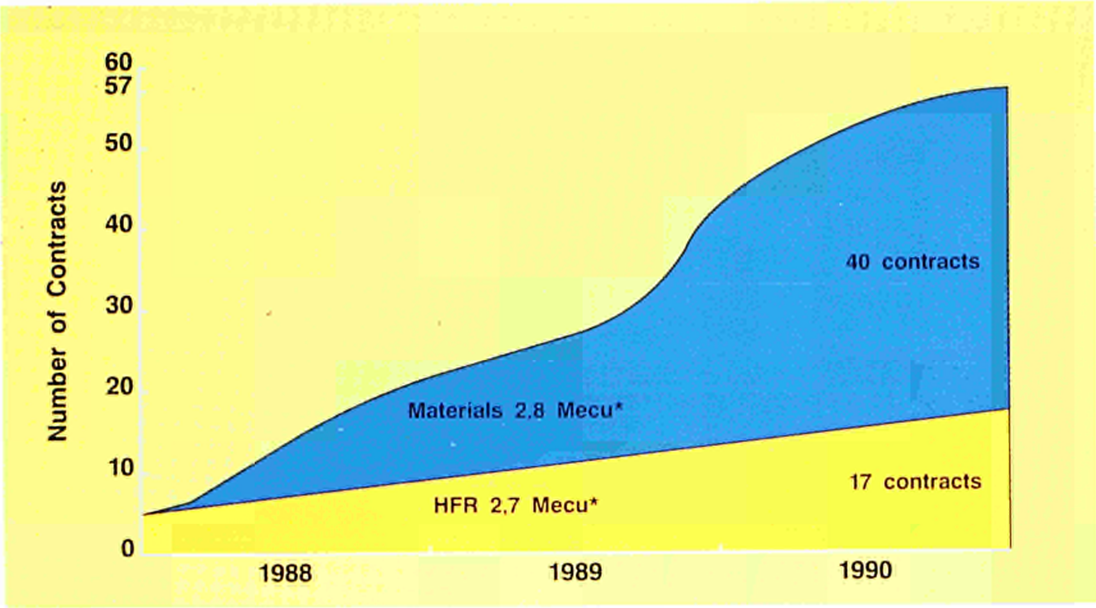
Standards for Advanced Ceramics (DG III)
- Support to and stimulation of the development of European standards and pre-standards
- Execution of R & D actions within European standardisation activities.

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

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

Materials Databanks (DGXIII)
Support to DGXIII (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.



Above: *Evolution of Third Party Contracts.*
** total earnings*

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.

- *Cold Fusion*
  To investigate the effect of deuteration on metal shavings.

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.

Third Party Contract Research

This is a new departure in the work of the Joint Research Centre. It is the most direct affirmation of the customer/contractor principle and represents a cultural adaptation in so far as the competences and facilities of the Institute are made available for research contracted by outside bodies. During this period, the Institute has progressed well in finding new contracts which reflect the range of interest of the Institute. The projects in general depend closely on the scientific experience gained in the execution of the Specific Programme. The evolution of this type of R & D is illustrated above.

Budget and Resources 1990

The table below lists the budget appropriations and the manpower resources allocated to each of the research areas during the year 1990.

Programme	Research Staff	Research Budget (Kecu)
1. Specific Programme		
Materials	82	2280
Fusion Materials	22	329
Reactor Safety (PISC)	22	495
Other Specific	5	70
2. Support to the Commission	9	630
3. Exploratory	8	384
4. High Flux Reactor		
Complementary	42	636
Common	3	330
Totals	193	5154

Notes **1.** In addition to the above resources, 8 research staff were engaged in contract work for third parties. The total revenue for contract research work performed in 1990 amounted to 2.300 Kecu.

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

European Materials Initiatives

The Institute has been engaged in setting up a number of materials related initiatives, in a pan-European context and with the aim of stimulating new forms of cooperation for an effective European presence in engineering materials. Progress has been made in the following initiatives:

European Initiative on Structural Intermetallics
In order to help create a wider awareness of the potential of intermetallic materials for future engineering applications, especially in high temperature situations, the Institute has initiated discussions and set up a Committee of European experts, the European Group for Structural Intermetallics (EGSI). Together with partners in DG XII from the BRITE/EURAM Programme, Institute staff have organised a special Workshop in Brussels, attended by more than 50 participants, to ascertain the degree of interest of research bodies in Member States of the EC to enter into a collaboration on intermetallics. Following this, the Committee has drawn up plans with a view to setting up together with BRITE/EURAM a Concerted Action on Intermetallics which will be launched in the course of 1991.

European Long Ceramic Fibres Initiative
In order to encourage the development and application of ceramic and metal matrix composites strengthened by continuous ceramic fibres and to create a favourable climate for European industry to manufacture such fibres, the Institute has catalysed a number of meetings of European experts in this field.
The general response and interest has been heartening, which has led to a number of actions.
In the first instance, together with colleagues in BRITE/EURAM, an expert marketing investigation will be carried out to determine the potential usage of fibres in future products, ranging from high temperature composite materials to, possibly, materials for the first wall of a thermo-nuclear reactor.
In addition, a EUREKA programme is being planned in order to conduct research by concerted action on routes for the efficient production of ceramic fibres and their properties characterisation.

The EMARC Contract Research Consortium
In order to provide a framework for pursuing and executing large industrial contracts on materials projects, the Institute together with CISE in Italy, has organised a Consortium of four leading European research organisations.

In addition to the above, this includes TNO in The Netherlands and National Engineering Laboratory (NEL) in the U.K. - the aim being to pool materials research capabilities and experience and to offer to carry out research contracts in this highly coordinated manner.

For this, a legal charter has been prepared and the means for conducting this type of activity has been agreed among all the parties. The intention is to provide a legal framework for conducting business within the system of a European Economic Interest Grouping.

Advanced Coating Centre

The Institute has ascertained that there is high growth potential in the individual application of protective coatings for the integrity of engineering components operating under extreme conditions of service in, for example, aerospace and petrochemical industries. The Institute here is planning to enter into a Joint Venture with a neighbouring Dutch Energy Research Foundation (ECN), to pool manpower resources, expertise, and facilities, in order to provide an Advanced Coating Service. The aim is to develop advanced coating techniques using processes such as chemical vapour deposition and plasma vapour deposition and to make available on a contract basis a central facility for research and innovation.

Institute Strategy for the year 2000

Together with the other Institutes of the Joint Research Centre, this Institute has entered into extensive studies and discussions with the aim of setting up a strategy up to the year 2000. The Institute has undertaken a lengthy internal study aimed at defining the research fields in which it can best contribute, given the likely materials associated requirements. Public and industrial sectors which on the basis of experience should have a high requirement for advanced materials related research include: energy; transport; manufacturing; environment; information technology; safety; construction; medical; R & D training.

The outcome of different individual studies on public and industrial sector materials R & D requirements were evaluated according to a set of criteria, namely; (a) requirement in terms of market pull and the perception of the future importance of the subject; (b) the customer aspect - institutional customers or industrial contract customers; and (c) the suitability of the work taking into account the advantages and constraints of the EC, such as the requirement of a high level of subsidiarity and the existence of available facilities and S & T expertise. All the studies were taken rigidly through the evaluation criteria and those retained were given high priority for future Institute activities.
Expressed in terms of materials themes, the following are considered to be the priority future orientations of the work of the Institute:

MATERIALS THEMATICS APPROACH

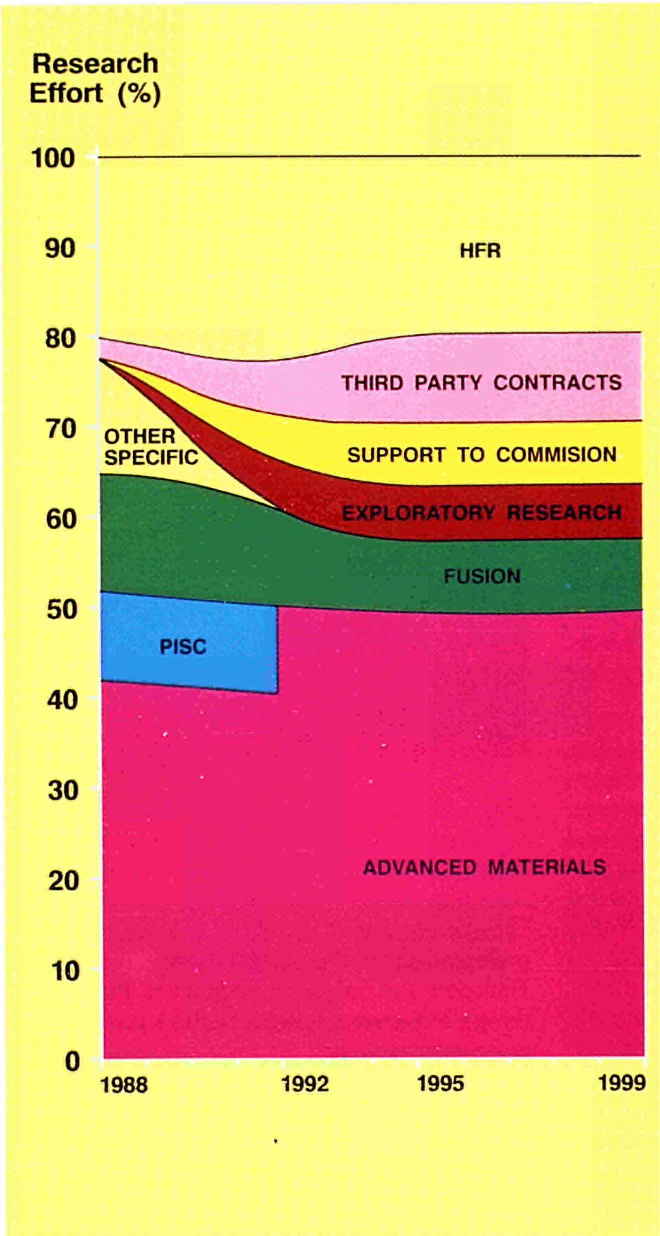
SYNTHESIS	- sub-micron structured materials innovations - ceramics coating innovations - composite fibre engineering - joining of dissimilar materials
CHARACTERISATION	- mechanisms of behaviour - life enhancement technologies - life prediction and reliability
PROCESSING	- computer aided intelligent processing, - sub-micron processing - quality control during fabrication - protection technologies
TESTING/VALIDATION	- NDE and NDT, for conventional and new materials - pre-normative R&D leading to codes, standards, reference materials - techniques for testing new materials and in complex operational conditions
MANUFACTURING	- computer integrated manufacturing (materials aspects)
INDUSTRIAL TRANSFER	- dissemination - specialist training.

The analysis shows that the R & D will contribute preferentially to the sectors: Power Engineering, Transport and Safety. In addition to these areas, there are themes suggested by the presence of unique central facilities:

- Boron Neutron Capture Therapy, based on the HFR

- Positron Emission Tomography, based on the Cyclotron.

Overall, this will require a gradual shift in the balance of the work throughout the Institute and in the type of materials R & D being conducted, with a growth in aspects of materials synthesis, materials processing (in particular through interfacial engineering), and test methodologies, in particular non-destructive testing. (See figure below).



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.

Much of the scientific activity of the project has continued to be carried out in cooperation with industrial and research institutes throughout Europe, particularly in the frame of COST 501 Round II but including also Joule, BRITE-I and BCR. In addition to the scientific involvement in three of the Work Packages of COST 501, two scientists (from component and fatigue areas) have the responsibility to manage WP5 which involves the coordination of the testing and modelling of over 30 European research projects aimed at the assessment of the (residual) lifetime of specific power engineering components under simulated service conditions.

The test and assessment methodologies activity continued to feature as a major effort since, in addition to the original objective of improving the techniques required for the scientific activities, it is increasingly required for the tailoring or development of systems to be used at a later stage in third party contracts. The project activities are described in terms of the principal type of degredation involved.

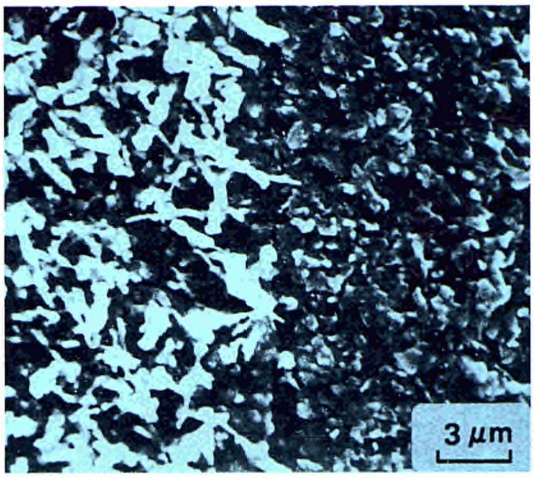
a. Corrosion

In comparison with the previous year a greater proportion of the Group's efforts have concentrated on research contracts conducted on a payment basis for third parties. This trend is illustrated by the increasing number of joint papers.

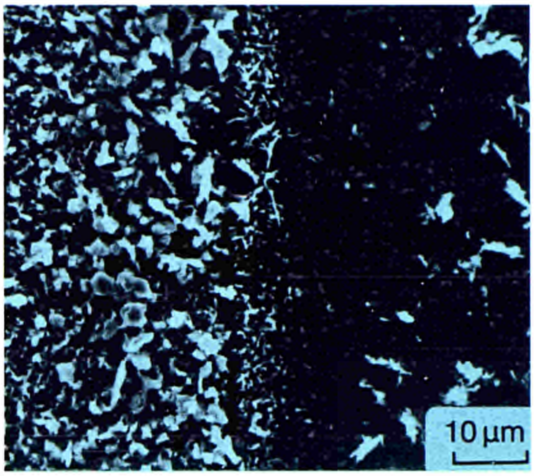
The major objective of the study within the COST 501/II, WP4, framework is to relate the corrosion behaviour established under carefully controlled laboratory conditions with those observed in commercial gasification plant for 4 alloys, i.e. AISI 310, Alloy 800H, Fecralloy, and MA 956. After first characterising the atmosphere of a pilot coal gasifier operated by one of the collaborating partners (British Coal), specimens of each alloy were exposed in the plant in various temperature regions and also in laboratory autoclaves in gaseous environments covering the range found in the gasifier. Specimens extracted from the top (~870^{0}C) and middle (~690^{0}C) regions of the gasifier were examined using X-Ray diffraction. The corrosion products which had formed during the 940h. exposure were mainly oxides, e.g. M_3O_4, Fe_2O_3, Al_2O_3 and Cr_2O_3 (see table below).

Tabel below: *X-Ray Diffraction Analyses of Specimens Exposed in British Coal Gasifier for 940 hours*

Material	Temp. (^{0}C)	Surface Condition	Phases Identified (Intensity/Parameter) γ	α-CrFe	M_3O_4	Cr_2O_3	Fe_2O_3	Al_2O_3	Others
AISI 310		Ground	224/3.58	-	19/8.38	5/4.79-13.62	-	-	-
		Electropolished	25/3.59	-	45/8.34	-	36/5.03-13.78	-	-
Alloy 800H		Ground	266/3.59	-	21/8.39	4/4.97-13.61	-	-	-
		Electropolished	-	-	42/8.32	-	40/5.03-13.74	-	NiS(15)
Fecralloy	690	Ground	-	315/2.88	-	-	-	-	-
		Electropolished	-	515/2.89-	-	-	-	-	-
MA 956		Ground	-	494/2.88	-	-	-	-	-
		Electropolished	-	23/2.88	11/8.38	-	-	-	-
1 x			-	35/2.89	21/8.34	-	16/5.03-13.72	9/4.75-13.02	-
2 x			-	-	35/8.34	-	16/5.03-13.72	-	-
3 x			-	-	29/8.39	-	12/5.03-13.76	-	-
4 x	870		-	-	32/8.37	-	20/5.03-17.75	-	-
5 x			37/3.59	-	39/8.36	-	25/5.04-13.74	-	-
6 x			13/3.58	-	37/8.41	-	13/5.04-13.76	-	-
MA 956		Ground	-	56/2.88	13/8.33	-	12/5.03-13.74	5/4.76-13.00	-
		Electropolished	-	35/2.88	42/8.34	-	36/5.03-13.73	22/4.76-13.03	-



A



B

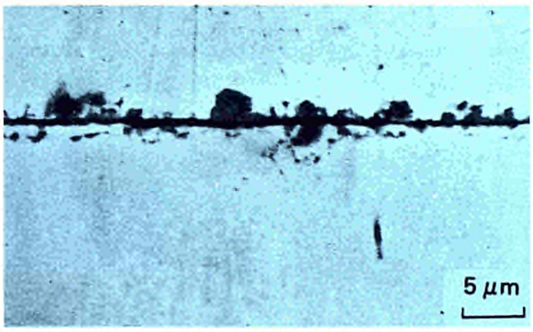
Samples which were exposed in the laboratory for 1000h at 600^{0}C to H_2-CO-H_2O-H_2S gas mixtures containing 0.6% and 2% H_2S were covered by sulphides. The MA 956 and Fecralloy specimens appeared more corrosion- resistant in the simulated atmospheres due to the initial formation of thin Cr-rich sulphides.

In a separate investigation, Alloy 800H surfaces were exposed to simulated coal gasification atmospheres.
The aim was to determine the effect of Ce ion implantation on corrosion resistance.
Less corrosion was found on surfaces implanted with 10E17 ions/cm^2 than on untreated surfaces: the scale was thinner, the development of isolated fast-growing sulphides was retarded, and the occurrence of localized internal corrosion products was considerably delayed.

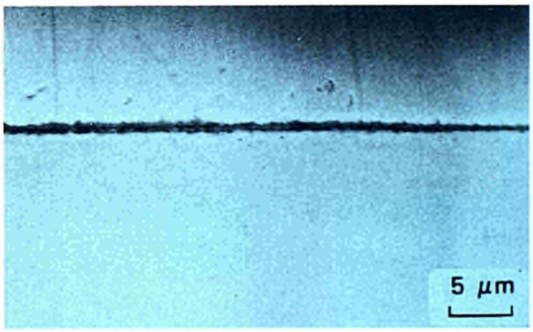
In addition, the corrosion products on the implanted alloy had a higher Cr:Fe ratio than on unimplanted surfaces. Figure above shows the difference in morphology of the scale on a region of the alloy surface implanted with Ce compared with a region outside the implanted area. Figure below shows how such surfaces appear in cross-section.

Above: *Surface-scale morphology near the boundary of unimplanted (left) and 10^{17} Ce ions/cm^2 implanted (right) Alloy 800H after exposure for 15 min (a) and 5 hr (b) in the S-O-C gas at 700^{0}C.*

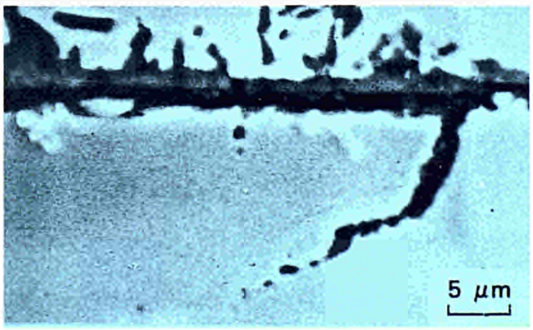
Below: *Cross-sectional SEM micrographs of Alloy 800H (a,b,) and Alloy 800H implanted with 10^{17} Ce ions/cm^2 (c,d,) after exposure for 5 hr (a,c) and 200 hr (b,d) in the S-O-C- gas at 700^{0}C.*



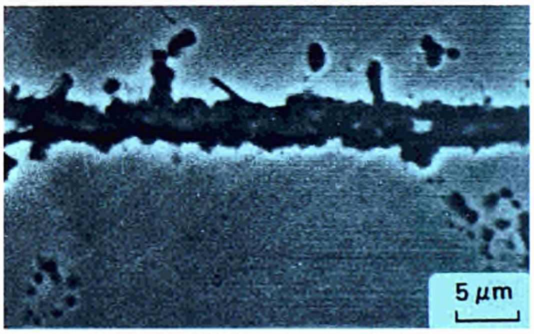
A



C



B



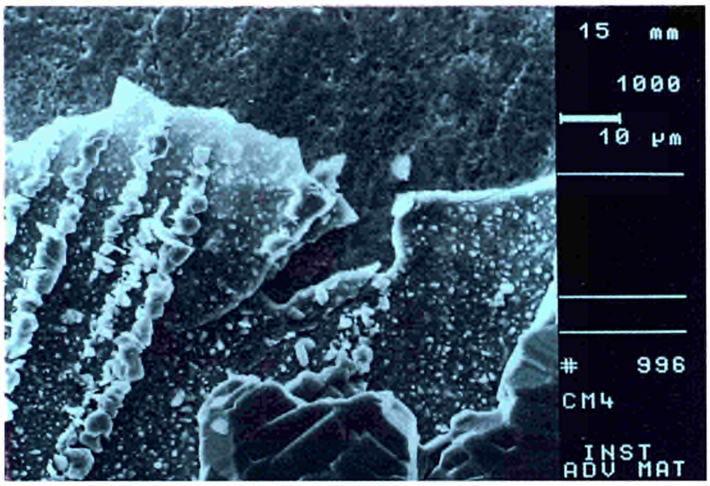
D

b. Creep

Most of the effort available for the Alloys project has been devoted to collaborative work within COST 501/II where Work Packages 1 and 4 are concerned respectively with heat exchanger and advanced gas turbine alloys.
Heat exchanger alloys: Experiments on the creep/corrosion behaviour of the iron-base ODS material MA 956 have been started.

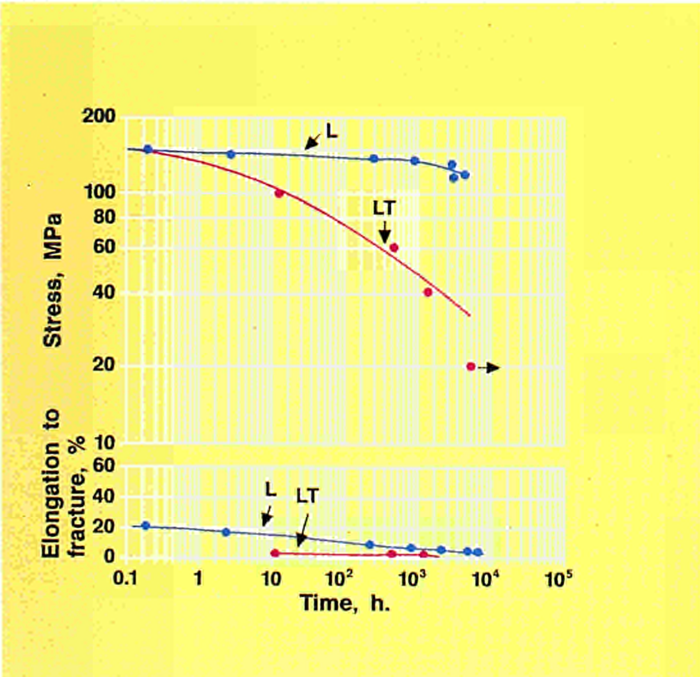
The main interest concerns the behaviour of pre-oxidized samples compared to untreated material. In order to improve the corrosion resistance of MA 956, the supplier has proposed a pre-oxidation treatment. However, the extent to which this preformed oxide layer (Al_2O_3 scale) can withstand the mechanical deformation which must be expected in service is not yet known. As shown by figure above, material degradation occurs in an H_2S bearing atmosphere for creep tested samples. The sample surface can be seen to be characterized by striations of small particles of corrosion product which were identified as Cr-rich sulphides.
Larger amounts of corrosion product, typically Fe-rich particles, which were found to be rather homogeneously distributed on the surface, overgrow the smaller Cr-sulphides. The striation-like geometry of the attack obviously marks places of fissure and crack formation in the oxide scale. Severe spallation of the scale could also be observed at various places as illustrated by figure above. Further investigations have been started to analyse the influence of the strain value and the strain rate on oxide scale deterioration for MA 956.

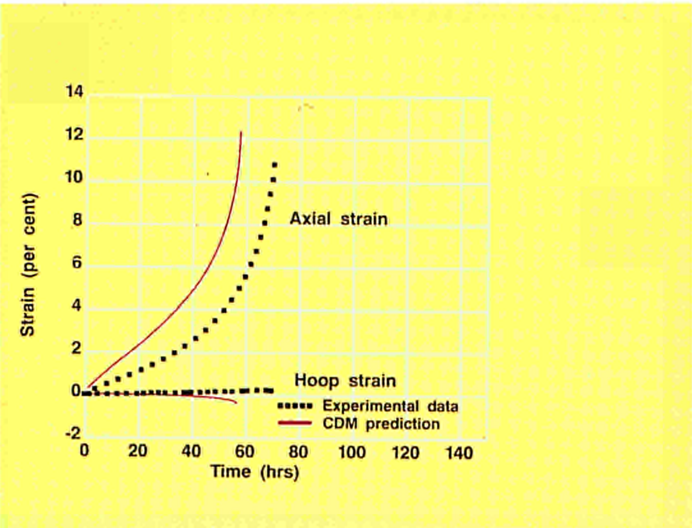
Gas turbine vane alloys: Creep investigations have been performed on the newly developed nickel-base ODS alloy MA 760 have been performed at 1050⁰C in air and tests up to about 6000 h have been finished. Some results on this topic are presented in figure below. As would be expected the main problem arises from the low strength and ductility if the samples are taken transversely to the orientation of elongated grains. Preliminary metallographic examinations have revealed that the creep samples exhibit severe grain boundary damage after only a few hours. Further work will be concentrated on structural analysis in order to characterize more completely the relationship between mechanical properties and microstructure.



Above: *MA 956 after 2.4% strain in an S-O-C bearing atmosphere at 600⁰C. Corrosion products on the oxide which has partially spalled.*

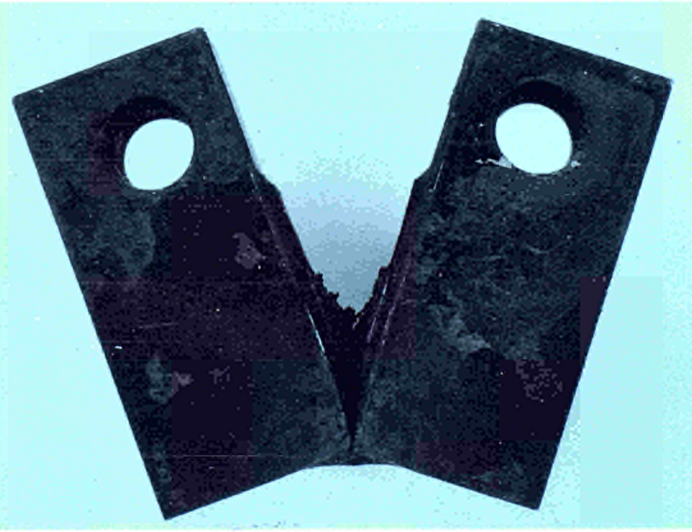
Below: *Creep test results for MA 760 at 1050⁰C.*
L = longitudinal direction, LT = long transverse direction.
Large deterioration for the LT-type samples.





Above: *Comparison of Biaxial creep deformation behaviour with theoretical predictions based on the CDM method*

Below: *2¼CR1MO minature compact tension specimen (Post test)*



c. Component behaviour

The purpose of the final phase of the BRITE-1 programme was to carry out specific benchmark tests by loading tubular components under multiaxial creep loads to verify predictions made from uniaxial creep data based on empirical and continuum damage mechanic models. The multiaxial creep behaviour of Alloy 800H tubes tested under internal pressure was found to be predictable using empirical θ-projection method adopted for multiaxial conditions. In contrast, creep deformation of 2¼Cr1Mo steels tested under internal pressure and axial loading met the predictions of a physically based continuum damage model as shown for example in figure above for a biaxial test.

The activities on 2¼Cr1Mo steel within COST 501-II WP5 continued in the multiaxial creep area, resulting in an important further development of the component creep crack growth method for the same alloy. Longitudinally and circumferentially notched tubes have been tested under internal pressure or axial load and the influence of notch geometry on creep strength and crack growth rate was elucidated.

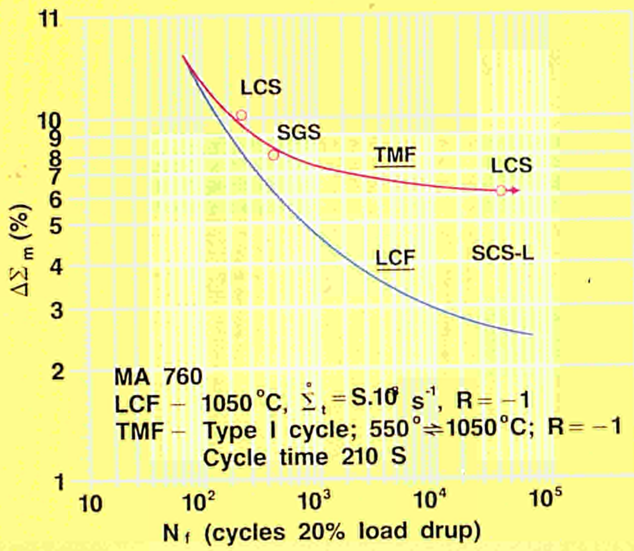
Two new activities were begun. First, in preparation for a study of the creep crack growth behaviour of ferritic steels in high pressure hydrogen environments some preliminary creep crack growth experiments have been successfully made using miniature compact tension specimens (figure below). In parallel, a facility for uniaxial creep testing in 200 bar H_2 environments was developed in connection with a third party contract. This facility will enable the rapid transfer of the CCG experiments to the conditions of potential hydrogen attack in various industrial processes.

The second new activity is a study of the multiaxial creep behaviour of oxide dispersion strengthened tubes for heat exchanger applications above 1000^0C. This study is being made in cooperation with partners within COST 501-II WP4. Preliminary experiments have shown that the hoop strength of this anisotropic material is at least a factor of five lower than the manufacturer's data for longitudinal strength and further investigations are required to establish the importance of such results for plant design.

d. Fatigue

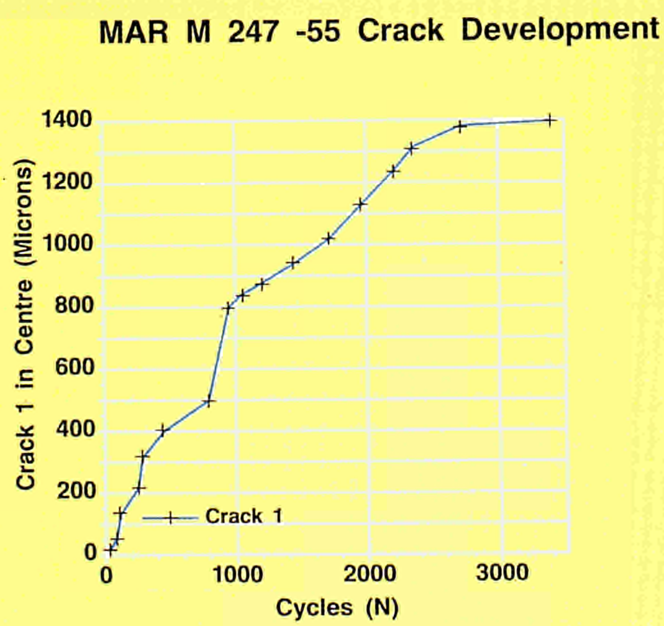
Experimental work in the COST 501-II programme is geared towards generating a data base on the nickel-base ODS alloy MA 760, in collaboration with outside laboratories participating in the work packages WP1 and WP5. The companion objective is the identification and analysis of the damage accumulation and failure mechanisms. Figure above compares preliminary results of the low cycle fatigue lives at 1050°C with thermomechanical fatigue lives using a 550°C-1050°C TMF cycle defined on the basis of the temperature/strain history in a gas turbine vane.

The development 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 deformed continued.
Several hardware extensions were made in order to achieve a separate handling of the command files and of the image and data files. Software updates concerned the implementation of the scan image acquisition mode and improvements of the analytical tools.
The system's capability was tested by the in-situ measurement of the growth of microcracks during the fatigue testing of a nickel-based alloy at high temperature. An example of a microcrack growth curve is shown in the figure below.
A major task with regard to equipment and test technique updating is the implementation of a digital data acquisition, analysis and presentation system. Decentralized data acquisition (DAQ) was selected as best suiting the group's testing environment. To promote data mobility all DAQ stations are interconnected to form a distributed file server communication network. The implementation of the DAQ concept started in 1990 in the E.T.L./S fatigue laboratory.
The development of the Expert System ARTIC for the assessment and management of the residual lifetime of steam headers in collaboration with ISE (JRC Ispra) continued with the definition of Phase II aimed at turning the prototype of ARTIC into a workable system. However, the activity in this area had to be temporarily stopped in the middle of 1990 because of lack of manpower.



Above: *Low cycle fatigue and thermomechanical fatigue lives of MA 760*

Below: *Growth curve of a microcrack on the surface of a fatigue specimen measured in-situ during the test by means of the computer vision system*



Engineering Ceramics

In recent years, 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 (up to 1600°C). 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.

Highlights of the year have been:

- Subcritical crack growth of monolithic and SiC-whisker reinforced silicon nitride: SiC-whiskers lead to a slight increase in fracture toughness over the temperature range 20°C-1400°C; the crack growth mechanism is not changed by the presence of the whiskers.
- The high temperature mechanical properties of monolithic silicon nitride were evaluated in aggressive environments.
- Test equipment for testing the mechanical behaviour in tension and in creep of a 2D continuous fibre reinforced, ceramic matrix composite has been developed and an evaluation of test methodologies for studying the mechanical properties of ceramic composites was started.
- Progress in mechanistic studies of gaseous corrosion of engineering ceramics was marked by confirmation of the volatility map model for oxi-sulphidation developed from earlier experimental and thermodynamic data.
- The reliability of Si_3N_4-Si_3N_4 joints with high temperature strength (400-500 MPa) has been significantly improved by using metallic NiCr interlayers. Tailoring of interlayer chemistry leads to optimum properties.
- Reaction pattern studies for the formation of strong reliable bonds show that the formation of Cr_2N and CrN species leading to stable silicide formation gives strong bonding. Atmospheric control to minimise Si_3N_4 decomposition is extremely important.
- Implantation of ceramic surfaces with Ar, Cr, Si of N ions to modify both microstructure and microchemistry can improve the surface and bulk mechanical properties of silicon nitride.
- The fabrication of a fully dense SiC (whisker) - Si_3N_4 composite containing up to 20% whiskers by low cost slip casting/low pressure sintering shows mechanical strength and toughness equivalent to high cost hot pressed material.
- The increase in whisker size from diameter 0.2-0.3 μm to 1-2 μm gives an increase in toughening.
- The addition of CeO_2 as a grain boundary additive phase greatly enhances the sinterability of Si_3N_4-ZrO_2 composites.
- A methodology for measuring residual stress profiles in machined ceramic materials by X-ray diffraction techniques has been developed.

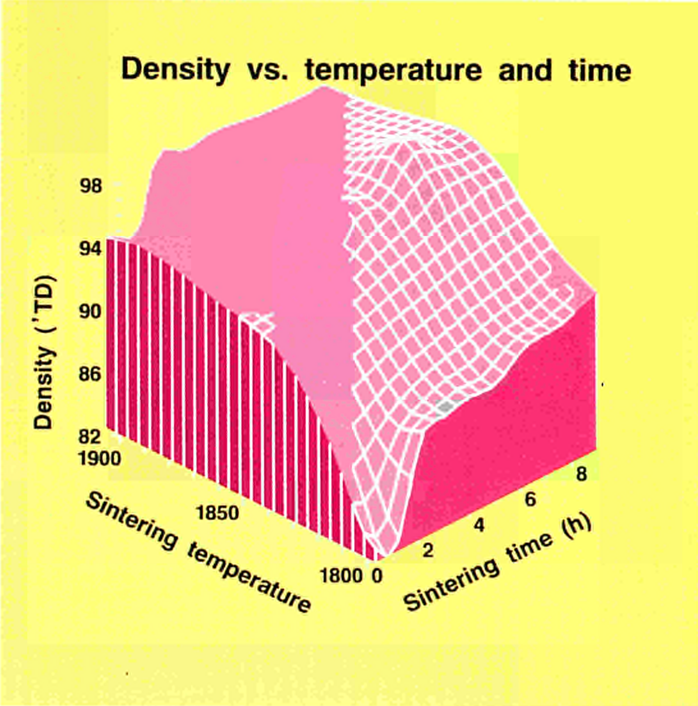
Research on mechanical properties has been focussed on the cyclic fatigue behaviour of Si_3N_4 and the subcritical crack growth of whisker reinforced ceramics in order to generate cyclic fatigue life curves and to elucidate toughening mechanisms of the reinforcement.

The gaseous and hot corrosion behaviour of a range of newly developed monoxide ceramics is being studied, to elucidate the fundamental corrosion mechanisms at very high temperatures.

Much progress has been made in understanding the relation between high temperature mechanical properties and ceramics machining techniques. Techniques have been developed for the preparation of reliable ceramic composite test specimens.

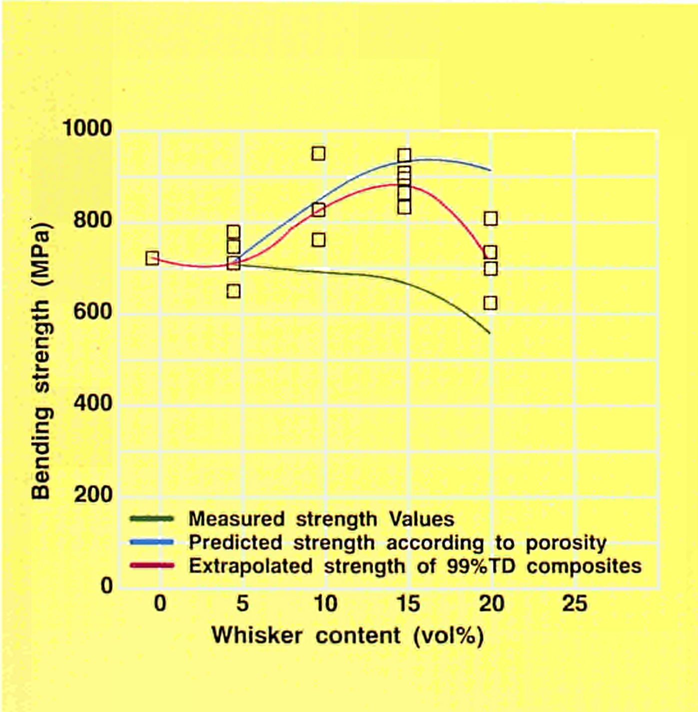
High quality ceramic joints have been developed showing good performance under conditions of high temperature corrosion and mechanical stress, which simulate industrial environments.

A state of the art study has been made in the field of non destructive testing for ceramics and ceramics composites.



Above: *Densification profile for a 10% SiC(w)-Si_3N_4 composite sintered under 10 bar N_2 gas*

Below: *Room temperature bending strength of a SiC(w)-Si_3N_4 composite*



The research activities are well integrated in the CEC-ceramics programmes 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,
- Mechanical Properties,
- Corrosion Properties.

Interfacial Engineering

a. Ceramic-Matrix Composites

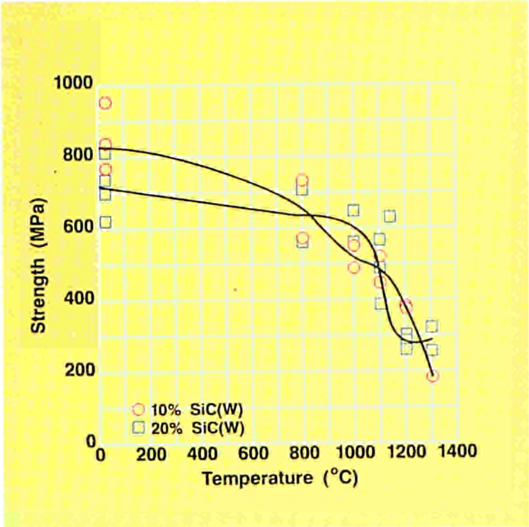
Silicon nitride reinforced with silicon carbide whiskers

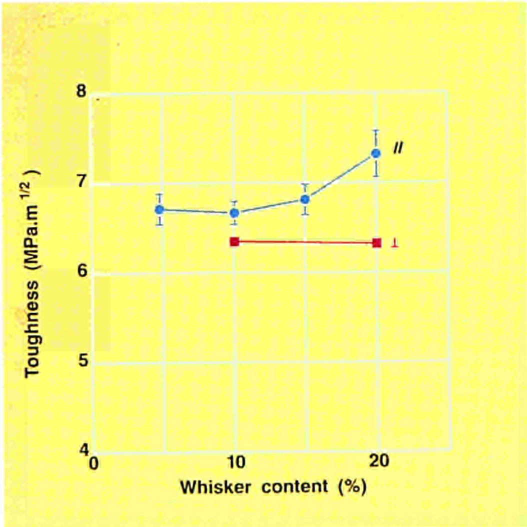
The reinforcement of monolithic ceramics by fibres or whiskers can lead to a significant improvement of mechanical properties such as strength, fracture toughness, strain at failure, susceptibility to surface damage and thermal shock resistance.

A processing route to fabricate SiC(w) - Si_3N_4 whisker composites by low-cost slip casting and low pressure sintering has been refined to allow optimum densification of composites containing 5, 10, 15 and 20% whiskers, homogeneously distributed through the matrix (Figure above).

The whiskers confer a significant increase in room temperature strength, up to 15% whisker content (Figure below).

At higher whisker content, an increase in matrix porosity arising from densification constraints imposed by the high whisker population, begins to exert a deleterious effect.





Above left: *Bending strength of SiC(w)-Si_3N_4 composites with increasing temperature*

Above right: *Toughness of a SiC(w)-Si_3N_4 composite*

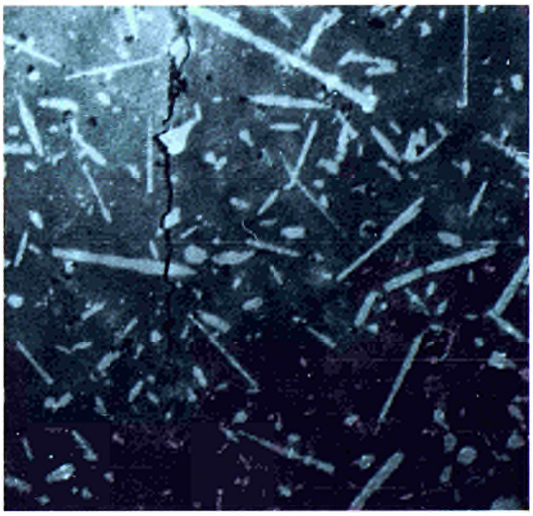
Below: *Room temperature fracture behaviour of a 10% SiC(w)-Si_3N_4 composite*

The composite strength falls off at higher temperatures as expected for high additive content Si_3N_4 based systems (Figure above left). Enhancement of strength at high temperature by control of intergranular chemistry is a clear priority for future research.

The enhancement of toughness in directions parallel to the slip casting direction was confirmed (Figure above right). Microstructural studies suggest that the composite toughness results from a whisker-induced decrease in grain size in the matrix, and by whisker toughening. Increasing whisker dimensions (from mean diameter 0.6 µm to mean diameter 2.2 µm) results in a minor improvement of toughness.

However, the enhancement of toughness has not been remarkable. Studies for crack propagation using real time video show that whiskers are fractured by the approaching crack with only a minimal degree of interfacial crack deflection (Figure below). The fibre matrix bond is clearly too strong to allow whisker debonding and the operation of the various mechanisms recognised to dissipate cracking energy (fibre pull-out, crack deflection, crack branching, bridging etc.).

The first phase of the project to develop an adequate processing route has been successfully completed; and the way has been opened to progress to the main project targets, i.e. to engineer the



matrix-whisker interface chemistry and structure in order to allow the toughening mechanisms to operate and develop a tougher composite. However, owing to the intrinsic toxicity of the SiC whiskers and the concomitant climate of industrial uncertainty regarding their future application - the project was considered as strategically too sensitive for the JRC and was terminated in June 1990.

Silicon nitride reinforced by zirconia particles

The stress induced transformation (tetragonal to monoclinic) of zirconia has been widely used to toughen many ceramic matrices including silicon nitride. The combined influence of ZrO_2 and cerium oxide to promote sinterability and to toughen silicon nitride offers the possibility to form tougher ceramics by low cost sintering process routes. A project has been initiated to explore the effect of these additions, in controlled proportion, on the processibility and mechanical behaviour of sintered silicon nitride.

First results indicate that both additives have very significant influence on densification properties by low pressure sintering, in particular, the inclusion of 5% CeO_2 resulting in near theoretical densities, even in the ZrO_2-free ceramic (Figure above).

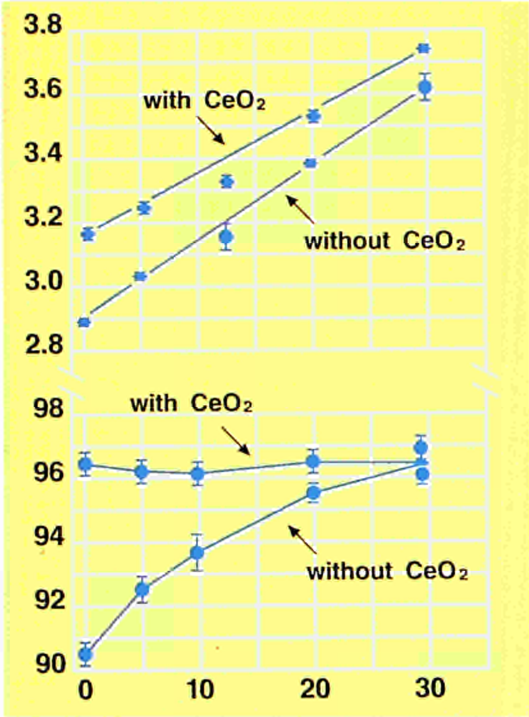
X-ray diffraction analysis (table below) suggests that the addition of CeO_2 strongly promotes the zirconia tetragonal to monoclinic transformation in contrast to published information, that CeO_2 should stabilize the ZrO_2 tetragonal form.

The project will proceed with the determination of the mechanical behaviour of the composites and the influence of composite chemistry and microstructure.

Residual Stress in Ceramic Surfaces.

The project builds on the foundation of earlier work to quantify machining damage in structural ceramics by X-ray diffraction measurement of surface residual stresses.

Switching from the usage of Cu-Kα radiation (penetration depth $\tau \approx 30$ µm) to the softer Cr-Kα ($\tau \approx 10$ µm) has revealed substantial machining damage and high stress gradients in the surface layers (< 10 µm). Independent research using a ψ-diffractometer (tilt perpendicular to the beam axis) gave



Above: *Bulk Density and Relative Density as a Function 3Y-ZRO$_2$ Content*

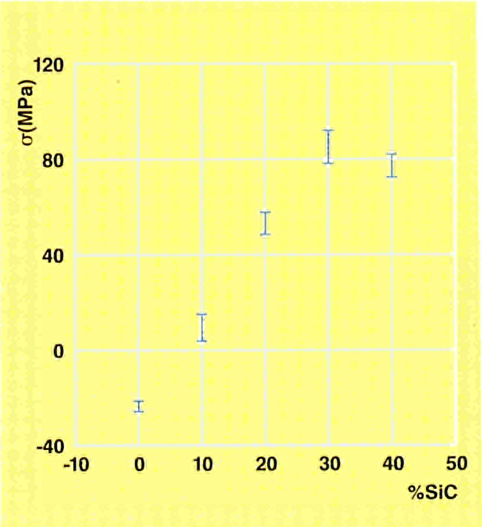
Table below: *The quantity of monoclinic phase zirconia as a proportion of:*
total zirconia content
total ceramic content

composition wt%				% Monoclinic to:	
Si_3N_4	Al_2O_3	CeO_2	ZO_2	total zirconia ratio %*	total content wt %*'
91	4	0	5	15	0.8
86	4	0	10	12	1.2
76	4	0	20	2	0.4
66	4	0	30	0	0.0
86	4	5	5	69	3.5
81	4	5	10	68	6.8
71	4	5	20	29	5.8
61	4	5	30	20	6.0

* : M(111) + M(111)/ M(111) + M(11$\bar{1}$) + T(111) + C(111) which are diffraction intensities of the monoclinic (111) + (11$\bar{1}$), tetragonal (111) and cubic (111) planes respectively.

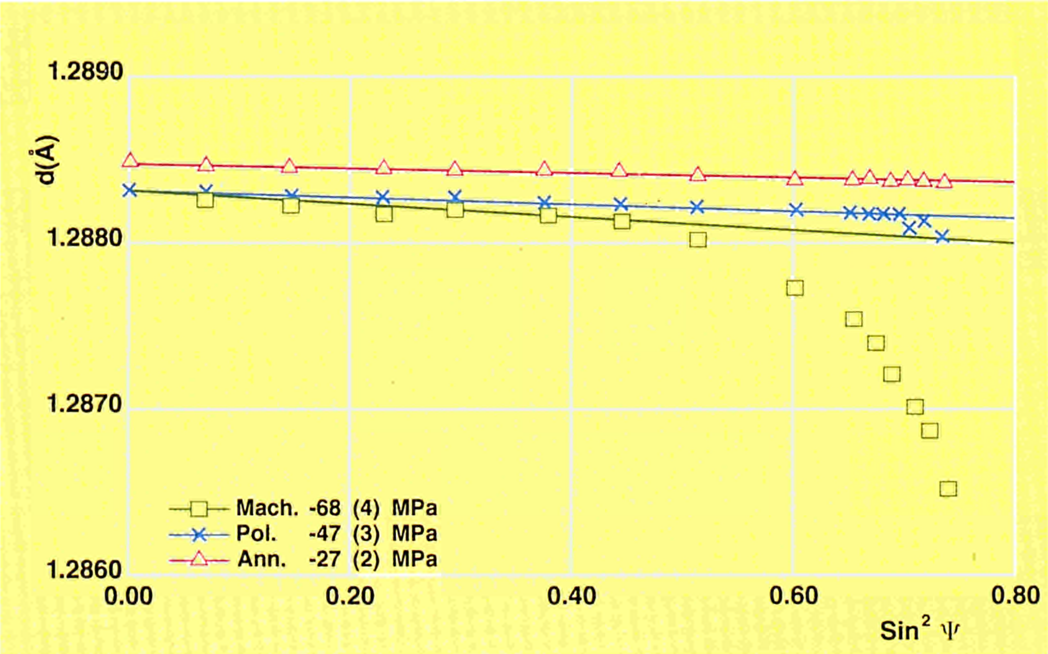
*' : Ratio x wt% 3Y-ZrO_2

much higher values than our diffractometer (tilt parallel to beam axis). Further research has revealed that the residual stresses and stress gradients reduce sharply after polishing and/or annealing of the specimens (figure below). The stress-depth functions, which are of the type σ = Kτn, were successfully calculated. More recent studies of interfacial stresses in Al_2O_3-SiC(whisker) composites show the progressive increase of stress with whisker content (figure above).



Above: *Stress versus percentage SiC in the Al_2O_3-matrix of the composites*

Below: *Residual stress profiles in silicon nitride surface layers. a) machined. b) machined and polished. c) annealed*



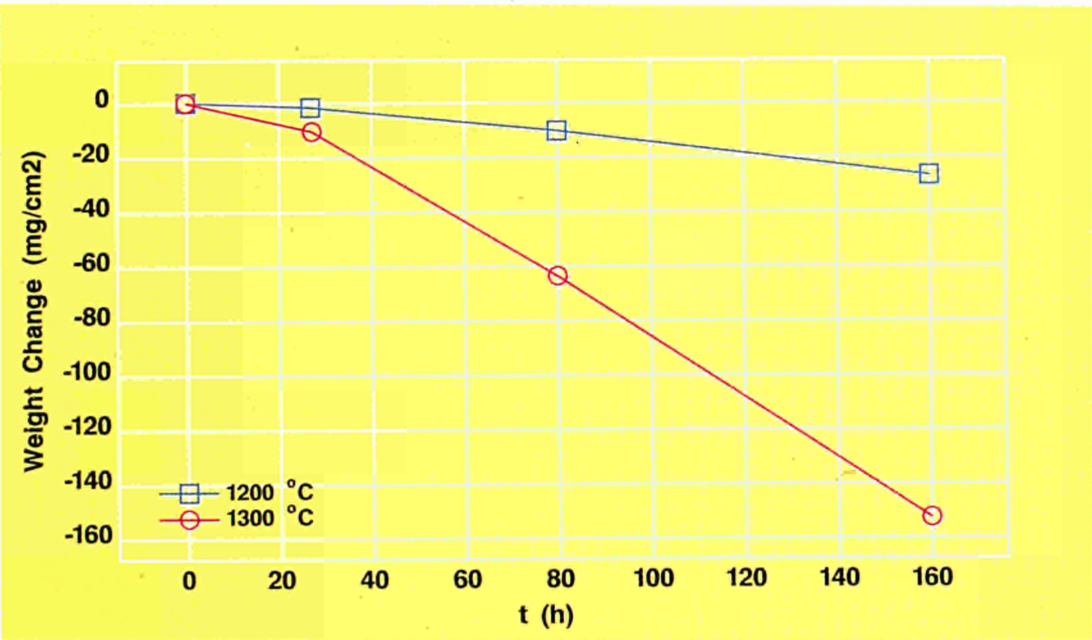
b. Ceramics Corrosion

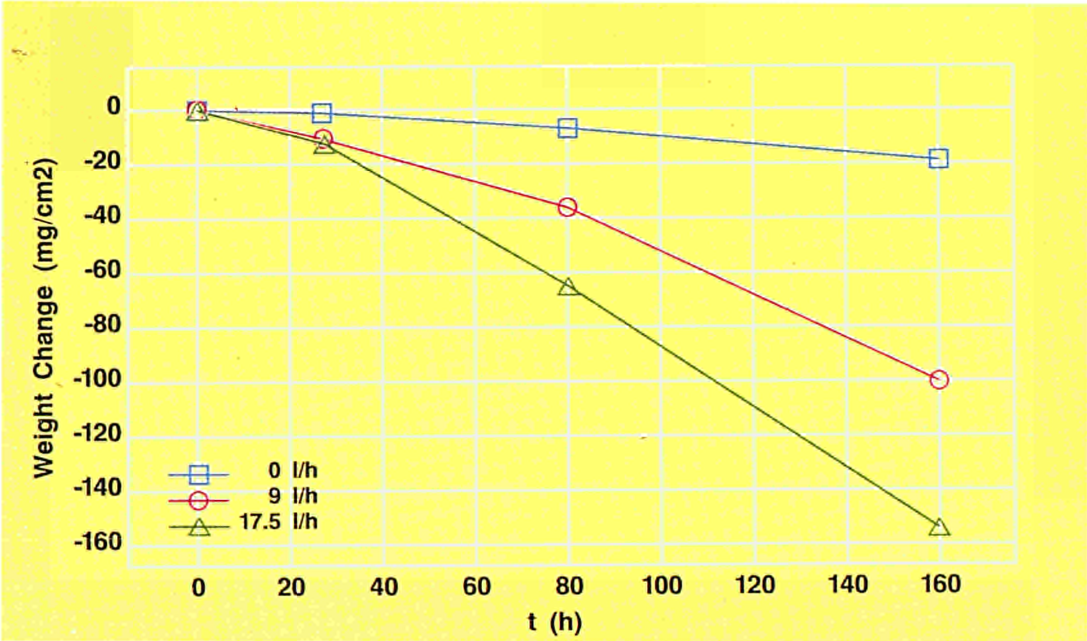
Activities in the field of high temperature corrosion of engineering ceramics have continued throughout 1990 in the direction established in previous years, namely towards an understanding of the mechanisms controlling corrosion processes of ceramics in environments of industrial importance. The activity is divided into aspects of screening commercially available ceramics for corrosion resistance in a given environment and more fundamental studies of the thermodynamic and kinetic mechanisms common to and underlying the behaviour of the materials.

Silicon nitrides still represent the major part of the activity which however is continuously evolving to include other engineering material types such as silicon carbides. In some instances, the latter show better corrosion resistance than nitrides, because there are usually lower levels of 'impurities' (residues of sintering additives in the form of secondary phases) which are inferior to the bulk material with respect to their high temperature properties. This reflects the relative ease of fabrication of dense silicon carbides compared to nitrides.

During 1990, research on silicon nitrides has concentrated on the understanding of the relative importance of oxidation and sulphidation under conditions of low oxygen potential where the thermodynamically controlling corrosion regime is active oxidation/sulphidation. Under these conditions, silica is thermodynamically unstable. Corrosion resistance in any reactive environment is usually dependent on the formation and stability of a dense protective layer which can act as an effective barrier to further reaction. When silica cannot form such a layer, corrosion proceeds via the formation of volatile silicon monoxide or sulphide with progressive loss of material. Under some conditions, the material becomes porous with a resulting increase in reactive surface area and a consequent acceleration of the rate of corrosion (see figure below).

Below: *Acceleration of corrosion rate with time for a HIPped silicon nitride at 1200° and 1300°C and constant gas flow rate in 0.1%H_2S-H_2*



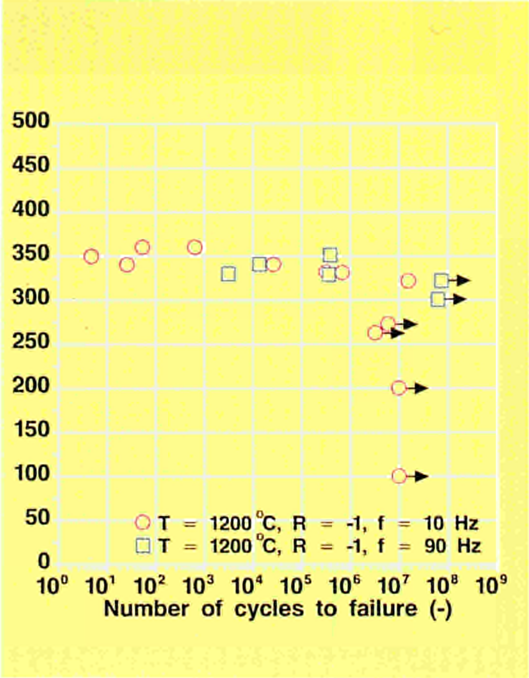


Above: *Effect of flow rate on extent of corrosion for a HIPped silicon nitride at 1300°C in 0.1%H_2S-H_2*

Other corrosion products which can form will be oxidation or sulphidation products of the secondary phases. They are rarely, if ever, structurally valuable and the resulting degradation of the material properties are both dramatic and catastrophic.

Work has concentrated on establishing the rate-controlling mechanism and on the measurement of corrosion rates from which meaningful predictions can be made. In relatively simple environments such as H_2S/H_2 containing only trace amounts of free oxygen or water vapour, the effect of the 'microenvironment' surrounding the specimen has assumed an importance which was unpredicted in its magnitude. The corrosion rate has been found to be dependent on the flow of active gas as well as the pressure, the proximity of other specimens, the geometry of the specimen and the means of presentation (freely suspended or placed in a crucible). An example is given in the figure above of the effect of flow rate on the rate of corrosion.

Towards the end of the year, a study was launched to research the behaviour and oxidation/corrosion properties of selected secondary phases commonly found in silicon nitrides densified with the aid of yttrium, cerium or zirconium oxides. After a literature study to establish the importance of the phases as well as to document the data already known about them, a selection will be especially prepared and studied in simple environments in isolation, i.e. in the absence of silicon nitride as primary bulk material. By this means, it is hoped to reach a better understanding of the role played by intergranular secondary phases in the corrosive degradation of silicon nitrides.



Above: *Fatigue lives of silicon nitride tested in the uniaxial mode, R = -1*

Below: *Weibull distribution of the 4-point bend strength of silicon nitride at different temperatures*

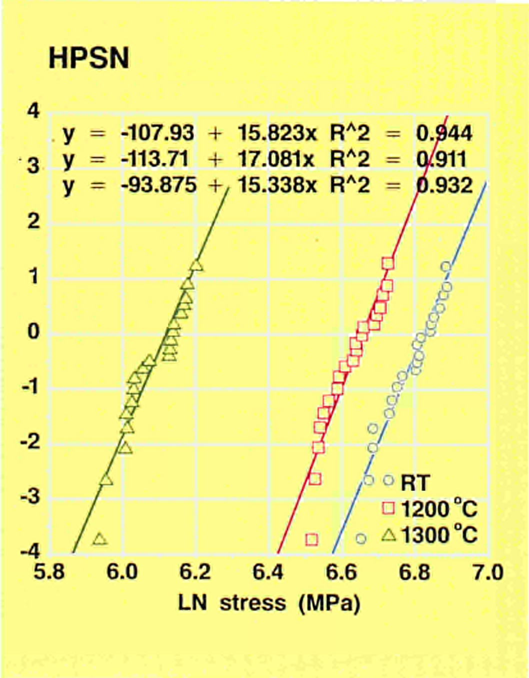
c. Mechanical Properties

The main objective of the research activity is the characterisation of the mechanical behaviour of silicon nitrides in terms of their properties, and of the mechanisms of deformation, of damage and of failure in relation to their microstructure. Because of their damage tolerant nature, continuous fibre ceramic matrix composites were included in the range of materials in 1990.

Four projects were covered in the reporting year. The project on the cyclic push-pull fatigue of HPSN at high temperature in the uniaxial testing mode was finalized.
It revealed the existence of an endurance limit at about ± 320 MPa for this particular HPSN material tested at 1200°C, R = -1 and frequencies of 10 Hz and 90 Hz, see figure above. The cyclic lifetime is observed to exceed the corresponding static life by a factor which increases with decreasing stress level. This could be explained by the beneficial effect of the viscous, intergranular glassy phase which retards crack growth because it acts as an extra energy dissipative medium and because it causes crack branching.

The project concerned with the subcritical crack growth of monolithic and SiC-whisker reinforced silicon nitride also ended in 1990. The presence of the SiC whiskers leads to a slight increase in fracture toughness over the temperature range 20°C-1400°C, which is attributed to crack deflection at the whiskers.
The path for subcritical crack growth is through the glassy grain boundary phase, and the crack growth mechanism is not changed by the presence of the whiskers.

A new project concerning the high-temperature mechanical properties of monolithic silicon nitride pre-exposed to aggressive environments started early 1990. It aims at investigating the effects of corrosive environments, typical of industrial gasification processes, on a range of mechanical properties. The testing material has been characterized microstructurally, from the point of view of surface roughness of the machined test samples, and the density of the samples has been determined. Prior to corrosive exposure, the Weibull distribution of the 4 point bend strength has been measured at temperatures up to 1300°C, figure below.



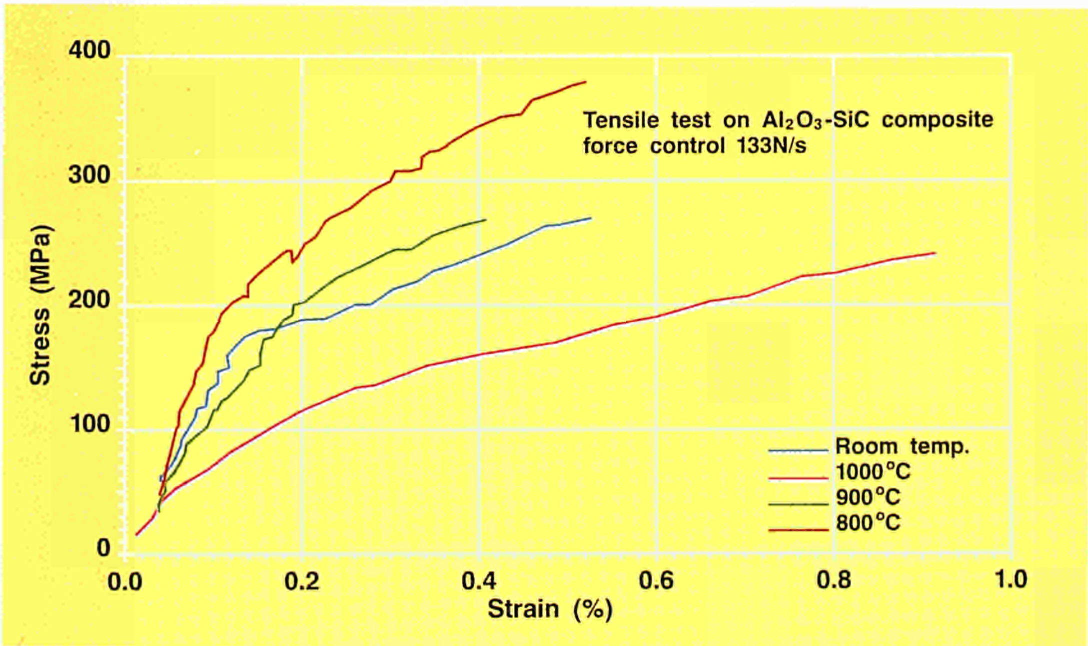
A fourth project deals with the mechanical behaviour in tension and in creep of a 2D continuous fibre reinforced, ceramic matrix composite. Testing under inert gas or vacuum is required in order to inhibit the masking of the material inherent properties by oxidation processes. Hence a high vacuum chamber was designed and installed on the mechanical testing machine, in combination with a strain measurement device; the furnace to heat the samples to the test temperature was built in-house.
The figure below shows a result of one of the first tensile tests performed on the composite material.

Several special purpose testing rigs were designed and, partly, installed and commissioned in the reporting period. Four creep/stress rupture rigs for the testing of ceramics up to temperatures of 1400°C and loads up to 2 kN were relocated to E.T.L./N and recommissioned for the 4-point bend testing in controlled corrosive environments.

The relocation of a 100 kN electromechanical testing machine to the E.T.L./N for the uniaxial mechanical testing of ceramics and metals in corrosive environments was initiated.

A 12 kW high frequency induction unit for heating of the samples was ordered, an environmental testing chamber to accommodate the corrosive gases was designed in-house and work to prepare the hardware for the hooking up of the testing machine to the corrosion gas lines of E.T.L./N was initiated.

Below: *Tensile test results on continuous 2D Al_2O_3-SiC composite at different temperatures*

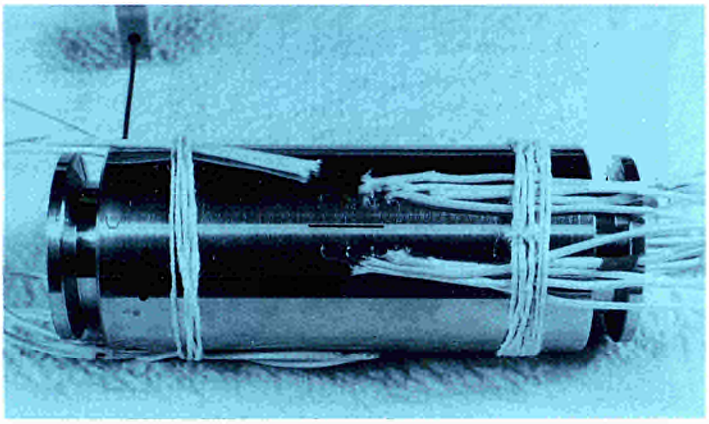


Components and Thermal Fatigue

The objective of this research area is the numerical modelling and the experimental verification of crack propagation in cyclic thermal gradient fields with a view to component life time prediction. The results of the study are relevant to industrial applications where thermal fatigue is the life limiting factor. The addition of an irradiation damage component aims at yielding results relevant to the design of the first wall of NET. The project progressed on all fronts in 1990.

The numerical analysis code, CRACK, was developed and implemented on a SUN 386 work station as a part of the thermal fatigue code CRKPRO. CRACK calculates stress intensity factors (SIF) in two and three-dimensional structures. It uses the weight function method for 2D cracks and the line spring model in combination with an empirical formula for 3D cracks. Hence, the means is now available to calculate numerically the LEFM life of a tube controlled by the growth of longitudinal or radial, semi-elliptical internal or external surface cracks. The other element required to calculate the life is the crack growth law for the material and the service conditions concerned. Fracture mechanics tests on centre-notched samples were used to determine this law. The results so far cover only a fairly small range of K. In order to widen this range CT specimen tests are scheduled.

The out-of-pile rig for the thermal cycle testing of tubes is now operational and it functions extremely well. Temperature gradients through the tube wall are measured during the imposed thermal cycle and these measurements compare well with the calculated values. Crack growth is monitored using the DC potential drop method.

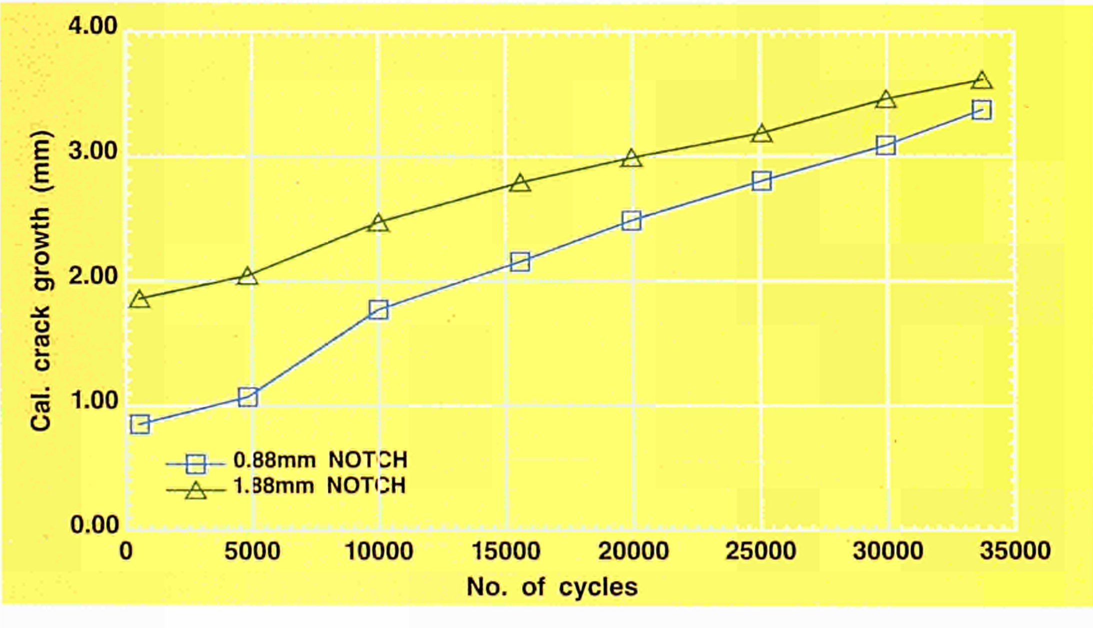


The figure above shows a test tube with a longitudinal external starter crack with the DC PD probes in place. Typical crack growth curves for internal longitudinal notches of different depths in the tube are plotted in figure below.

The preliminary design phase of the in-pile rig has been completed following the installation of the necessary hardware and of software to model the rig. A range of techniques for the measurement of crack growth has been experimentally assessed in terms of the effect of neutrons on their sensitivity.

Above: *Tube test geometry for thermal fatigue testing, showing notch and DCPB probes*

Below: *Crack growth from internal, longitudinal notches during thermal fatigue cycling (80°C ⇌ 350°C) of a tube*



Operational Defects in Materials and Lifetime Prediction

To predict the failure of components due to a deterioration of material properties, information is required on those microstructural defects that are responsible for the decrease in load bearing capacity. Improving the reliability of the assessment of the residual life of components requires more soundly based methods for quantifying the internal defects in the material.

Non-destructive techniques are of great interest for in-service inspection. The main efforts in 1990 were concentrated on validating non-destructive ultrasonic velocity (US) measurements by comparison with the conventional metallographic method for the detection of creep damage.

This was successfully achieved for a Mn-Cr austenitic steel in which creep damage is manifest by the formation of intergranular microcracks the planes of which were preferentially oriented perpendicular to the applied uniaxial load. This anisotropic damage is characterised by the value, q, which is the relative velocity difference of US-shear waves which are polarized perpendicular to one another and which propagate in the direction perpendicular to the applied stress.

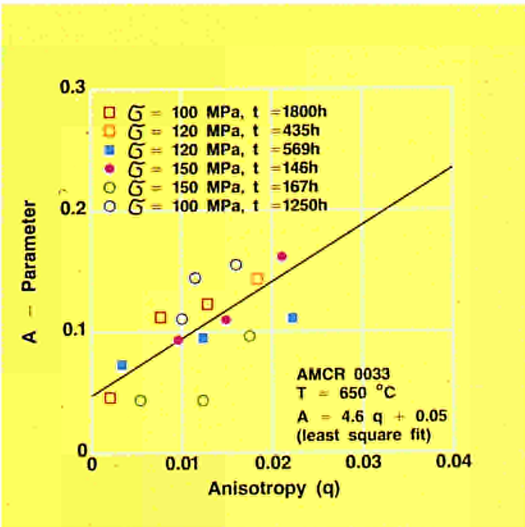
The metallographic findings were quantified by determining the "A"-parameter which is defined as the number fraction of cracked grain boundary facets intersecting a straight line parallel to the stress.

From theoretical considerations, it follows that both quantities A and q must be proportional to the same damage variable, NA^3, where N is the number of cracks per volume and "A" is the radius of a circular crack, which is taken to represent the average crack geometry.

As demonstrated in figure below, where A-values are plotted versus the corresponding q-results measured at the same place, the slope of the linear A-q fit coincides rather well with the theoretically expected value $A \approx 3.5 - 4$. The offset value for A is due to uncertainties in determining the A-parameter. For the alloy considered here, it can also be seen that the sensitivity of the non-destructive US-techniques can compete well with metallographic methods.

The investigations are being extended to other alloys, e.g. low-alloy ferritic steels which are examined in the framework of COST 501, WP5. It is the aim of this research project to establish conditions under which US-methods can be successfully applied, to relate them to other methods of damage determination, and to determine how they can be used together with constitutive laws for residual life assessment.

Below: *Plot of the metallographically determined A-parameter versus the relative velocity difference of orthogonally polarized shear-waves (q) (polarization direction parallel and perpendicular to the applied uniaxial stress)*



Wear and Corrosion Resistant Coatings

Introduction and Objectives

Lifetime and load capacity of tools and other construction components depend on both how the particular material reacts to wear as well as to 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 led (simultaneously) to prolonged lifetimes and to 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 to contribute towards an improvement of both 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 film deposition techniques available at the film laboratory of the IAM Ispra and the Advanced Coatings Centre of IAM Petten. Surface modification techniques such as ion implantation, ion beam mixing, chemical vapour deposition, plasma diffusion and laser treatment might successfully be used to modify and tailor the composition, structure, hardness and adhesion of protective surface coatings.

Results

1. TiN-coatings

TiN-coatings have been deposited by reactive ion beam sputtering onto two representative tool steels, C35 and TANTUNG G. Wear tests by using a pin-on-disc tribometer have revealed that i) the substrate wear was significantly reduced by the TiN-coating but ii) the wear of the counterpart of the sliding couple was increased due to the high surface roughness of the coatings.

2. Boron Nitride Films

BN films were deposited by magnetron sputtering under various substrate temperatures and bias voltages. Characterization was performed by SEM, TEM, FTIR, XRD and with an ultra low load, depth-sensing nano-indenter. By comparison with computer calculations, based on models of various structure, grain size, shape and orientation, the best agreement was obtained assuming a mixed phase model of hexagonal (graphitic) and cubic BN with grains consisting of 50 - 1000 atoms. Structure and grain size were sensitive to bias voltage and deposition temperature. The hardness correlates with the relative amount of cubic and hexagonal structured grains in the BN film.

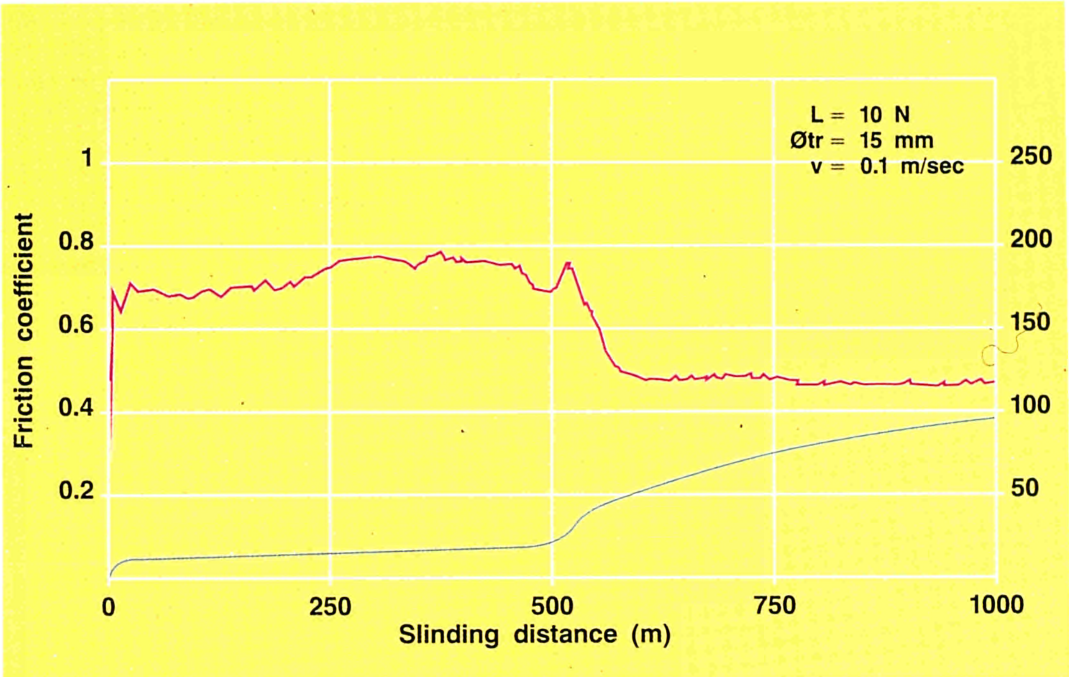
3. Ti-B-N Films

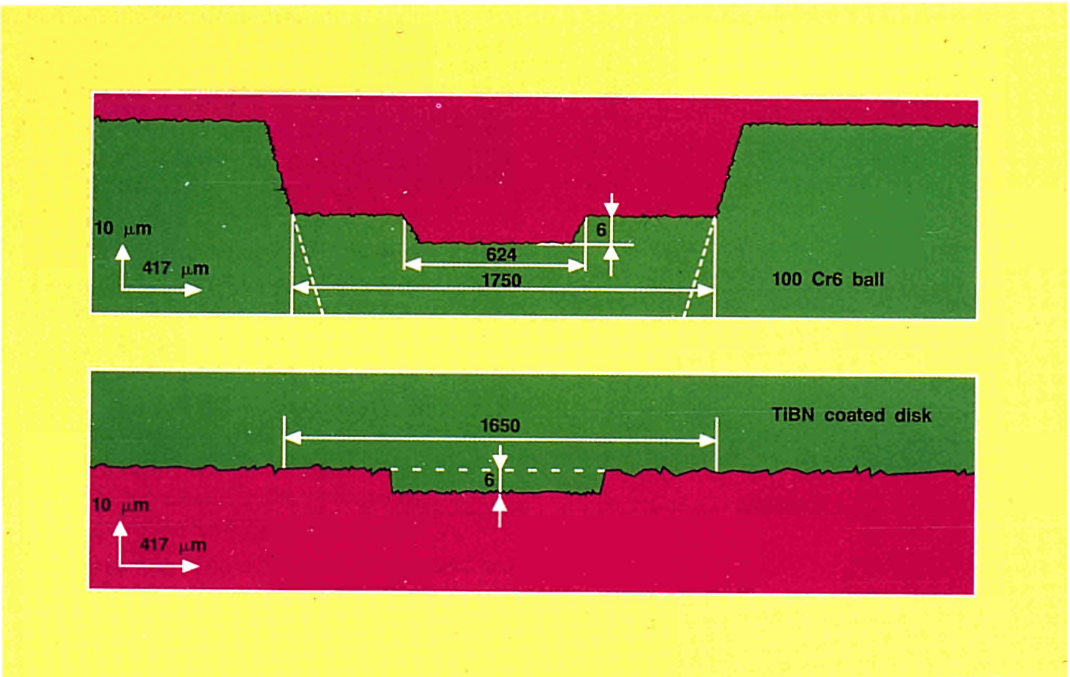
Two new techniques for the preparation of very hard Ti-B-N coatings were developed. The first method is accomplished in two steps: the deposition of a multilayer coating of the sequence TiBN by non-reactive sputtering from a Ti and a BN target, respectively, followed by thermal treatment to induce a diffusion-controlled mixing process leading to phase transformations. This technique allows ultra-hard, almost stress free coatings to be obtained which have good adhesion to metallic and non-metallic substrates. The second method is a co-sputter process from a Ti and BN target. With this method, however, coatings of considerable compressive stress (comparable with sputter-deposited TiN coatings) are obtained.

Ti-BN-coatings deposited by the two stage procedure onto a high speed steel substrate (HSS) have been subjected to wear tests by the pin-on-disc machine. As described in figure a) on the next page the Ti-BN coating, when run against a 100Cr6 steel pin, is characterized by a fairly high friction coefficient but significantly reduced disc wear up to about 500 m sliding distance. After coating breakthrough, increased wear of both substrate disc and pin is observed, as follows from the wear track profiles also shown in figure b) on the next page.

Page 30 above: *Record of friction and total wear during sliding of a Ti-BN-coated HSS/100Cr6 pair*

Page 30 below: *Wear track profile of the 100Cr6 ball and the Ti-BN-coated HSS disc.*


L = 10 N
Øtr = 15 mm
v = 0.1 m/sec
Friction coefficient
1
0.8
0.6
0.4
0.2
250
200
150
100
50
0
250
500
750
1000
Slinding distance (m)


10 µm
417 µm
624
6
1750
100 Cr6 ball
TiBN coated disk
1650
6
10 µm
417 µm

4. Hard Carbon Films

Hard carbon coatings were prepared under various conditions using a dual beam deposition facility and analyzed with respect to their sp^2/sp^3 ratio. Progress was achieved by developing a computer programme to derive from experimentally determined IR-absorption spectra the relative concentrations of sp^2 and sp^3 bonds. Measurements with a new ultra low load and depth sensing nano-indenter showed a good correlation between the concentration of sp^3 bonds and the hardness of the hard carbon films.

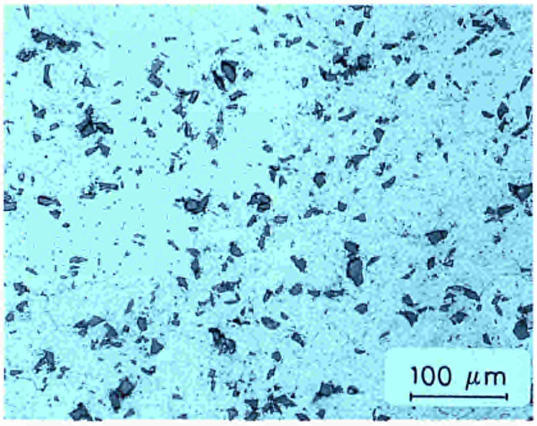
Properties, Performance, Characteristics and Innovation of FUNTIONAL MATERIALS

Composite Materials Properties Improvements

The study has focused on an Al-SiC particulate composite produced through a vacuum plasma spray co-deposition technique. This process features the high cooling rate experienced by the sprayed materials which has been assessed to be of the order of $10^7 Ks^{-1}$. This gives rise to products with a fine microstructure and to the complete elimination of macrosegregation which leads to a significant improvement of the mechanical properties.
In particulate composites the co-spraying route allows an excellent particle distribution to be achieved even with high volume fractions. This characteristic avoids the embrittlement of the composite due to premature crack nucleation and growth along the intergranular composite regions.
The composite materials were manufactured using a Plasma Technik AG (Switzerland) equipment. Several samples, in the form of platelets a few milimeters thick, were obtained by spraying the powders onto a Cu substrate. Two different composites were taken into consideration: Al-SiC and Al(7Si)-SiC as shown in the table below.
Figure above supplies an example of the composite microstructure obtained; it refers to the Al-20% SiC composite. The SiC particles are homogenously dispersed in the Al matrix without any evidence of significant variations in the local volume fractions. In fact, in plasma sprayed composites the molten metal is deposited, along with the solid SiC, through the build up of several thin layers so that the reinforced particles, which are distributed in a stochastic manner, retain their original impact position in the deposit even after the complete solidification of the material.

Table below: *Materials investigated*

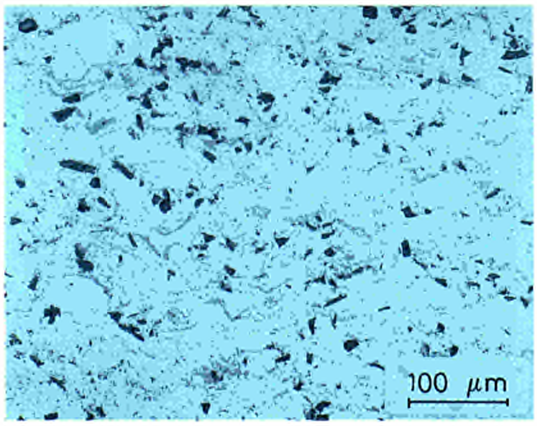
matrix	nominal SiC fraction (wt. %)
Al	0
	20
	33
	50
Al-7%Si alloy	0
	20



Conversely, in ordinary casting processes, the SiC clustering is promoted by the growth of the solidification front which shifts the particles to the regions which solidify finally, namely the interdentritic or intercellular zones. Since it has been recognized that the actual local SiC fraction in these regions is strictly related to the extent of embrittlement which takes place in this category of reinforced materials, the optimum particle dispersion obtainable through the vacuum plasma spray process can be considered to be one of the main advantages of this production route. A micrograph of the Al-7% Si-20% SiC composite is shown in the figure below.

Above: *Microstructure of the Al-20% SiC composite*

Below: *Microstructure of the Al-7% Si-20% SiC composites*



The matrix microstructure is not affected by the SiC addition; as mentioned, it is the result of the rapid solidification of the sprayed blended powders; it is composed of almost full-Al regions along with distinct coarse Si-rich phases. The SiC particles are still homogeneously dispersed in the matrix without showing any preferential disposition between the Al or the Si rich regions.
As far as the actual SiC amount deposited with the Al-7% Si matrix is concerned, the quantitative metallography data do not indicate any significant variation in comparison with the corresponding fully Al matrix composite.
The production of Al-SiC particulate composites through the vacuum plasma spray co-deposition technique features the following characteristics:

1 - The sprayed deposits show limited amounts of microvoids even at the matrix-particle interfaces.
2 - The SiC particles are dispersed in a stochastic manner without showing any segregation.
3 - The maximum SiC fraction obtainable in the sprayed composites is limited by the amount of the impacted SiC particles that can be retained on the semi-solid layer of the deposited material.
4 - From a microstructural point of view, the Al-SiC interface does not show any reaction product and reveals a close contact between the two phases.
5 - The use of Si powder as alloying element for the Al matrix does not influence the composite microstructure in terms of SiC dispersion or interface properties. The Si is present in the matrix in the form of distinct coarse phases because of the low solid state elemental diffusion.
6 - Further developments on Al-SiC particulate composites produced by vacuum plasma spraying for industrial applications are feasible and promising.

Chemical Sensors

The aim of the activity is to investigate the sensing properties of solid state sensors for chlorine and nitrogen oxides. This activity has been developed following two independent lines:
1) Semiconductor sensors
2) Potentiometric sensors

1) Semiconductor sensors (for NO_x)

1.1. Completion of measuring set-up for surface electrical conductivity

A system for electrical conductivity measurements and for preparation of calibrated gas mixtures has been designed, developed and assembled. It is fully automated both for the control of the feeding gas mixture and for data processing.
A second even more sophisticated line has been set up with the possibility of controlling even quaternary gas mixtures, thus allowing fairly accurate evaluations of cross sensitivities.

1.2. Preparation of samples

The samples, which consist of doped SnO_2 films on different substrates, have been prepared starting from the thermally oxidized (under controlled conditions) silicon sheets and from polished alumina supports, on which SnO_2 was deposited.
The deposition of the layers was carried out both by RF sputtering of SnO_2 and by spray pyrolysis of organometallic compounds.

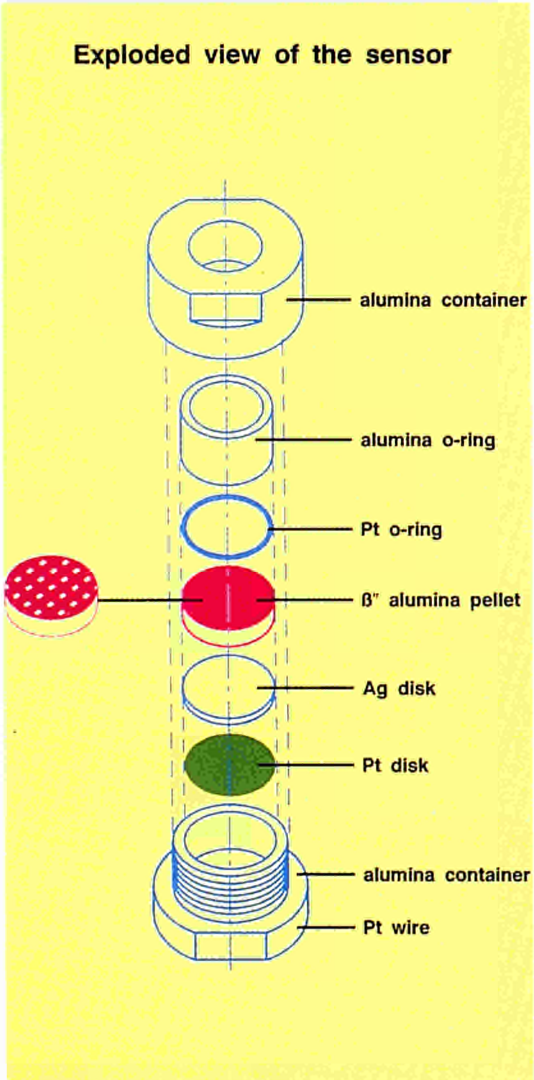
1.3. Characterization

The characterization of the layers for their structure and chemical composition is of paramount importance for the sensitivity and selectivity of the sensor. Therefore some surface characterization procedures have been adopted. The layers are polycristalline structures, the thickness of which has been determined by ellipsometry and Auger Electron Microscopy. The morphology (grain size) and the crystallographic structure were analyzed by X-Ray Glancing Angle Diffraction. The degree of oxidation has been determined by Electron Spectroscopy for Chemical Analysis (ESCA). An important problem has emerged, relevant to surface conductivity measurements: when oxidizing silicon sheets, the sensing SnO_2 film is a homogeneous semiconductor only in the first 40-50 Å layer. At greater depths SnO_2 (semiconductor) is mixed with SiO_2 (insulator), thus making the conductivity response more complex.

2) Potentiometric Sensors

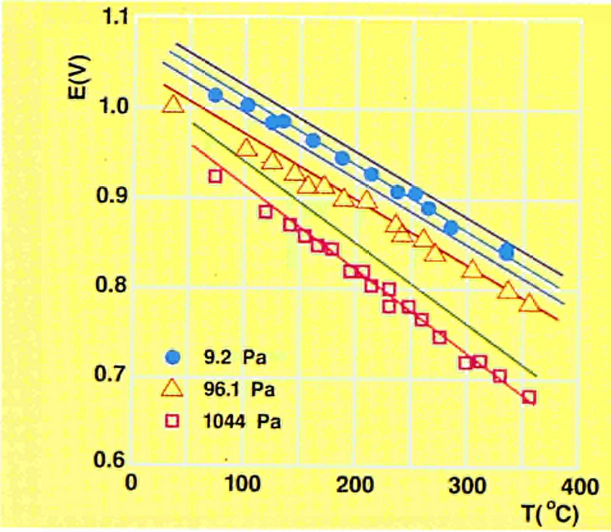
Electrochemical cells having a semiconducting solid electrolyte have been built up and tested for monitoring chlorine (Figure below).

Below: *Exploded view of the sensor*

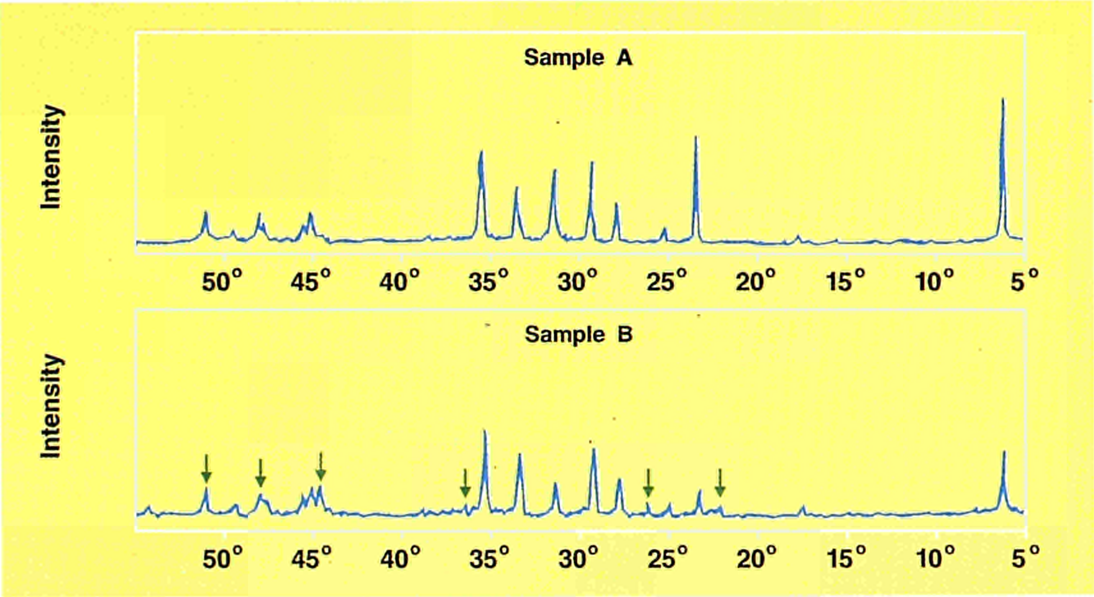


As a solid electrolyte both silver chlorine and silver ion conditioned beta-alumina have been utilized with satistatory results. The potentiometric response is shown in Figure above.

Studies are in progress for extending such potentiometric sensors to the detection and determination of nitrogen oxides.
The results for nitrogen oxides, however, are not very encouraging at the present time.



Above: *Electromotive force vs. temperature for a β"-alumina sensing cell for different chlorine partial pressures*

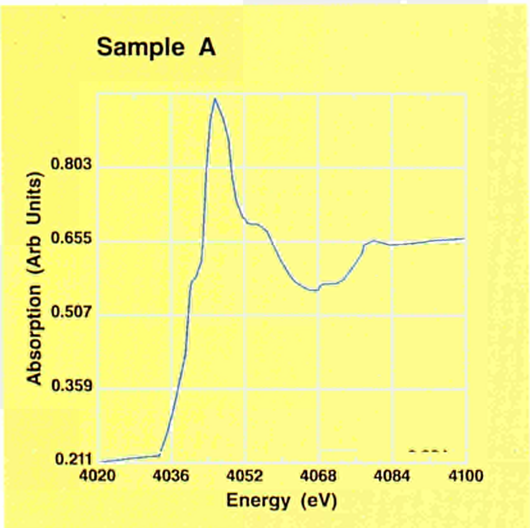


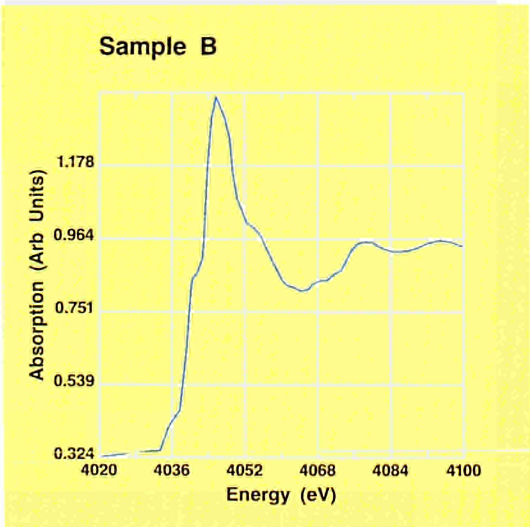
An Investigation of the crystalline and electronic Structures of a 4:3:3:4 layered Bi-Ca-Sr-Cu Oxide superconducting system.

Semiconducting samples of a Bi-Ca-Sr-Cu oxide, having a nominal composition 4:3:3:4, were prepared by melting high purity oxides of the various elements. Superconducting samples were obtained by annealing the semiconducting samples at a temperature close to the melting temperature. Electrical resistivity measurements indicated the presence of two superconducting phases, one with the onset temperature at about 110 K, the other with a significantly lower onset temperature. The presence of this second phase is proven by X-ray powder diffraction patterns. (Figure above). The crystalline structure of both the semiconductor and superconductor samples have almost equal lattice parameters, are orthorhombic but belong to different space groups: Fmmm and Bbmm, respectively. No significant amount of the 110 K superconducting phase was present in the diffraction pattern of the superconducting compound. XPS and XANES (Figures below) data on the Ca atom indicate that a different electronic state of Ca is present in the superconducting compound, which still contains an appreciable amount of the semiconducting phase.

Above: *X-ray powder diffraction patterns of sample A (semiconductor) and B (superconductor) Ni filtered CuKα radiation. The arrows mark the six extra reflections of sample B*

Below: *XANES data at the Ca absorption edge of a) the semiconducting A sample and b) the superconducting B samples. Note the small shoulder at the beginning of the absorption curve of the superconducting B samples*





Modulation of SURFACE PROPERTIES

Surface Treatments for Improved Performance

Surface Modification Centre

Equipment.

The different parts of the centre have been working satisfactorily since their final installation in 1989. The ion implanter has been equipped with a ion sputter source which substantially simplifies the implantation of metallic ions. It is now rather easy to change the equipment from one ion species to another and the source is no longer exposed to the heavy corrosion occasioned by the presence of chlorides. The indication of the total energy delivered by the laser has been calibrated by a calorimeter developed in the lab. Similarly, a measuring procedure for the energy absorbed by the different surfaces has been elaborated and used for studies on the energy dependence of the laser treatment. The coating facilities have been enlarged by a flame spray equipment.

Ancillary Facilities

- Corrosion in aqueous solutions at temperatures up to 200°C

Five autoclaves, each of 4 l volume have been delivered. Two of them with PTFE liners and PTFE internally protected armatures are provided for corrosion tests in aqueous solutions containing reducing agents, in particular hydrogen sulphide. The other three arrived with transport damage, which had to be repaired in cooperation with the manufacturer. The installation, which requires a section of the hall of building 28F to be prepared with fumeholders for the work with hydrogen disulphide, electric power lines and tubings for security valve exhaust etc.., will be completed in 1991.

Ion Implantation

Implantations of a number of different ions have been performed on a variety of surfaces (Table below).

Table below: *Ion Implantations*

Specimens	Target	Coating	Ions	Energy KeV	Dose $10^{17}cm^{-2}$
5	Al_2O_3	Al	Ar, He	200	2 - 6
1	SiO_2	Au/Ni	Ar	100	1
3	Fe Cr Al	Y	Ar	200	0.1 - 2.5
3	Steel	Al	Ar	200	1 - 10
3	Fe	Al	Ar	200	1 - 10
3	Al_2O_3	Al	He	200	2 - 6
8	316	-	N	100,200	1 - 5
4	Fe Cr Al	Y	Ar, Y	200	2.5, 5
5	SiO_2	Cr/Y	Ar, Y	100,200	0.2 - 2
3	Al 3Mg	-	N	150	10 - 60
3	Al	-	N	150	10 - 60
1	AISI	-	Y	200	1
1	Ni Cr	-	Kr	200	1
4	Al_2O_3	Al	Ar	100	4 - 10
1	Steel	-	N	200	5
3	SiO_2	C	N	20	0.01 - 1
4	Fe	-	N	200	2 - 8
4	Fe 42 B	-	N	200	2 - 8
2	In 800	-	Xe,Cr	100,200	1
1	Diamond	-	N	190	1
1	Al_2O_3	-	Cu	200	1

Ion beam mixing of the system Al/Al_2O_3

In the production of composite materials, such as aluminium matrix reinforced with aluminium oxide fibres, the metallic Al is known to adhere badly to the ceramic Al_2O_3, thus causing interface structure problems. The adherence of metals to ceramics is reported to generally improve when they are ion beam mixed.

In a preliminary experiment an Al-layer of 100 nm + 10% was ion beam mixed with an Al_2O_3-substrate by argon ions, Ar^+ of 200 KeV.
The total doses were in units of 2.10^{17} Ar^+/cm^2 for 600 sec. The vacuum was about 10^{-6} mbar and the target temperature between 300 and 350°C.
After the Ar^+-implantation the concentration profiles of Al and O_2 have been determined by means of AES. For technical reasons it was not possible to obtain the profiles of argon.
All the samples, including the unimplanted reference samples, had the same shapes of concentration profiles, independently of the Ar^+ dose. An Al-layer (or otherwise altered structure) of about 80 nm was found on the surface of each sample, indicating that during implantation the Al-layer was transformed into Al_2O_3 and that no ion beam mixing had occurred.

The microhardness of AISI 316 after the implantation of 200 KeV nitrogen ions

The microhardness of stainless steel, implanted with nitrogen ions is reported to improve either because of the formation of nitrides (only in presence of martensite) or because of solid solution hardening.

The role of thermal treatments before and/or after implantation as well as the presence of high chromium concentrations is not yet agreed upon by different authors, nor is the dependence upon the N^+ dose.

The samples were obtained from a sheet by cutting (S) and from a bar by spark-erosion (B). The surface was only lapped to about 0.4 micrometer of roughness and not polished.

Between 1.10^{17} and 5.10^{17} N^+/cm^2 at 200 KeV were implanted into the target surfaces.
The vacuum was 2.10^{-6} - 8.10^{-6} mbar and the target temperature below 200°C.

The following results are in Vickers hardness.

	S	B
initially	200	360-430
after lapping	400	390-430
after annealing 30 min., 1075°C	170	200
after implantation	180	190
after annealing 4 h, 400°C		

ESCA and AES graphs indicate that nitrogen is present in the surface and that it is distributed to a depth of 550 nm (0.5 micrometer), whereas the maximum penetration depth is 400 nm.
Possibly in this case, with an altered layer of only 550 nm, the measurement of the microhardness with the usual standard method was not justified. No nitrides were formed in spite of the stimulating treatment at 400°C for 4 hours. This may be the reason why the microhardness has not improved.

Laser and electron beam melting

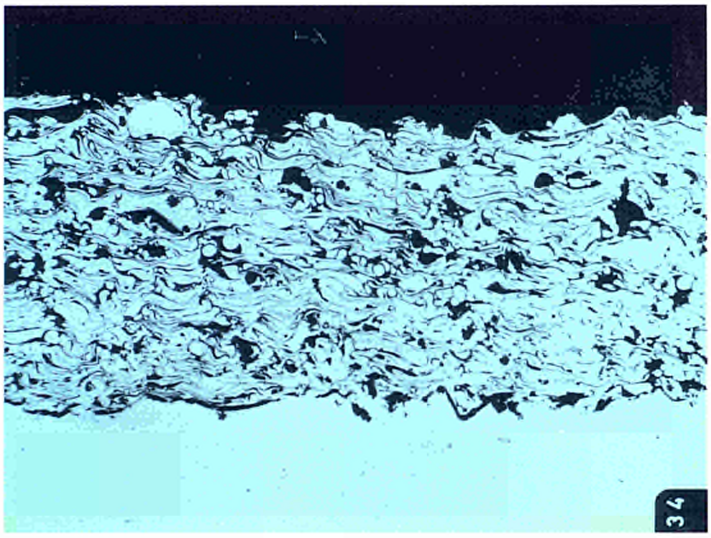
Copper plates coated with aluminium by low pressure plasma spray have been subjected to electron beam and laser surface melting of about the same power density and with similar scanning speeds.
The alloy structures which formed were similar and no systematic dependence on the melting method could be detected. The alloy structure produced at the copper surface could be improved substantially by heating the substrate at higher temperatures during surface melting. Partial overlapping of the different melt traces which led to multiple melting improved the homogeneity of the alloy layer.
Laser surface melting of stainless steel was attempted in order to improve the hardness of its surface. At high speeds of the beam across the specimen it was very difficult to produce constant melt depth. The coupling of the beam and its variation with time at high speeds was followed with high speed cameras. At the end, an optimised regulation of the protection gases from the nozzles was found. Depending on the microstructure produced by laser melting, a wide range of hardness could be obtained, the maximum hardness reached so far being 520 VH in fine dendritic structures. For wear tests these structures have now to be produced on a larger scale and with sufficient thickness to allow post melt machining.

Plasma Spray Coatings For Power Plant Application

The aim of this study is the development of erosion/corrosion resistant coatings to be used in pressurized fluidized bed combustion environments. Tribaloy T800 (a cobalt-based alloy having excellent wear, corrosion and oxidation resistance) and coatings of the type MCrAlY/Al_2O_3, have been deposited in air and in vacuum on nickel-base alloys by plasma spray techniques. The coatings have been characterized in terms of structure and morphology by optical microscopy and by SEM analysis. The adherence of the films has been tested by thermal shock.

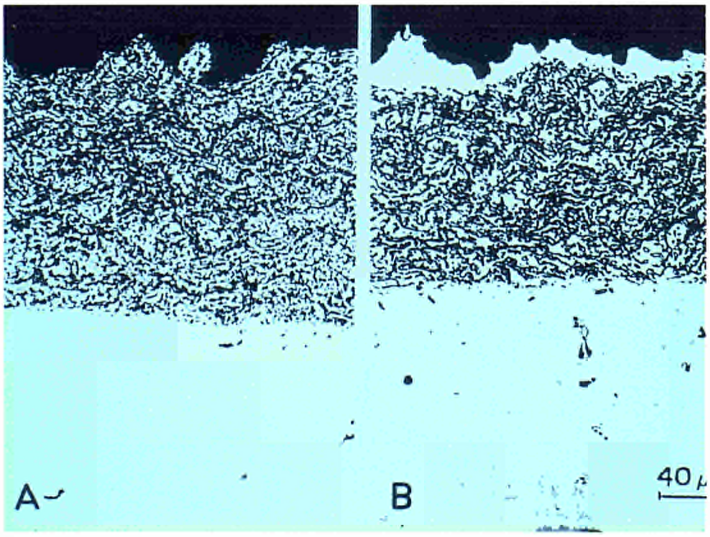
A thermal treatment in vacuum at 1080°C for 4 h reduces the porosity of the coatings and increases the adherence. A diffusion layer is produced at the coating-base material interface on all treated specimens. By heat treatment, the metastable phases are transformed to more stable phases and to a crystalline structure. The grain size increases and the hardness of the coatings decreases with the thermal treatment (600-700 HV_{500} for Tribaloy T800).

Surface remelting of the coatings has been tried using an electron beam gun and a laser facility.
The Figure above shows a cross-section of Tribaloy T800 film produced by air plasma spray.



Above: *Cross-section of Tribaloy T800 film produced by air plasma spray*

Below: *A - MCrAlY coating*
B - same coating after corrosion test;
500 hours - 950°C - Ar + 10% O_2 + 0,1% SO_2



High Temperature Corrosion

The corrosion loop (described in detail in the last report) for the study of the behaviour of superalloys in gas turbines environments, became operative in September. The delay, with respect to our original planning (April-May) was due to the late delivery of essential parts of the installation: mass spectro-meter and circulating pump.

A first 500 hours corrosion test was carried out on samples of both coated and uncoated IN 738 at 950°C and 1100°C in an environment 10% O_2 and 0.1% SO_2 with Ar as carrier gas. The specimens were coated in our laboratories by VPS with a MCrAlY type coating.

From Figure below, it appears that the uncoated samples are corroded with evidence of internal attack, while there is no sign of corrosion on the coated specimens for both temperatures. A complete metallographic analysis of the samples is underway.

Microstructure development in laser remelted Fe-10Cr-18Mn-0.2N alloy

The present work introduces the results of a study of the solidification microstructures conducted on an Fe-18Cr-10Mn-0.2N alloy, after laser remelting at different energy inputs.

In the case of high energy density processes such as laser welding, the cooling rate (about 10^3-10^6 Ks^{-1}) and the energy density per volume are the fundamental parameters which influence the materials microstructure (grain form and dimension, the amount of micro and macrosegregation, inclusion distribution, presence of defects, etc). With respect to conventional welding characterized by lower cooling rates (about 10-10^3 Ks^{-1}) laser welding changes the solidification mode and the possible solid state transformations during cooling after solidification.

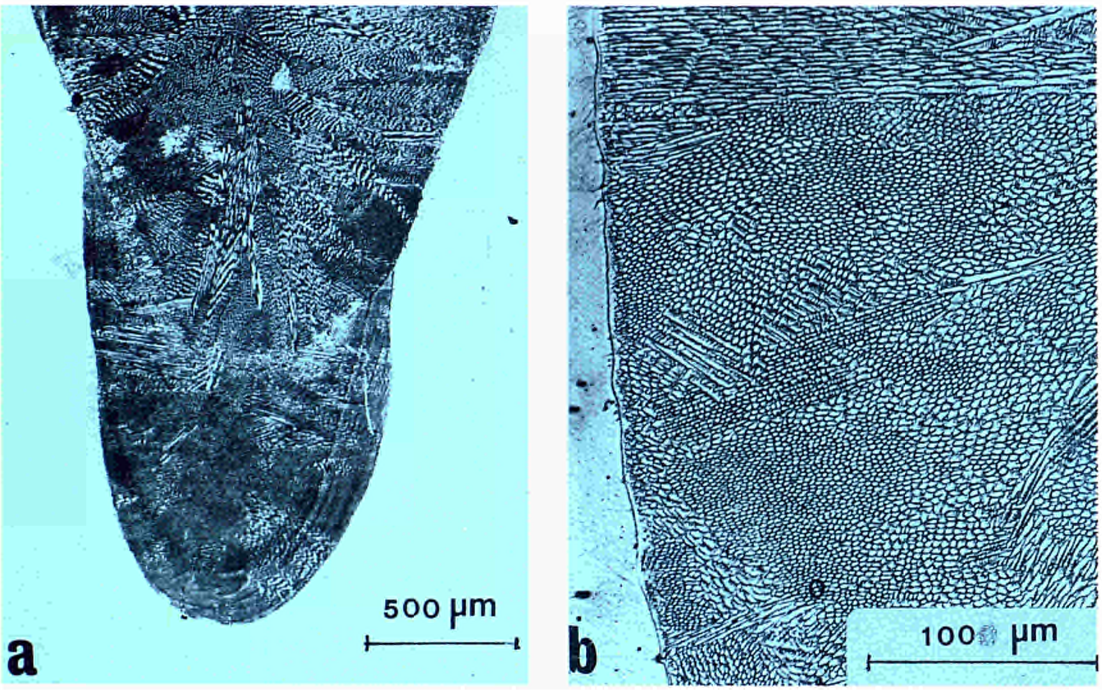
Weldability tests were performed with various laser scanning speeds at different energy inputs on Fe-18Cr-10Mn-0.2N specimens (see Table below).

The corresponding microstructures were characterized from the structural and mechanical strength points of view.

As shown in the table below, in the first series of the tests (melt depth 3mm), remelting has been limited. In the other cases the full specimen thickness has been affected. In the second and third series the authors used passes from both sides (up and downwards), while in the fourth, because of the high power of the laser used, a single pass was sufficient.

Table below: *Characteristic parameters of the laser remelted Fe-18Cr-10Mn-0.2N specimens.*

Test No.	Specimen thickness (mm)	No. of passes	Nominal Power (W)	Scanning Speed (mm/s)	Melted zone (depth)	Melted zone (width)
1 ROFIN-SINAR 5000 W LASER CCR-Ispra	6	1	1250	16.7	3.0	1.5
2 ROFIN-SINAR 5000 W LASER CCR-Ispra	6	1+1	2480	16.7	4.0	2.0
3 2500 W LASER CISE-Segrate	6	1+1	2500	13.3	4.0	3.0
4 7000 W LASER RTM-Vico Canavese	6	1	7500	33.3	6.0	4.0



Regarding the microstructure, the results depend on the scanning speed and the energy input of the laser. As will be seen from Figure above, which represents a specimen remelted in the J.R.C. Ispra laser (Test 1, table on page 46), the structure varies mainly as a function of the melt depth; it goes from a columnar-dendritic type near the surface (figure above a), to a fine cellular one at the bottom of the melted zone (figure above b).

At the interface between the melted and the heat affected zones we can find a plane growth front with about 3 µm width.

The plane transition from growth front to cells to columnar dendrites, could be explained by the variation of the rate G/R (G = thermal gradient of the weld pool at the solid-liquid interface, and R = solidification front growth rate) which has a maximum at the bottom and the sides of the melted zone, as was already found for laser and electron beam melting of stainless steels. At high G/R rate values, a plane growth front prevails.
As G/R decreases cellular or columnar dendritic growth occurs.

Above: *Macrostructure (a) and microstructure (b) of a transverse section of an Fe-10Mn-18Cr-0.2N specimen laser melted at JRC Ispra.*
Fig. b shows an area at the left side of the melted zone, seen in figure (a) at lower magnification, with evidence of a cellular-cellular dendritic fusion morphology type.
A thin clear strip corresponding to the plane growth front can be seen at the melted zone-base material interface.

Temperature (°C)	Ultimate Tensile Strength (MPa)	0.2% Yield Strength (MPa)	Fracture elongation (%)
20	852 (854)	370 (390)	38.4 (54)
450	459 (470)	197 (187)	37.7 (63.0)

* Values between brackets refer to the base material.

Table above: *Tensile characteristics of the laser melted material (transverse surface to the solidification direction of the specimen).*

The decrease of the G/R value which occurs with the advancement of the solidification front is not sufficient to produce a equiaxed dendritic growth, not even in the central zone, where G and R take respectively the lowest and the highest values.
Similar considerations are valid for the structures obtained with the CISE and RTM lasers.

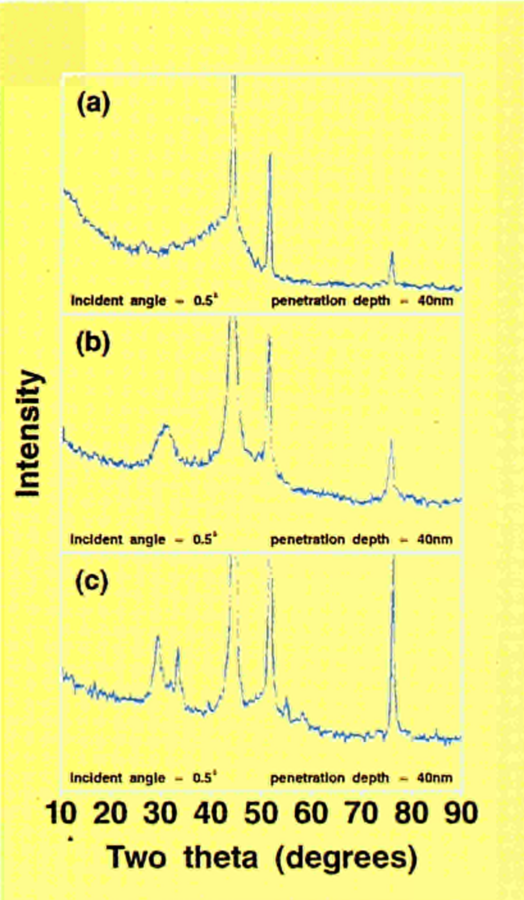
Chemical and structural analysis on coatings and implanted materials

To improve their corrosion resistance Fe/Cr and Ni/Cr alloys have been implanted by Y, Ce and Kr ions (200 KeV). Chemical and structural analysis have been performed by (XPS), Auger and X-ray glancing angle techniques. In particular, we have made glancing angle X-ray diffraction studies on a 200 KeV yttrium ion implanted Ni-20Cr alloy, and shown that such treatment amorphises a thin (about 500 Å) surface layer. Upon oxidation in a flowing gas mixture containing 1% oxygen and 99% argon at 700°C, the first structural change to occur is the segregation of yttrium to form "nanocrystallites" embedded in a reformed fcc Ni-20Cr lattice. The formation of thin surface oxide layers following this stage has been studied and compared to the behaviour of untreated Ni-20Cr and to an advanced mechanically alloyed Ni-20Cr (MA 754) containing 0.6 wt% yttrium oxide (see Figure below).
The same system has been studied using glancing angle X-ray absorbtion (EXAFS) at SERC, Daresbury SRS Laboratory.

XPS, Auger and X-ray glancing angle techniques have been applied also to investigate metal-ceramic bonding before and after ion mixing (Al-Al_2O_3, Si_3N_4-Cr), as well as TiN, BN, C coating to improve mechanical properties.

X-ray reflectivity was also employed to follow the very initial stages of surface oxide growth of pure chromium deposited on optically flat glass substrates. Again this system has been investigated using soft X-ray EXAFS at the Synchrotron Radiation Laboratory at Daresbury.

Below: *X-ray diffraction patterns of yttrium ion implanted Ni-20Cr alloy before oxidation (a) and after 1 minute (b) and 4 minutes (c) at 700°C in 1% oxygen/99% Argon*



Data and Information Management for ADVANCED MATERIALS

Data Banks

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 the requirements for data management, evaluation and input for computer aided engineering, finite element methods, computer aided processing and information services. It further serves the dissemination of data between collaborating parties in joint projects.

In 1990 a major effort has been devoted to the further improvement of the quality of the Databank functions up to the level set by modern commercial products. To this end, a beta test has been conducted amongst selected users in the different member states. This activity has led in several modifications of databank modules.

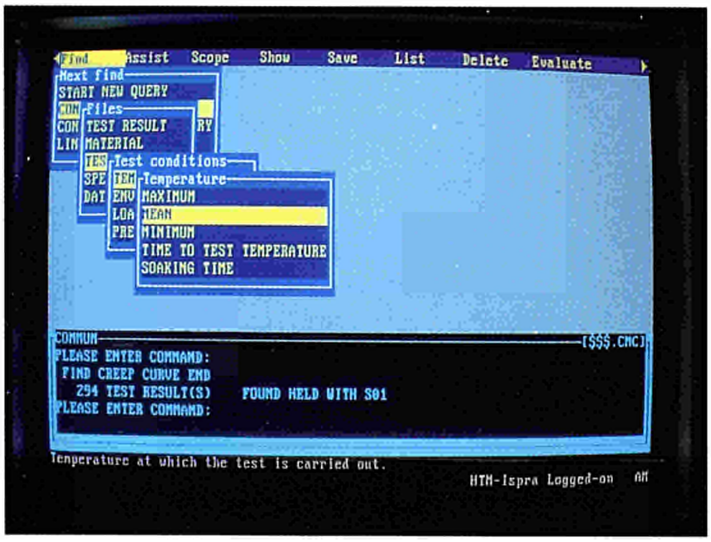
The user-friendly PC-based query interface underwent the most important changes. This remote shell requires minimal user training. It performs automatic logon and logoff and it uses advanced windowing techniques to assist the user in formulating his queries. Typing mistakes and non-relevant queries are avoided as the user selects from lists of allowed terms, such as the list of treatment types in the figure below. The PC-based interface furthermore eliminates syntax errors by gradually and automatically building up the command string, making the syntax fully transparent to the user.

Among other modules to have been completely revised, the data input PC-programme received extra functionality like pull-down menus, thesauri and some facilities for validation.

Old evaluation programmes have been improved and made more user friendly. New routines have been added to the Evaluation Programme Library to which datasets retrieved from the HTM-DB can be submitted for further elaboration.
The HTM-DB programme library now contains more than 70 Fortran programmes which can be selected from a menu system. An example of the recently implemented evaluation programme "KACHANOV EQUATIONS" with a sophisticated non-linear fitting procedure is given in the figure on page 52.

Some of the routines are simple spline or linear regression programs, whereas other modules of the Evaluation Programme Library allow the calculation of constitutive equations with user guidance through the different programme steps. These calculations allow the user to calculate actual material parameters for design purposes.

Below: *Example of a menu selection on the PC-based interface*



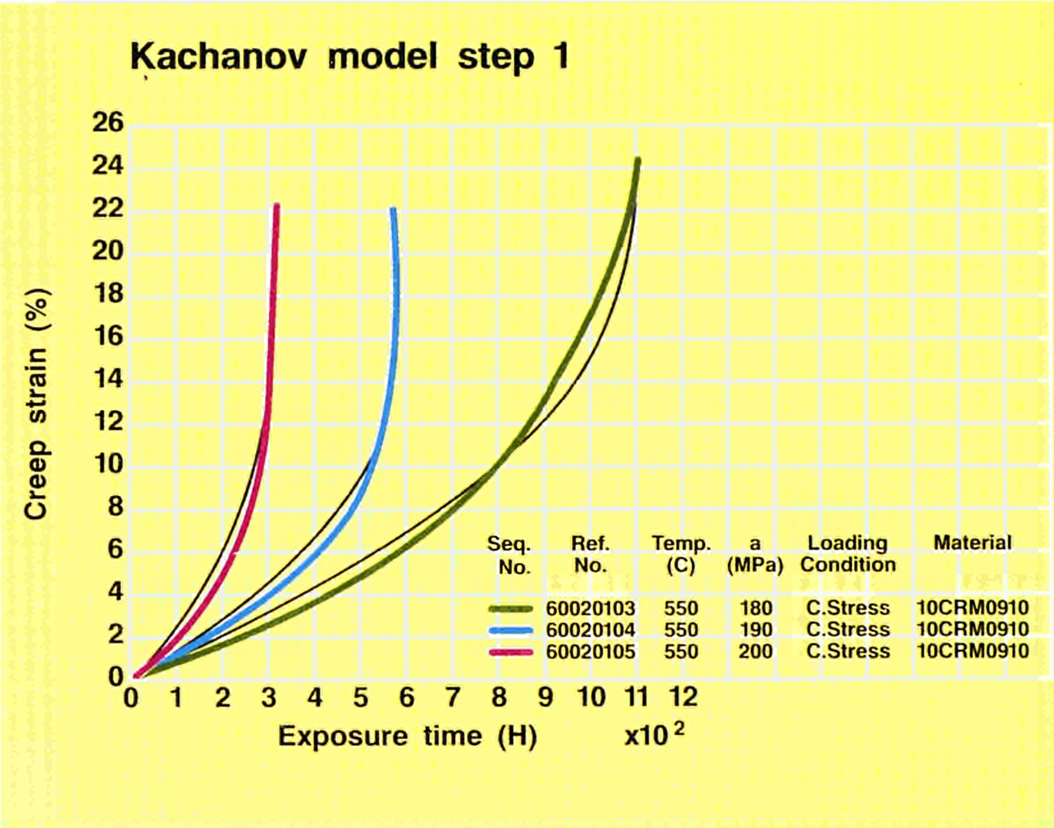


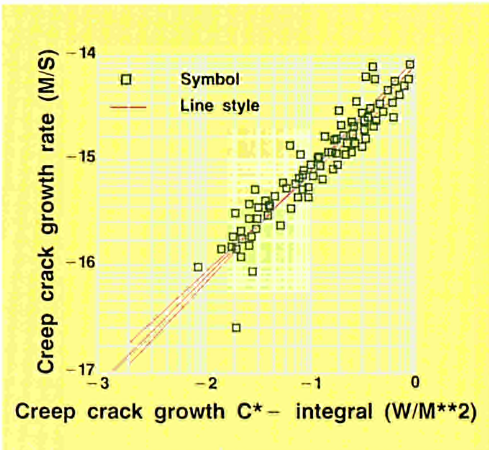
Figure below illustrates one of the possibilities to treat creep crack growth curves. Other additions were made in the areas of fracture mechanics and theta projection of creep phenomena whereas the overall statistical and graphical presentation was improved.

The extensive testing of the Evaluation Programme Library has lead to a de facto validation of the data through the systematic correction of errors whenever they occurred. With respect to new data, major input activities in 1990 were concentrated on the support of joint projects like VAMAS, COST 501 WP1 and WP5A, and BRITE 1209.

The modifications described were rounded off by a revision of the manuals and the elaboration of accountancy routines and contracts in preparation for a more widespread exploitation of the Databank. 1990 was characterised by a continued effort to promote the use of the HTM-DB and to exploit the acquired expertise. The Fusion Materials Expert Group agreed to use the Databank for data evaluation and dissemination.

Above: *Kachanov model step 1*

Below: *Creep crack growth rate vs. C*-integral, alloy 800H at 550°C*



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 1990, 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 two meetings were organised in February 1990 at Petten, i.e.

- the Ad-hoc Committee for pre-normative R&D for Advanced Ceramics identified in a Round Table research requirements pertaining to future standardisation,
- the CEN Technical Committee 184 discussed and confirmed its workplan for the period 90/92 comprising 42 standards and pre- standards for test methodologies and a classification system.

An industrial workshop (April 1990) on "Designing with Standard Ceramics", was organised in cooperation with the European Physical Society and the Energy Research Foundation (ECN) to discuss the status of thermo-mechanical applications of engineering ceramics. 50 experts attended. The following conclusions resulted:

- An increasing range of materials is available for high temperature use.
- The application of fracture mechanics has made considerable advances in understanding properties and performance, severe gaps in knowledge for high temperature conditions for monolithic ceramics and most topics concerning composites exist.
- Enabling technologies like machining, joining, NDT and fabrication procedures have made significant recent progress.
- Engineering applications have been established for the high tech aerospace/military industry, where materials costs are not paramount. For a broadening of use e.g. automobile and energy conversion there is still market resistance. Proceeding are under preparation.

VAMAS Workshop: Classification of Advanced Ceramics" in June 1990 at Ispra. Details are reported under the action S/T Support to the Services of the Commission: Standards for Advanced Ceramics.

Conference "User Aspects of Phase Diagrams" (June 1990) was co-organised with the Institute of Metals, U.K. The scope of the meeting focused on all aspects of the application of phase diagrams in the extraction, production, processing and the use of materials ranging from advanced ceramic superconductors, composites, electronic materials, as well as conventional ferrous and non-ferrous alloys. The impact of computer calculations of multicomponent phase diagrams via thermochemical experimental techniques provide more knowledge concerning the phase relationships in complex systems. The meeting reviewed in 30 papers and 11 posters the state of the art and provided opportunities for demonstration of software packages and thermochemical databases, operated by various laboratories. 70 experts attended the conference.

2. Contribution to the Specific Programme:

REACTOR SAFETY

Project for the Inspection of Steel Components (PISC)

Assessment of the Safety of Nuclear Structural Components

Safety assessment of structures involves mainly the structural integrity assessment of these components. Such an assessment can be divided into three major areas of R&D as follows:

Margin to Failure Evaluation (MFE)

Failure has to be avoided by evaluating the correct steps and margins to avoid any failure mechanism initiation; this also implies that the structural material must have suitable properties, such as adequate ductility or toughness. Several failure assessment methods have been developed which require the knowledge of the defect sizes and characteristics present in the structure.

Non-Destructive Testing (NDT)

The development of NDT techniques suitable for material and welded joint testing has been such that they could be incorporated into Codes and Standards. Well known techniques for thick wall vessels and piping are Ultrasonic Techniques (UT) and Radiographic Techniques (RT). An important feature is the "In Service Inspection". Inspection effectiveness and reliability generally depend on the availability of suitable NDT techniques, of the knowledge of the material characteristics (e.g. acoustic properties) and of automatic inspection devices.

Residual Life Evaluation (RLE)

If "thinking" schemes of margin to failure evaluation are available such as computation methods using material properties, stress analysis, fracture mechanisms and the status of the structure under evaluation, inspection of the component, then a new margin to failure can be estimated through hypothetical logic schemes or probabilistic schemes on loading history and material degradation. The residual life expectation can then be evaluated.

The PISC Programme

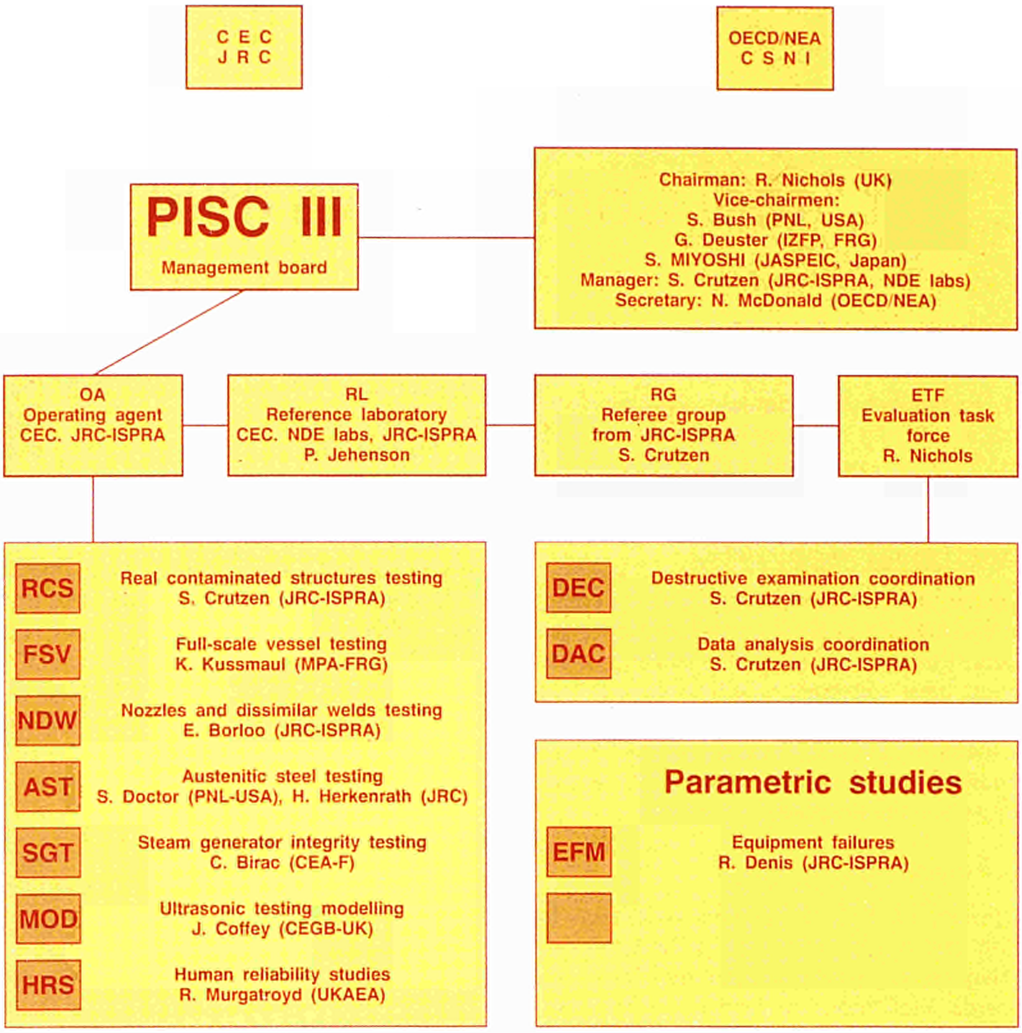
The PISC Programme has the general objective of assessing procedures and techniques in use for the inspection of pressure components, particularly vessels and piping.

The series of projects 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 to better assess the capability and reliability of Non-Destructive Inspection procedures on structural components.
The programme is now in its third phase PISC III (Table on page 58); the activities concentrate on the validation of the PISC II results (e.g. modification of the ASME Inspection Codes) on real structures containing service defects and the extension of the PISC methodology to important structural components made of different materials. Most of the PISC test assemblies and structural pieces are representative of (or coming from) nuclear reactor components.

PISC III Programme Status

PISC III requires demonstration using 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 austenitic steel testing is of real value for the inspection of LMFBR components. Eight programme Actions have been established as identified in the organization scheme presented in the previous annual report.
Round Robin Testing activities are presently performed in several of the PISC III actions. The final work to be undertaken in all eight Actions has been defined and most of the necessary test samples and other resources have been obtained. The uncertainties that prevailed during 1986/1990 concerning the level of budgetary support have been partly resolved by accepting delays for the Actions 4 and 5. Resources from the CEC and from participating organisations (participation in inspection, and contribution in kind) are now assured to carry out the essential elements of most of the planned actions.
The objectives of each of these eight Actions of PISC III were defined in the previous annual report. The status of the 1990 work is as follows:

Action No. 1 (Real Contaminated Structures)
In 1990, samples were available from Spain, Sweden, USA and Finland. Several of these components have been certified in the PISC hot cells; they are now allocated to the Reliability exercise of Action 4 (Austenitic Steel Testing).



Programme for the inspection of Reactor Steel Components, PISC III

The Programme is put under the aegis of

OECD/NEA and CEC/JRC

Budget	CEC budgets half the resources; countries bear own costs and make "in kind" contributions
Secretariat	NEA/OECD
Management	CEC Joint Research Centre, Ispra, Italy
Members	Belgium, Denmark, Italy, Finland, France, Germany, Japan, Netherlands, Norway, Spain, Switzerland, United Kingdom and United States

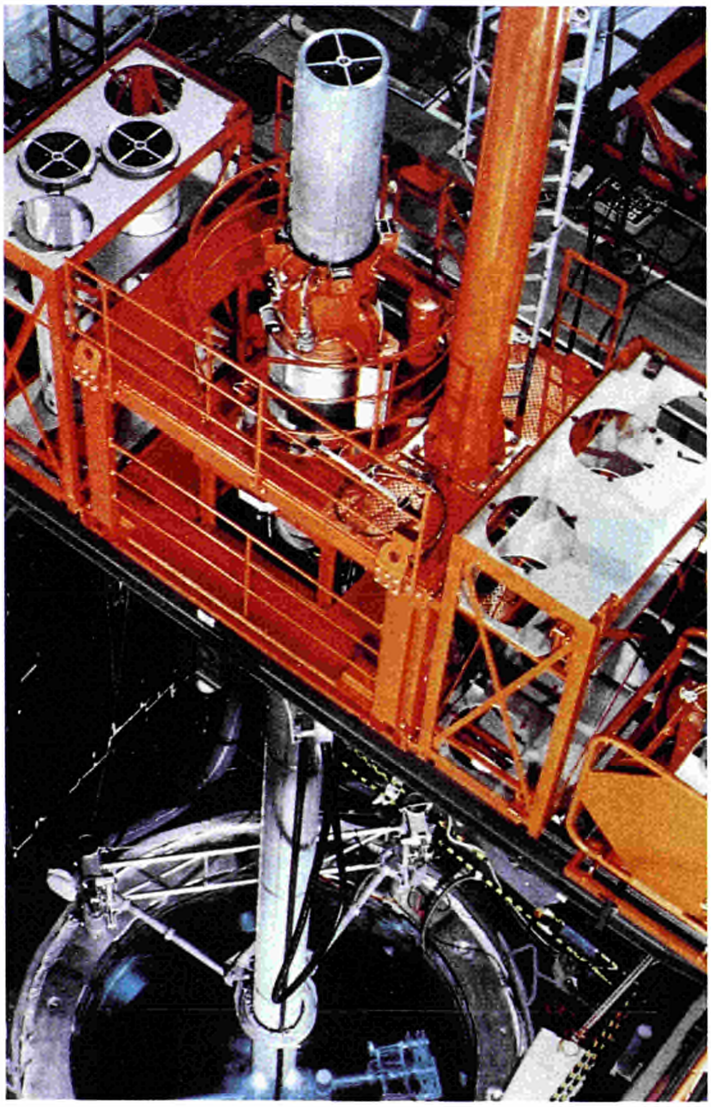
Above: *Organization and Funding of the PISC III Programme*

Action No. 2 (Full Scale Vessel Tests)

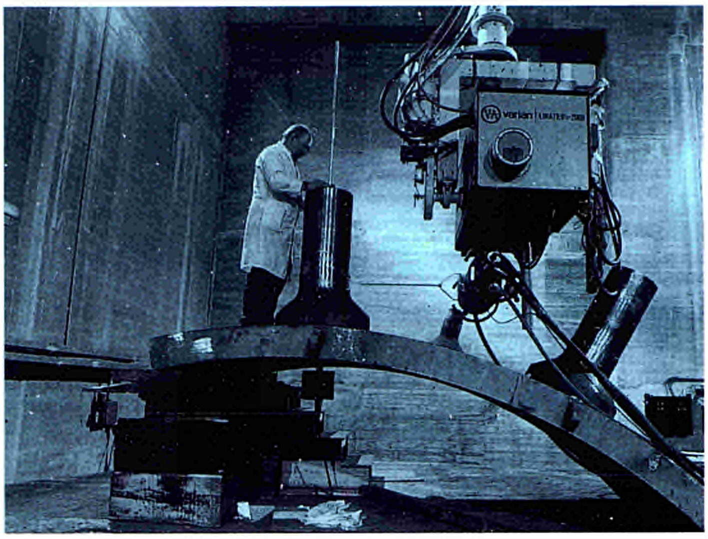
Eight organizations have registered their interest in the phase 2: validation of ASME type procedures by an international team using an ISI automatic scanner offered by RWE and MAN to PISC for the period of the exercise (September 1989 to April 1990) (Figure below). This Central Mast manipulator for vessel inspection from the inside was operated by MAN crew for PISC. KWU and MAN ultrasonic and recording/evaluating electronics as well as technicians were used for the execution of this phase 2 by the "Super Team", comprising staff of the JRC Reference Laboratory and national experts (Germany, Spain, Switzerland, Italy, Belgium and USA for ASME certification). The same crews and electronics were used for Phase 3 type inspection (national full procedures) from September 1989 to end 1990. One team of the ex-DDR took part in the exercise in autumn 1990.

Phase 1 and Phase 2 have reached the level of data evaluation and the very first trends can be drawn from the results, such as good sizing capability of several techniques and effective validation of the PISC II results obtained on the Assembly No. 3.

The work of Action No. 2 was largely supported by Germany through large assemblies, availability of the MPA installation and logistics, Central Mast Manipulator and Action Management.



Below: *RWE/MAN (Germany) central mast manipulator used with UT electronics and crew of MAN and SIEMENS, on the full scale components at MPA (University of Stuttgart, FRG) for Action 2, Phases 2 and 3 (1989-1990).*



Action No. 3 (Nozzles and Dissimilar Metal Welds)

An American BWR assembly with two nozzles and safe-ends (Figure above) and a Spanish PWR safe-end began their circulation mid 1989 and this is now completed. Destructive examination has started at the JRC Central Workshop.

This complex exercise, requiring various machining techniques, proceeds by alternating machining and new non-destructive testing to guide this machining.

It is an essential role of the JRC as Reference Laboratory of PISC to ensure the correct evaluation of the PISC results.

The Management Board of the programme has indicated during its last session that such work, even if delegated, had to remain under the strict continuous control of the JRC-IAM experts.

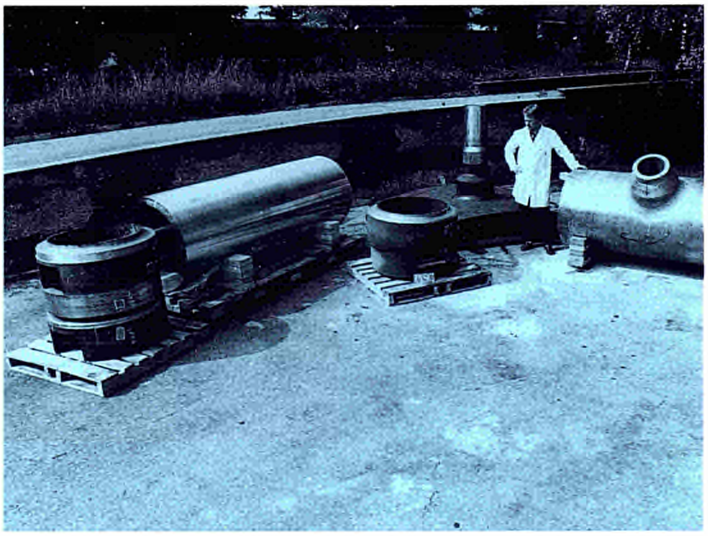
Action No.4 (Austenitic Steel Testing)

Twenty five teams have registered their intent to participate in one or more phases that will extend up to 1992. Large assemblies are still under fabrication for the RRT on cast austenitic steel (Figure left below). However, the Round Robin Test of forged piping assemblies started in October 1990 for a 2 years' circulation in 11 countries (Figure right below). This Action No. 4 suffered very much from the unavailability of resources necessary to procure the raw material, manufacture assemblies and introduce realistic defects.

Above: *PISC III Action No 3 (NDW). Assembly No. 20 with three nozzles and safe-ends of the BWR type: No. 21, No. 22, No. 23. Assembly No. 20 is also a key element for any evaluation of new inspection possibilities of old plants considered for life-extension (offer and manufacture from USA, Japan, Spain).*

Below left: *PISC III No. 4, (AST). Large castings ready for assemblies manufacturing when the last budget allocation will be available. Offer and manufacture from France, JRC, Japan, Spain.*

Below right: *Wrought Austenitic steel pipes welded assemblies (BWR type) now circulating among 20 teams. Offer and manufacture from Japan, USA, CEC, Italy.*



Action No. 5 (Steam Generator Tubes Testing)

Thirty-one teams from ten countries have registered an interest to participate in the RRT planned until 1992.
The validation of defects has involved experts from Belgium, France, Germany, Italy, Japan, Spain, United Kingdom and the United States of America. The Reference Laboratory (JRC/IAM) is preparing many artificial defects in tubes.
Orders have been placed from the Operating Agent to get realistic corrosion defects by CEA, MITSUBISHI, CEGB, KEMA. Emphasis has been put on corrosion defects (IGA, SWSCC, PWSCC) at three key locations: Tube Sheet (above the rolling zone), Tube Support Plates, U-bend transition.
Three training boxes constructed at JRC/IAM are in circulation (Figure above); the full RRT is expected to last until the end of 1992 due to the wide participation.

Action No. 6 (Mathematical Modelling on NDE)

The validation of the three first models has now given the assurance that models can be used to draw graphs and tables, without imperfections or errors due to the characteristics of different transducers, the various calibration and normalization operations, the imperfection of artificial defects and the secondary effects like wave transformation often depending on defect sizes.

Action No. 7 (Human Reliability Studies)

As already reported in 1989, part of this work was performed in the United Kingdom and at the JRC-Ispra utilizing the PISC II data and supplementary questionnaires.

Studies on human variability and influence of the limited access of welds for UT inspection were considered. Systematic observations are carried out at Risley UKAEA and at JRC Ispra through the inspection by industrial UT operators of special assemblies and components used in PISC Actions.

Some of them will be inspected under conditions reproducing practical industrial reality such as temperature, humidity and time constraints (Figure below).

Above: *Two of the three training boxes of Steam Generator Tubes containing realistic and artificial defects for the RRT on loose tubes*

Below: *PISC III Mobile laboratory for human factors identification*

Action No. 8 (Support to Code and Standard Organizations)

This Action, proposed by the Management Board and approved by OECD-NEA and CEC-JRC, has to give, through the PISC group of experts, direct support to Codes and Standards organizations. Such a proposal has been supported by all PISC members and involves three aspects:
- Information to Codes and Standards Technical Committees of PISC results and PISC related programmes results.
- On request, critical review by PISC members of technical documents made by national/international technical groups.
- Preparation of technical reports by PISC members related to Codes and Standards problems, for the benefit of National, CEN, ISO, IIW technical groups which develop standards.

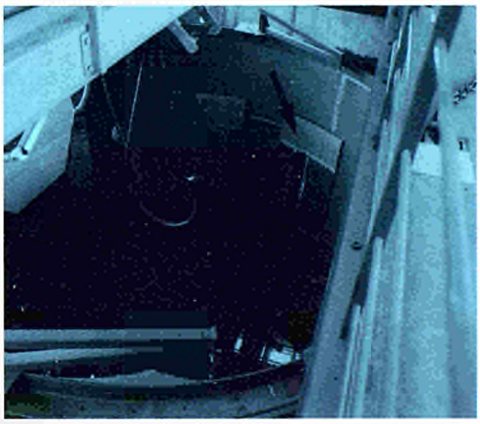
The organization and support of this Action 8 involves mainly the Operating Agent (JRC/IAM). A key theme of work is the "Performance Demonstration". The introduction of artificial "realistic defects" in blocks and structures allows affordable and reproducible validation samples.

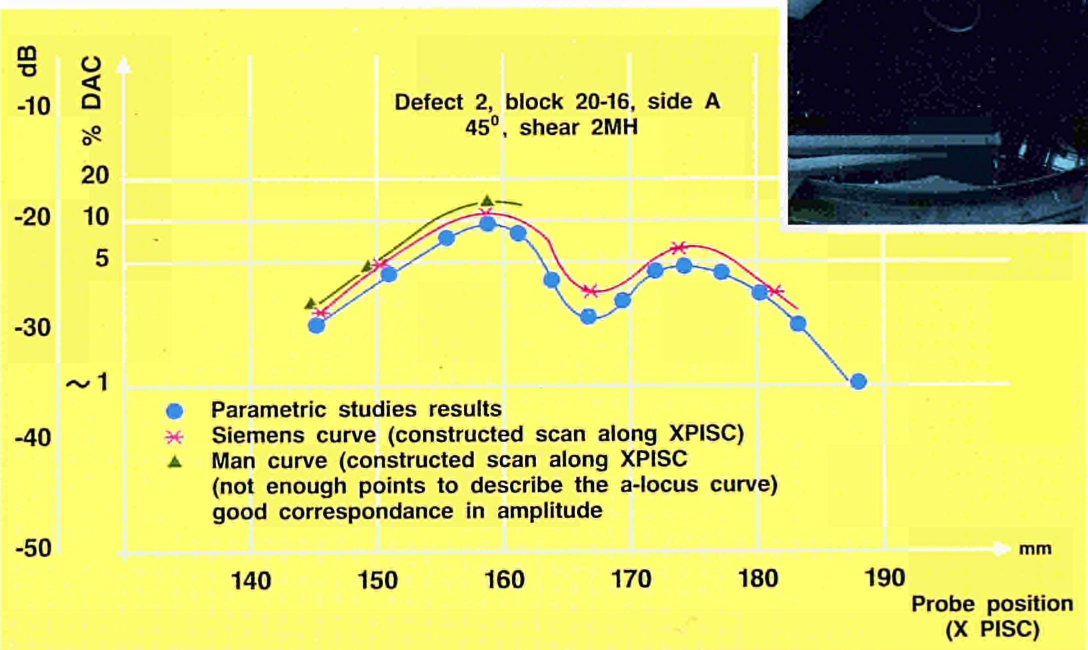
Mathematical models, involving a better understanding of the physical phenomena, indicate which artificial defects could be used to replace natural ones for NDT techniques validation and inspection teams training.

PISC results and the growing recognition of the value of validation assemblies persuaded ASME to modify significantly the Code for Pressure Vessels and Boilers, Section XI by the introduction in 1990 of a new appendix "Performance demonstration for Ultrasonic Examination Systems".

Also in Europe, performance demonstration should be used to harmonize the ISI procedures without "technical dispute". JRC/IAM has developed, with a network of European industries and laboratories the know-how to introduce realistic defects, as opposed to real defects as recommended by ASME. This principle of performance demonstration was already applied during the exercise on full scale vessel testing. During the inspection at Stuttgart with the Central Mast Manipulator (Figure on page 59), simple blocks of the parametric studies were positioned on the top of the vessel (Figure below) and scanned as well.

Below: *Performance Demonstration blocks on the top of the Full Scale Vessel (MPA, Stuttgart) and Amplitude Locus Curves (perpendicular scanning to the defect) drawn from MAN data, SIEMENS data and PISC II Parametric Studies data (CIDE-JRC) Validation of ASME type procedures for ISI*





The ultrasonic response of the huge Central Mast Manipulator corresponded well to the measurements made in the laboratory, validating the whole inspection system in the sense of performance demonstration (Figure on page 62).

Conclusions

The PISC programme is an example of international cooperation with effective conclusions, technical results and input to Codes and Standards.

The erosion of the political support to Nuclear Energy in many countries is such that no proposal can be made presently of further PISC work in the overall framework of Reactor Safety even if the ageing of the existing nuclear plants would require PISC type actions.

It is hoped, however, that the need for international Codes and Standards will be such that the Operating Agent of PISC (CEC) will continue to support Actions like Austenitic Steel Testing, Steam Generator Tubes Testing, Human Reliability Exercises until the effective possible use of their results by Codes and Standards bodies like CEN.

3. Contribution to the Specific Programme:

RADIO-ACTIVE WASTE MANAGEMENT

Safety of Final Storage in Geological Formation: Materials Research Aspects

Tests have been performed to analyze the influence of a backfilling, rich in calcium carbonate, on fissure behaviour of mild steel container under heat flow condition.
Under such conditions leaching and corrosion can be varied due to variation in either the diffusion coefficient of the different chemical species or to alteration in the composition of the backfilling.

A large number of leaching and corrosion tests have been performed in an apparatus producing a thermal gradient across a column of wet porous media in contact with the sample.

The main effect of the thermal gradient was in both cases the formation of a layer of calcium carbonate on the corroded surface, acting as a barrier against leaching and corrosion.

The tests performed on fissured system show that the protective layer was unable to fill, or even cover the fissure. As a consequence, the leaching of the glass at the bottom of the fissure continues at a reduced rate due to a decrease in the effective diffusion. During 1990 a series of samples of borosilicate glass loaded with Tc^{99} was prepared under reducing conditions. The glass was examined with an electron microprobe in order to define the form in which the technetium was present. Only small amounts, around 250 wt.ppm, were homogeneously dissolved in the glass. Most of the technetium was concentrated in metallic inclusions dispersed throughout the glass. Two different phases were identified, i.e. technetium as the main constituent with iron and nickel being the other elements and the reverse.

4. Contribution to the Specific Programme:

FUSION TECHNOLOGY and SAFETY

Materials Integrity

Behaviour of Mechanical Properties under Irradiation

HFR Irradiations

The FRUST (Fusion Reactor Utilisation of Stainless Steel) experimental series provides irradiation data for stainless steel tensile samples irradiated in a central position of HFR, using the SIENA (Steel irradiation in Enhanced Neutron Arrangement) irradiation capsule.

During this reporting period the irradiation of 316 steel welds at 250, 300, 350, 400 and 450°C up to 15 dpa has been completed. The welds were obtained by Electron Beam (EB) and Submerged Metal Arc Welding (SMAW).

During the same period new optimized Cr-Mn steels (IF type) were irradiated in SIENA at 250 and 450°C to 10 and 25 dpa. Irradiation creep experiments were performed at the HFR. Various type 316 stainless steels and AMCR-type steels were irradiated between 300 and 470°C up to doses of 5 dpa. The applied stresses were varied between 25 and 300 MPa. A negative creep elongation was found at the beginning of the irradiation, which was attributed to the formation of radiation induced precipitates in the steels. The negative creep elongation increases with decreasing irradiation temperature and decreasing applied stresses.

Cyclotron Irradiations

Fatigue Crack Growth under Light Ion Irradiation

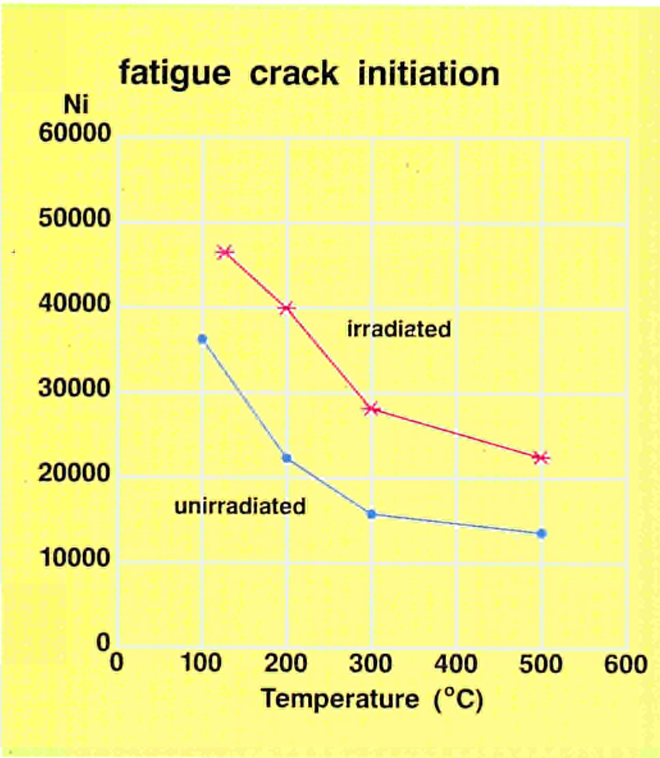
Fatigue crack growth initiating from pre-existing mechanical flaws is one of the major factors to be considered when evaluating the endurance life of the first wall of Tokamak Reactors.
An understanding of the interaction of fatigue crack growth and radiation damage is thus necessary for accurate estimation of first wall operating lifetimes.

In line with NET requirements, fatigue crack growth under cyclic tensile stress was measured in AISI 316 type stainless steel under simultaneous 20 MeV proton irradiation, producing a displacement damage rate of the order of 10^{-7} dpa s^{-1}, at 100, 200 and 300°C.

From the experimental results the following conclusions can be drawn:

- A clear effect of proton irradiation on in-beam fatigue crack initiation in miniaturized thin specimens of AISI type 316 stainless steel has been demonstrated in the present work. The number of cycles to crack initiation, N_i, is larger than that obtained in unirradiated specimens (Figure below), and the difference in N_i between irradiated and unirradiated specimens slightly increases with decreasing test temperature.
- The influence of proton irradiation on fatigue crack propagation appears to be more significant at the primary stage of crack propagation. In the low crack growth rate region, the corresponding K values are larger in the irradiated material than in the unirradiated specimens.

Below: *Influence of temperature and irradiation on fatigue crack initiation*



- Proton irradiation results in a slight decrease of the fatigue crack growth rate during the secondary stage of crack propagation.
- Irradiation tends to increase fatigue life, except at high temperature where it results in a decrease. The ratio of N_i to N_f is about 0.51, which is larger than that of 0.4 obtained from unirradiated experiments.

SEM examination of the fatigue fracture surface of AISI type 316 stainless steel specimens, in both irradiated and unirradiated condition, provided additional information useful in the interpretation of the low temperature fatigue behaviour.

Fracture surfaces of the specimens irradiated by 20 MeV protons are quite different from those of unirradiated specimens. This is attributed to irradiation hardening which causes a slightly lower rate of fatigue crack growth at low fluence and low temperature.

The effect of deuteron irradiation on the fatigue damage in type 316 L stainless steel

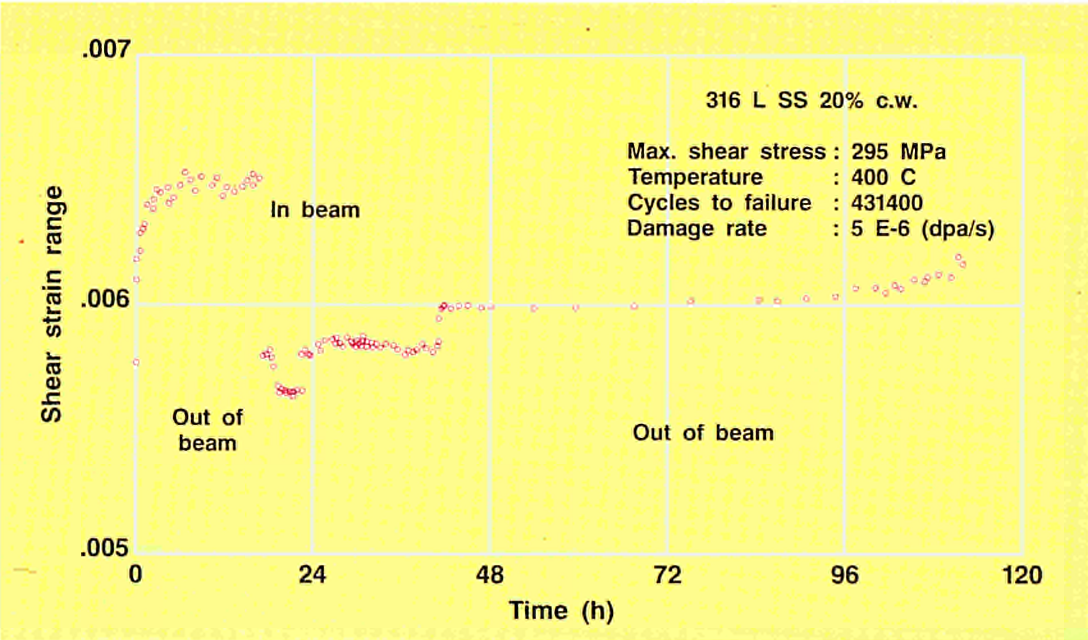
Torsional fatigue tests were conducted on AISI 316L stainless steel specimens under both thermal and irradiation conditions, in the temperature range from room temperature up to 400°C.

The thermal fatigue tests showed that fatigue damage D can be defined, for testing in torsion, in terms of bulk deformation parameters such as the load drop during strain controlled testing or the strain accumulation during load controlled tests. This damage description is apparently equivalent to defining fatigue damage by the surface crack length or the surface crack density, but avoids the difficulties of directly measuring small cracks.

The first fatigue tests under irradiation were performed on 20% cold-worked 316 L specimens under load controlled conditions and with a stress ratio of R = -1 for different maximum shear stresses at a temperature of 400°C.

An example of these tests, which demonstrates the main experimental results, is shown in the figure below, where the total shear strain S is plotted versus time. S is defined as the fatigue damage parameter in this case.

Below: *Strain-time behaviour of a stainless steel specimen under deuteron irradiation*



The specimen was first subjected to cyclic loading under thermal conditions before being irradiated for about two hours without applying a load.
Thereafter, cyclic loading was started, without interrupting the irradiation for about 24 hours, and was continued under thermal conditions until rupture
occurred.
This plot shows that:

i) irradiation hardening reaches saturation already after about 1000 s of irradiation, i.e. a dose of approximately 5×10^{-3} dpa.
ii) Cyclic loading during the irradiation softens the material and this softening effect is completed after a small number of cycles.
iii) Post-irradiation cyclic loading softens the material further, indicating that post-irradiation fatigue data for load controlled conditions differ from in-beam fatigue data.

Irradiation point defects in metals

The radiation enhanced diffusion coefficient of nickel in nickel has been measured in the near surface region and in the bulk. In the near surface region the concentration of vacancies does not depend on the irradiation temperature so that the activation energy derived from the experiments is the migration activation energy of vacancies. This means that the migration activation energy of vacancies can be determined in an irradiation experiment. From measurements of the self-diffusion coefficient in nickel single crystals the migration activation energy of self-interstitials could be derived. Both the migration activation energy of vacancies and of interstitials decrease with increasing high energy particle flux, a finding which is an important feature of the modified two interstitial model.

The modified two interstitial model has been developed in the last year and it could be shown that the dynamic steady state solution of the chemical rate equations is a mathematical solution rather than a physical description of the behaviour of radiation induced point defects.

The initial rate of formation of interstitial clusters and the equilibrium concentration of interstitial clusters has been determined as a function of the irradiation temperature for alpha-copper-zinc alloys containing 30% zinc. The transition temperature below which interstitial cluster formation occurs in this alloy was T_t = 158°C. It was further recognized that the transition temperature for interstitial cluster formation is also that of vacancy cluster and void formation in the alloy, i.e. above T_t = 158°C only vacancy clusters are formed.
It could be shown on the grounds of the chemical rate equations that the transition temperature depends only on the vacancy migration energy. Thus the transition temperature does not change with varying zinc content and even in pure copper is the same as in copper-zinc alloys.

This result is in agreement with the previously determined migration activation energies of vacancies in pure copper and in copper-zinc alloys, which do not change as zinc is added to copper.

It has further been established that the interstitial clusters formed in alloys are enriched in the undersized component atoms of the alloy e.g. are enriched in silicon in nickel-silicon alloys. In this alloy the phase boundaries in thermodynamic equilibrium during irradiation are almost independent of the irradiation temperature and the transition temperature of the precipitate formation is also that of interstitial cluster formation.

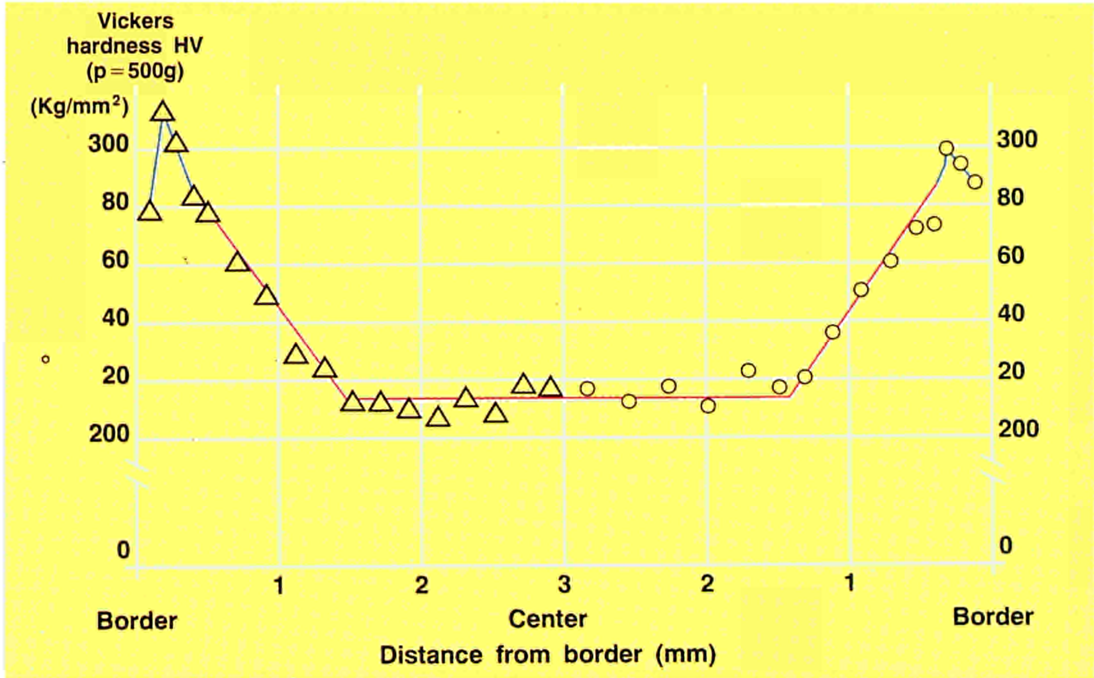
Plastic behaviour of CrMn-Steels

The tensile properties of metastable chromium manganese steel are influenced by the deformation induced martensitic transformation.
While beneficial in increasing the strain hardening rate and ultimate tensile strength, the presence of substantial amounts of martensite can lead to a brittle, transgranular fracture mode.
An investigation has been made of the effect of prior thermomechanical processing on transformation and plastic deformation in a 10Cr18Mn steel.
Two different approaches were used:

i) low temperature deformation and subsequent heat treatment;
ii) high temperature deformation alone.

The influence of these treatments on stress-strain behaviour and fracture is discussed. Transformation kinetics were studied by means of X-ray diffraction and magnetic measurements.

Microstructural observations have shown that dislocation substructures introduced by prior deformation inhibit transformation by acting as barriers



Above: *Variation of microhardness along the diameter after rotating bending fatigue*

to the growth of platelets of martensite. The resultant decrease in the volume fraction of martensite leads to an increase in ductility and the appearance of a region of non-uniform elongation before failure.

Optimization of chromium-manganese steels

The precipitation hardening processes of both IF-B and IF-D alloys can now be controlled and monitored by Vickers hardness against time plots.
For the D-alloy the times are longer and the temperature is somewhat lower.
A first series of tensile tests at 20, 200 and 400^{0}C on Alloy-B specimens shows very encouraging results, especially at high temperature.

In collaboration with CNR-ITM at Cinisello Balsamo a series of strain- controlled LCF tests at 450^0 and 550^{0}C has been performed on AMCR-33 in comparison with AISI-316, giving good results, especially at 550^{0}C.

In collaboration with the Politecnico of Milan a first series of rotating bending high cycle fatigue and instrumented tensile tests at room temperature was made on alloys IF-A, IF-C and IF-E. The tensile and fatigue properties of alloys E and A are interesting with respect to AISI-316L, but the fatigue properties are inferior to those of AMCR-33. Alloy C shows less good results for both the elastic and the fatigue limits.
The ratio of the fatigue limit divided by the elastic limit is very near to 1 for alloy C and E, while for alloy A it is 1.25. Thus in the external border zone the alternating bending stresses will cause plastic deformation and work hardening, especially for alloy IF-A. This was confirmed by microhardness testing of the cross-section at the minimum specimen diameter of 6mm. The figure above shows for the alloy-A specimen G3 - which did not rupture after 22.886.000 cycles at a maximum bending stress of 360 MPa - the mean of three Vickers indentations.

The indentations were made with a load of 500 gr. at a distance of 0.2mm from the border to the center. The figure shows a central plateau with a hardness Hv of about 210 Kg/mm^2, corresponding to the central inner zone diameter of about 3 mm.
This hardness level is the same for the non-fatigued alloy A. Therefore it is clear that the inner core zone was only deformed elastically. On the contrary, in the outer boundary zone between 1.5 mm and 0.3 mm from the border, the hardness varies up to 50%, indicating plastic deformation and work hardening. This result is in contrast with the usual calculation of the maximum bending stress, based on pure elastic theory.

Advanced low activation materials

Recent activation calculations have shown that SiC appears to be a candidate material which can satisfy the criteria for low activation materials relating to safety and maintenance of fusion reactors.
However, only fibre reinforced SiC could in addition satisfy the structural requirements for the first wall of a fusion reactor. We have therefore started investigation of the advantages of SiC/SiC as a fusion reactor structural material.
They are:
- rapid decrease of induced activity after reactor shut-down
- low afterheat in the case of a loss of coolant accident
- high operating temperature possible
- low-Z material in first wall applications
- low chemical reactivity
- very abundant elements.

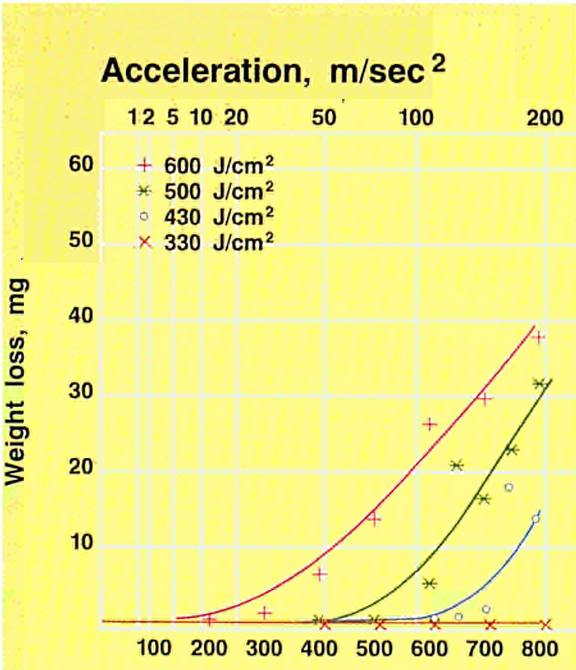
Like all low-Z materials, SiC will produce substantial quantities of He under fast neutron irradiation.

Therefore, the first experiments will investigate the effects of He implantation on the stability and mechanical properties of SiC/SiC composites.
During 1990 preparation of these experiments and the testing equipment has commenced.

Disruption Studies

During disruptions,surface melting will occur in metallic first walls of NET. Due to the contemporary eddy-currents in the metal and liquid layer, magnetic body forces act on both. Equipment has been installed which allows generation of body forces in the liquid. The weight loss on specimens subjected to disruptions and variable inertial forces has been determined (Figure below). It appears that the weight loss increases suddenly at a force density which depends on the power and the discharge time.

Below: *Loss of material during a disruption in dependence of the acceleration for different energy densities.*
Material: Stainless steel AISI 316
Body forces perpendicular to surface
Disruption time 10 ms
Spot diameter 5 mm



Ceramic coatings for enhancing thermal emmissivity

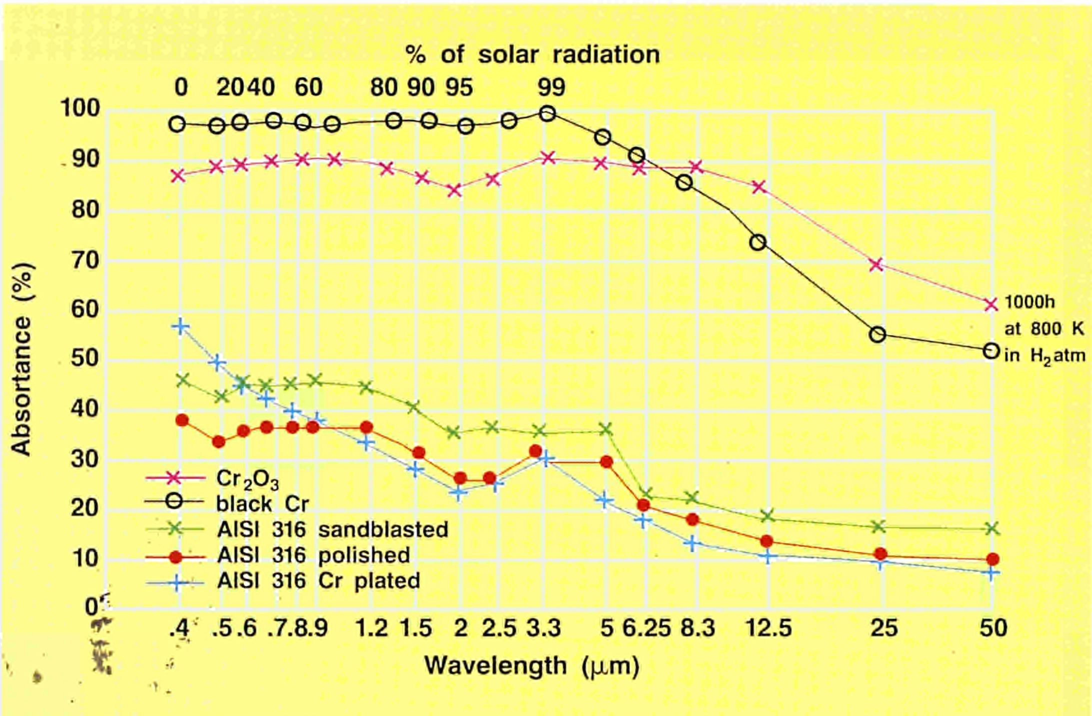
Selective absorbers have been proposed as a means to increase radiative heat transfer in the NET project.

The aim of the present study is the production of films having low emissivity for wavelengths < 4 μm and higher emissivity at higher wavelengths. Considering the operational environment, refractory and stable oxides are taken into consideration (Cr_2O_3, MnO_2, TiO_2 and Al_2O_3). During the last year coatings have been produced by galvanic and plasma spray techniques.

Black Cr has been deposited galvanically on AISI 316. Films 2-5 μm thick have been tested for chemical composition, thermal stability, hardness, adherence and spectral absorptivity. Ceramic films (Cr_2O_3, TiO_2 and Al_2O_3) have been deposited by plasma spray techniques in air or vacuum.

The effect of deposition parameters on the structure, composition and spectral absorption have been studied. Tests of stability under thermal cycling conditions and in H_2 atmosphere are being developed. We are now waiting for components to be coated by galvanic and plasma sprayed films as proposed by the NET Team. The figure below shows the efficiency of the black coatings.

Below: *Hemispherical near normal spectral absorptance of Cr_2O_3 APS, galvanic black Cr, after thermal treatment in H_2 and the effects of surface treatment*



Influence of Pb-17Li on the properties of blanket materials

Production of corrosion barriers

This work is a continuation of the study of the effect of oxygen on the corrosion of structural materials by Pb-17Li. The addition of oxygen to Pb-17Li leads to a short term increase, but a long term decrease in the corrosion rate. This observation can be explained by the formation of $LiCrO_2$ on the surface of the steel.
We have shown that $LiCrO_2$ can also be formed by the reaction of Pb-17Li with Cr_2O_3 formed on steels by pre-oxidation. We have constructed a special apparatus to pre-oxidize the surface of steels under a controlled H_2/H_2O atmosphere (ratio 1000:1).
The corrosion resistance of steels with pre-oxidized layers of different thicknesses will be determined by testing in a thermal convection loop at 723K for 4000 h.

Mechanical properties

The combined effect of hydrogen and Pb-17Li on the mechanical properties of notched tensile specimens of 1.4914 steel is being tested under constant uniaxial load and under slow strain rate ($2.8\ 10^{-7}\ s^{-1}$). Tests will also be made to determine the susceptibility to hydrogen embrittlement of welded specimens of 1.4914. The welded material will be provided by KfK within the framework of the European Fusion Programme. The effect of a constant uniaxial load on the corrosion rate of AMCR 0033 and 1.4914 has been investigated using three thermal convection loops. The loop containing the AMCR specimens has completed 20,000 h and the experiment has been stopped. The other two loops containing 1.4914 specimens have reached 17,500 h. Analysis of the AMCR specimens has now started.

The lithium-lead-bismuth system

As bismuth is a neutron activation product of lead, it is important to know the interaction of bismuth with lithium-lead alloys. A resistivity monitor, developed in collaboration with the University of Nottingham (U.K.) is being used to study the solubility of bismuth and to investigate the ternary lithium-lead-bismuth phase diagram.

The migration of deuterium in the presence of helium in AISI 316

The migration behaviour of D_2 in steel is of interest for estimating the D_2-permeation through the first wall of a nuclear fusion reactor. In order to obtain a preliminary picture of the transport process, "mirror grade" surfaces of AISI 316 targets, cleaned by means of standard procedures, have been implanted with He and D_2, to obtain the following types of targets:

a) 2.10^{17} He^+/cm^2, 200 keV, at 200°C
   + 2.10^{17} He^+/cm^2, 100 keV, at 200°C

b) like a) + 6.10^{16} D^+/cm^2, 40 keV, at 100°C

c) 6.10^{16} D^+/cm^2, 40 keV, at 100°C

d) 2.10^{17} He^+/cm^2, 60 keV, at 300-350°C
   + 2.10^{17} He^+/cm^2, 40 keV, at 300-350°C

e) like d) + 6.10^{16} D^+/cm^2, 30 keV, at 300-350°C

f) 6.10^{16} D^+/cm^2, 30 keV, at 300-350°C

The respective concentration profiles of the deuterium and helium for the targets a, b, and c showed that He (maximum penetration depth 800 nm for 200 keV) has migrated beyond a distance of 1400 nm from the surface. In the presence of He, the D_2 was continuously distributed between the surface and the bulk, whereas in its absence it concentrated near the surface of the implanted target. Although the implantation conditions for the targets d, e, and f were different from those of a, b, and c (lower keV, higher temperature) the results are similar: the presence of He enhances the migration of D_2 into the bulk, i.e. increases the permeation of deuterium and tritium through the first wall of a nuclear fusion reactor.

5. Contribution to the Specific Programme:

NUCLEAR FUELS and ACTINIDE RESEARCH

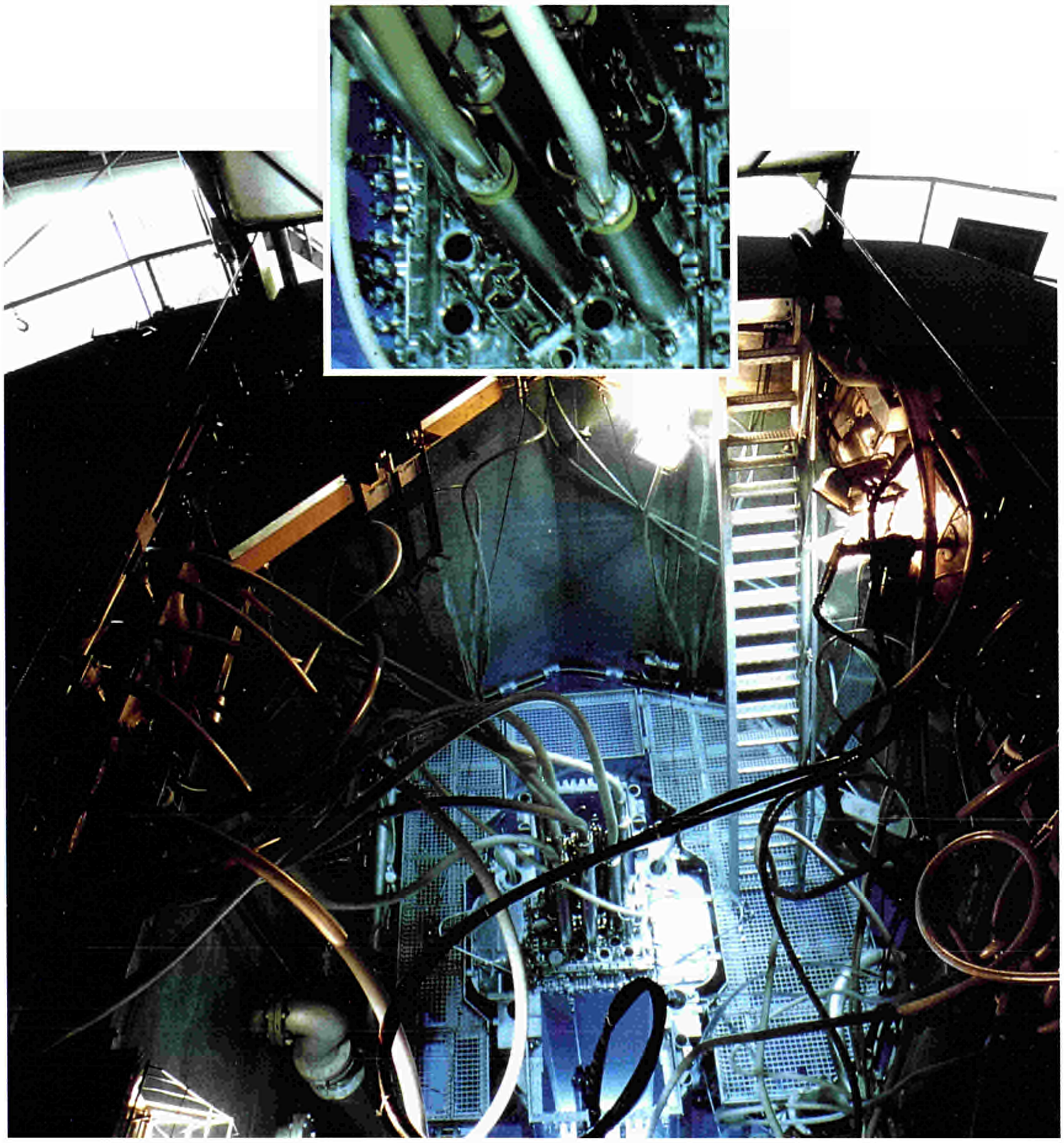
Irradiation Experiments in the High Flux Reactor

In 1987, as part of the programme on the development and improvement of Advanced Fast Breeder Reactor fuel, a series of experiments were sponsored by the Transuranium Institute of the Joint Research Centre in Karlsruhe. In the pursuing two years, the irradiation experiments BUMMEL (study of fission gas bubble mobility in UO fuel) and the two experiments NILOC 1 and 2 (consisting of 6 fresh, mixed nitride fuel pins) were completed.

In 1989, an extension of the NILOC series, ie. NILOC 3 and 4, was proposed. During 1989 and 1990, the 6 irradiation capsules were manufactured at Petten. Due to fabrication problems of the complex mixed nitride fuel at Karlsruhe, the transport of the fuel pins to Petten and the irradiation were postponed to 1991.

Similarly, the experiment POMPEI (study of mixed nitride fuel at high burn-up), also ordered in 1987, is delayed until 1991. The manufacture of the irradiation capsule and its component parts was completed during 1990.

Below: *View into the reactor pool*



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 managed via Forschungszentrum Jülich (KFA) and Kernforschungszentrum Karlsruhe (KfK). It is generally related to the German nuclear energy programmes.

Irradiation programmes related to light water reactors addressed studies of the behaviour under transient conditions of fuel rod segments which have been pre-irradiated in commercial power reactors. In 1990 for the first time a pre-irradiated fuel rod was re-instrumented with pressure transducers at the Petten hot cells and successfully operated at the HFR in a project for study of fission gas release. Two tests with a newly developed and very complex irradiation device for study of iodine solubility and degassing from a fuel rod after a LOCA scenario were successfully completed with pre-irradiated PWR fuel rods.
Considerable progress was made in the development of a "low power" boiling water fuel capsule. For a performance test programme with MOX fuel the burn-up accumulation irradiation started in 1989 was continued. Feasibility studies were performed for irradiation testing of corrosion samples made of Zr- based alloys in typical water reactor coolant conditions and of CT-samples made from BWR vessel material under BWR irradiation conditions.

Irradiation programmes related to the high temperature gas cooled reactor comprise graphite and fuel irradiations. The "fundamental properties graphite programme" which contributes to the data base on irradiation effects on graphite progressed to schedule. About 10 individual irradiations are under evaluation, in the reactor or under preparation. In the graphite creep programme two irradiations were performed with intermittent measurements of dimensional changes of the samples. A third experiment was started.

The irradiation data of an in-pile experiment on HTGR coated particle fuel with 10 % designed-to-fail particles, carried out in the period 1987 to 1989 were evaluated.

The objectives of this safety related experiment, namely the investigation of fission gas and iodine release behaviour under temperature transients and ramps, and purge gas chemistry, e.g. water vapour up to 10^4 μ atm, were fully achieved. A new series of reference tests of spherical HTR fuel elements for the German HTR programme was started. These tests comprise the simulation of HTR-module and HTR-500 power plant operating conditions. 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 main feature of these experiments is the on-line measurement of short, medium and long lived volatile fission products.

Fast breeder reactor fuel irradiations were continued with an overpower equilibrium test, a short transient test and the start of a second long term irradiation test for further fuel and cladding axial displacement experiments. Further effort was put into the development of future SUPER-KAKADU and HYPER-KAKADU experiments which study fuel performance under power cycling conditions, continued development of the application of noise analysis techniques, and a 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 super conducting magnets, investigation of tritium release kinetics from different ceramic lithium compounds under irradiation, and damage studies on metals and welded metal joints as well as graphite base first wall coating materials and molybdenum and molybdenum alloys to be used as divertor materials.

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

The EFR related programme concentrates on damage studies on structural materials. For crack propagation investigations compact tension specimens of different stainless steels have been irradiated. Another series of irradiations provides samples for post-irradiation creep fatigue testing.

For the fusion programme martensitic steels and vanadium alloys are irradiated at different temperature and fluence levels. A programme to study fracture mechanics properties of irradiated steels at lower temperatures is under preparation. This programme also includes advanced welds, such as electron beam and plasma welds. In collaboration with ECN and other European research centres, irradiation testing of tritium breeding ceramic blanket materials is in progress. The main objective is to obtain data on tritium residence times of a variety of different zirconates, aluminates and silicates at different irradiation temperatures, ^{6}Li burn-ups, and at varying purge gas chemistries.

Nuclear physics experiments at three beam tubes have been re-oriented from fundamental to applied nuclear physics, focused on BNCT, during the transitional year 1990. After completion of the study of the exchange currents in neutron capture by ^{3}He at HB11 (resulting in a PhD-thesis for the Delft Technical University) an epithermal neutron-gamma filter has been installed in preparation of the beam facility for BNCT in collaboration with JRC Petten.

At HB7 pilot experiments on prompt gamma determination of boron concentration have been performed as part of the BNCT programme.
For the nuclear orientation facility at HB2 polarized neutron beam, an adequate applied programme is being searched for.

Five beam tubes - HB1, 2, 4, 5 and 9 - are in permanent use for condensed state physics and materials science applications.
The topics addressed by the present programmes are listed below, 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).

The scattering equipment is continuously modified and upgraded in order to meet the requirements of new fields of application; this year a new spectrometer, solely dedicated to residual stress measurements, was completed and installed.

As a joint effort between ECN and JRC, the Petten Neutron Radiography Services (PNRS) provided neutron radiography inspections to materials research, space- and aircraft industry and research. It started a research contract related to the study on the application of neutron radiography in space component technology and continued promotional activities for more applications of this NDE-technique.

The main source of funding for the HFR operation including maintenance and upgrading is borne by the HFR supplementary programme. More detailed information on the HFR programme and its results can be found in the Annual Report, ref. /1/.

Reference

/1/ Annual Report 1990,
Operation of the High Flux Reactor,
EUR 13590 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 the development 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.

Classification of Advanced Ceramics

Classification of Advanced Ceramics is considered as most important standard to enable international applicability, to provide a basis for unanimity in the transfer of information between researchers, designers, manufacturers and product users. The development of a European (CEN) standard for the "Classification of Advanced Technical Ceramics" will built on concepts presently elaborated by a VAMAS working group. The Institute for Advanced Material organised the VAMAS workshop (June 1990): Classification of Advanced Ceramics - Development of the First International System for Producer and User Industries. The objectives of the workshop were the identification of classification requirements based on national and international perspectives, of critical classification elements and terminology, of major product categories and the construction of a possible skeleton classification scheme. The workshop fully met these objectives. Proceedings are under preparation.

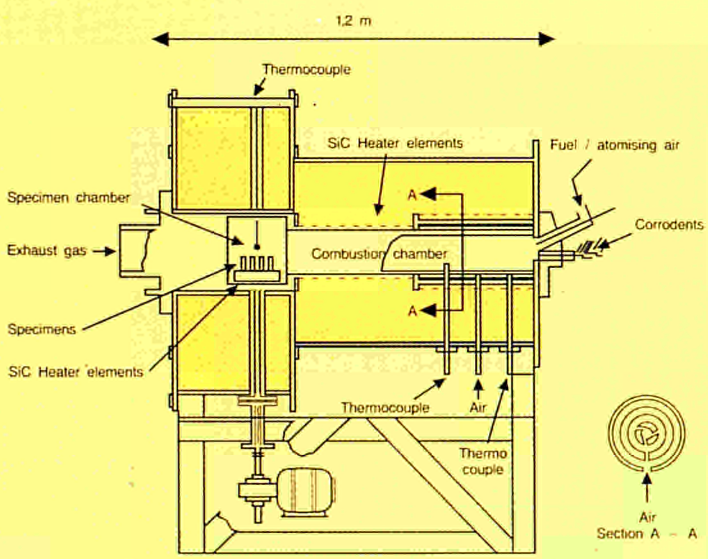
Experimental Activities

In the field of pre-normative R&D activities were focused on three subjects relating to the development of test methodologies, i.e. ceramic corrosion, mechanical properties and non destructive testing.

Ceramics Corrosion

During the year, work on the construction of a new purpose-built "Hot Corrosion Laboratory" has progressed. A burner rig will be housed in this laboratory which will enable the behaviour of advanced engineering alloys, ceramics and composites to be studied under dynamic conditions during exposure to various molten salt/gaseous atmosphere combinations.
A necessary prerequisite before any testing can be done is to establish a standard and reproducible test technique which will ensure that meaningful and industrially-relevant data is generated.
The construction of the laboratory building is completed. The burner rig, illustrated schematically in figure below, is in position and some very preliminary commissioning trials have been initiated.

Below: *Sectional View of the Burner Rig*

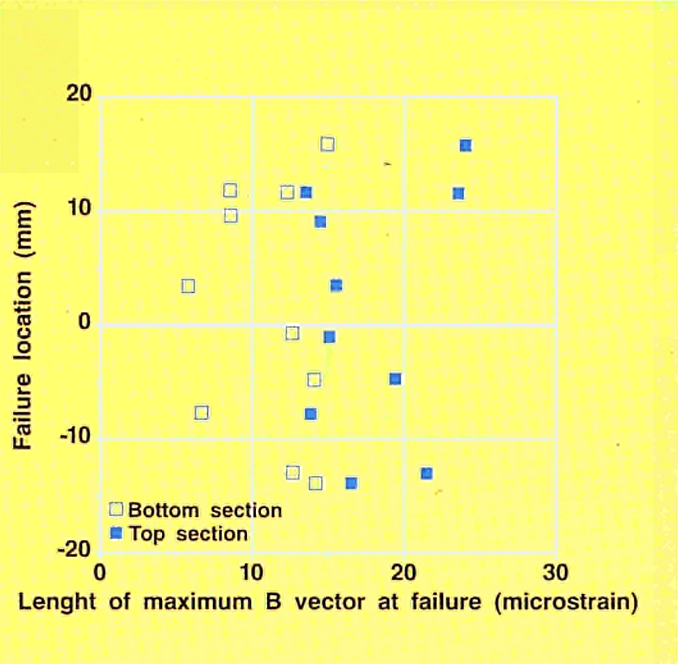


Sectional View of the Burner Rig

Mechanical Properties

Activities during 1990 included:

i. Participation in the VAMAS round robin on fracture toughness determination at room temperature;
ii. The preparation, in collaboration with BCR and NPL (U.K.), of a European round robin on uniaxial tensile and creep testing at high temperature;
iii. The participation in working groups of CEN TC 184 concerning standards for test methods of monolithic and composite ceramics;
iv. The further development and the critical evaluation of two experimental facilities for the accurate uniaxial tensile testing of ceramics. Both systems apply cold gripping. The first system consists of a rigid, very accurately aligned loading train which does not allow for correction after mounting of the specimen. The second system contains a universal coupling, which permits alignment of the specimen by adjustment of the position of precisely machined knife-edge supports. Both systems have been carefully analyzed in terms of their uniaxial alignment capability by means of series of tests on over 70 strain gauged Al_2O_3, ZrO_2 and Si_3N_4 specimens. The maximum bending levels along the gauge length of these samples at failure are smaller than 3,5% corresponding to centre-of-gauge length bending levels (this is the quantity used in literature) of smaller than 1%. The lack of correlations between the failure locations and the lengths of the bending vector at failure which is observed in figure below, proves that the quality of alignment is high enough in order not to obscure the intrinsic uniaxial material behaviour.



Below: *Failure location versus maximum B vector at failure using the tensile setup.*

Ultrasonic Non Destructive Characterization of Cracks in Ceramics

Ultrasound is one of the most promising techniques for the nondestructive testing of engineering ceramics, especially for the characterization of cracks.
In 1990, a work was initiated on the detection and sizing of radial cracks resulting from Vickers indentations in Reaction Bonded Silicon Nitride (RBSN) by means of ultrasonics at relatively low frequencies (30 MHz). The crack lengths varied between 100 µm and 600 µm. Bulk shear waves and surface waves were used to obtain the C-scans. The interpretation of the images obtained was complicated by the presence of the indentation. The methods to size the radial cracks were used: the -6 dB amplitude drop and the relationship between maximum amplitude of the echo and the reflective crack surface; the results were compared with the size measured by optical microscopy.

Standardization of Quality Control Protocols for Cyclotron Produced Radio-Pharmaceutical

This report refers to the Supporting Activities for the practical application of the European Council directives for radiation protection of persons undergoing medical examination and treatment.
Principal aim of this action is to assist DG XI, proposing prenormative protocols for chemical, radiochemical, radionuclidic and biological purity of Radio-Pharmaceuticals.
In 1990, activities were developed according to the following lines:

Production of Radioisotopes:

The yield of nuclear reactions at well definite beam energy, in order to avoid unnecessary and/or dangerous contaminants was studied. The energy calibration of the extracted beam was completed for proton beam at different energies.

Radioisotopes 68-Ge/68-Ga and 73,75 Se:

Ga-radioisotopes are frequently used in nuclear medicine. Ten samples of metallic gallium were irradiated to produce 68-Ge with different activities. The radioisotope 68-Ge decays to 68-Ga with a half life of 288 days.

Studies and experimental work of radiochemical separation have been carried out, especially in view to reduce the risk of contamination. Studies for a remote controlled radiochemical separation of Ge-Ga have been completed. Since the generator 68-Ge/68-Ga is of special interest in nuclear medicine, a generator system has been developed and completed. The first two prototypes have been loaded with 550 KBq and 3.7 MBq, respectively, in order to study loading and elution methods.

These radioisotopes are of particular interest for the application in the BORON NEUTRON CAPTURE THERAPY. Extensive studies have shown that the production of 73,75-Se can be achieved through the bombardment of intermetallic Cu-As alloy. First irradiations and radiochemical separation have been performed.

A Round Table on "Methods for the Standardization of Quality Control Protocols for Cyclotron Produced Radio-Pharmaceuticals", sponsored by DG XI, with experts in radioisotope production, radiochemical separation and labelling is under preparation. It will take place in April 1991.

Materials Information Management

The work during the year has been directed to two main goals: to digest the lessons of the Materials Database Demonstrator Programme; to formulate new proposals for the DG XIII to adopt in order to improve the market for information on engineering materials in the EC. To achieve these aims it was necessary to also take into account rapid developments which are taking place in this field outside the EC and to develop new directions for the Institute to take which would give it a strong role in this field in the future.

During the first half of the year the Proceedings of the Concluding Workshop of the Demonstrator Programme were edited and prepared for publication in camera-ready form. The Proceedings were published in 1990, entitled: "Materials Information for the European Communities". These are now being widely distributed.

From the experience of the Demonstrator Programme a new basis for further actions has been developed in close consultation and cooperation with DG XIII/B in Luxembourg. These proposals are currently being discussed as part of Action Line 4.1 of the more general proposals for a new five-year programme: IMPACT 2. The proposals are in two parts: some direct actions which the CEC would carry out to provide some centralised services and developments of common benefit; some co-operative actions which would involve industrial companies, research and trade associations, scientific institutes and local user support agencies. The whole proposal has the general aims:
- to create an industrial structure for the materials information sector of the European information industry;
- to make European information of industrially strategic importance more accessible to more sectors of the manufacturing economy and the research community;
- to establish a European identity for that information by improved standardization and harmonisation and so increase the impact of European information on the world market.

As result of the studies and discussions on which the new programme would be based a new direction and role for the Institute has been suggested. The new task would be to apply engineering methods and modern computer techniques to the efficient and economically valuable management of scientific and technical information. This new field of Information Management is currently receiving a great deal of attention elsewhere. It would be both within the interests of the Institute and would also support other actions of the CEC as outlined in the new proposals above.

In support of the increasing recognition of the need for international standardisation as a necessary requirement for the development of a market in computerised materials information and for its efficient management there has been participation in some of the actions currently in progress.

These actions have been:
- Participation in a round-robin test of curve fitting procedures for creep data. The tests were organised under the auspices of VAMAS and supervised by the National Research Institute for Materials (NRIM) of Japan.
- Participation in the first trials of a simple protocol for the electronic interchange (EDI) of materials data supervised by the American Society for Testing and Materials (ASTM).
- Strong contributions to the development of a materials model which forms part of the STEP programme of ISO (International Standards Organisation).
  The aims of STEP are to achieve a standardised way of interchanging data on manufactured products by electronic means and the development has important consequences for European industry and for the research community.
  The contributions were made through participation in meetings of an advisory panel of the British Standards Institute, which provides the secretariat for the European contribution to this international effort, and by attendance at official meetings of ISO.

Valorisation of Research Results

Spontaneous Downward Heat Transport

The campaign of comparison tests between the spontaneous system with the new double-effect valve and the forced circulation system has been completed and reported.

The performance of the prototype of the solar system conceived for snow melting and hot water production at a high mountain refuge (3650 m) has been monitored during the whole year. Satisfactory results were obtained, also under the severest conditions it exhibited 100% reliability. A new plant completely assembled in the factory, avoiding the necessity of field plumbing work, has been installed at another mountain refuge at 2100 m and monitored. A proposal for other application of solar heat, again in the mountain environment, is under evaluation.

The first licensing contract for the commercialization of our system in Portugal was signed by a Portuguese firm for the production of hot water in domestic applications. Assistance for the construction of the first plant was given.

A full size working plant was exhibited at the CLIMA Fair in Athens, to stimulate interest among the Greek solar industries.
Contact has been established with the largest solar manufacturer in Greece.

Oxygen Sensors

The development of a prototype oxygen sensor for industrial furnaces started in 1990, in cooperation with an industrial firm and the University of Milan.

The sensor has a continuous measuring capability of oxygen partial pressure both in oxidizing and reducing atmosphere (10^{-30} - 1 atm).
The measuring electrode of this novel sensor is a cermet electrode, covered by a European Patent, which shall be extended to other countries.
The advantage of the sensor is its tolerance for changes in furnace atmosphere, occurring during service or by accident.

Testing experiments of the sensor prototype will continue in 1991.

8. Exploratory Research

Boron Neutron Capture Therapy (BNCT)

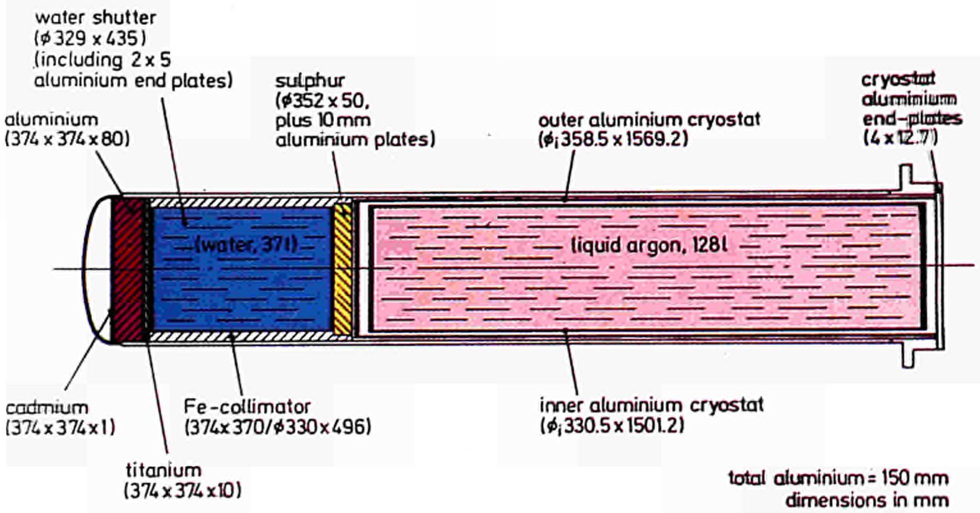
During 1990, the Petten BNCT project has advanced towards its intended goal to treat glioblastoma (brain tumour) patients at the HFR facility.
The important and essential aim to install the necessary components to produce an epithermal neutron beam for BNCT applications was achieved on schedule. The various components that constitute the BNCT facility, ie. filter, emergency and main beam shutters, and shielding, were all installed during the long summer stop of the reactor. The choice of filter components, in both material type and geometric dimensions, was finalised. All components were delivered in time for the installation during the summer shut-down period.

The design of the facility had been jointly carried out by the JRC's at Petten and Ispra, and the UKAEA Harwell. The principle computational tool was the Monte Carlo code, MCNP. The work at Ispra used the deterministic code, DOT, which served as a valuable tool in both supplementing the MCNP results, but more importantly, in validating the results of the MCNP design. The final filter components for the required epithermal neutron beam are : Al, S, Ti, Cd and liquid Argon. The configuration of the filter is shown in the figure below.

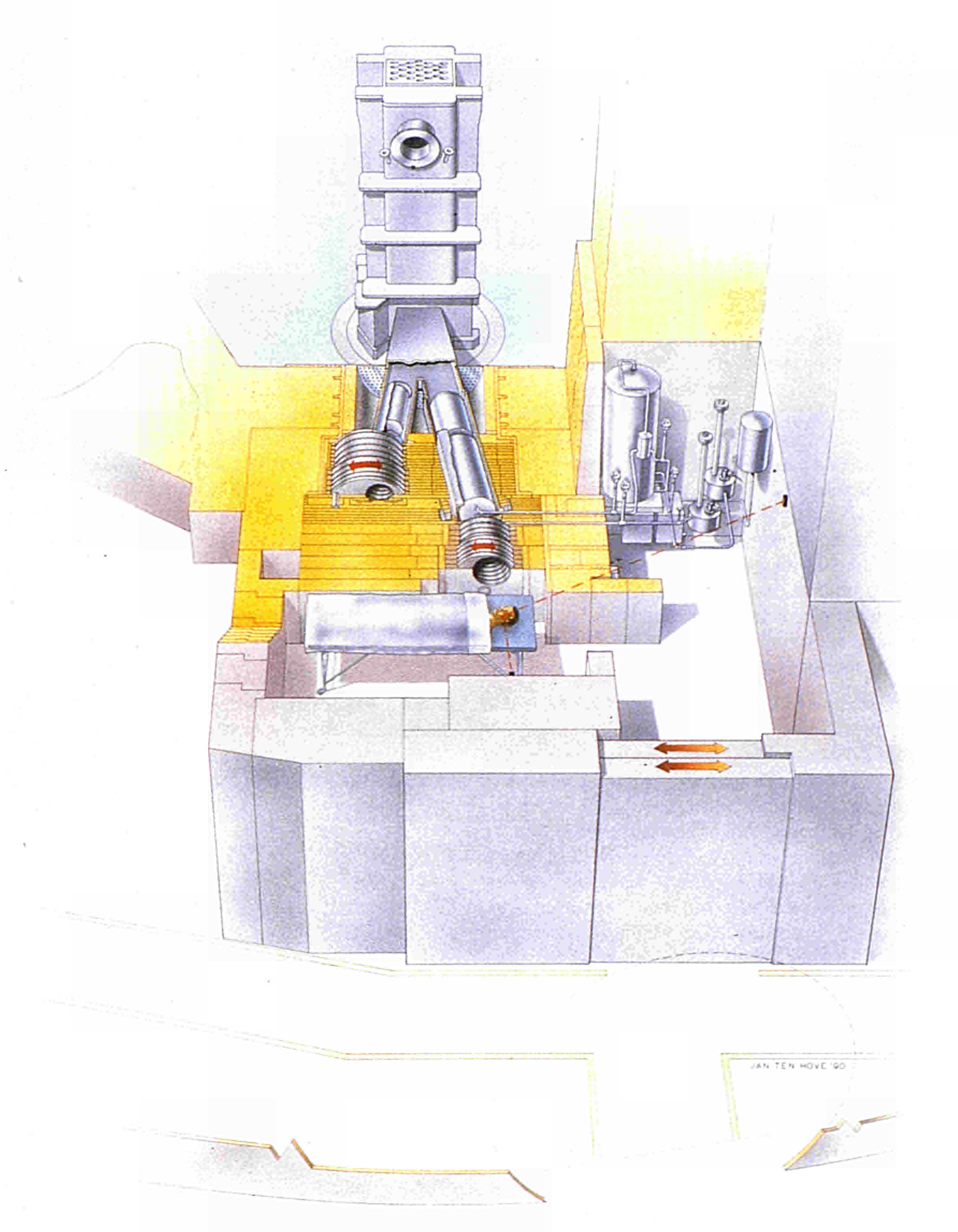
The nuclear characterisation of the beam at full reactor power, and the start of the radiobiological and phantom irradiation experiments planned towards the end of the year have been delayed a few months. This is primarily due to the complexity of the control system to regulate the liquid argon facility, and partly due to the design of the safety logistics for the main and emergency beam shutters. The liquid argon system involves a complex array of cryogenic valves and piping, cryo-coolers and pumps, and an array of feeder pipes for the supply of gas and compressed air to operate an assortment of components.

A provisional set-up was completed in November, when it was possible to open the beam for the first time, albeit at low reactor power (300kW), to perform some nuclear measurements. The results of the exercise are being analyzed.

Below: *Filter configuration for BNCT neutron beam (HB11)*



Petten BNCT Project : The HB11 filter configuration



Further preparatory work in conjunction with our collaborators at ECN Petten and The Netherlands Cancer Institute in Amsterdam, has continued. The techniques of prompt gamma ray spectroscopy, using beam tube HB7 at the HFR, and ICP-AES at the neighbouring ECN laboratory, for determining boron levels in samples has been perfected. A general laboratory at ECN for the preparation and analysis of irradiated radiobiological samples has been jointly set-up.
Phantoms for dose distribution studies, nuclear instrumentation for beam monitoring and dose distribution measurements, and computational tools, including a dedicated workstation for developing a treatment planning program, are now all available. It is expected to complete the set up of an operational beam early in 1991. The first experiments are scheduled for March 1991 and the first clinical trials in Europe on patients in 1992. The envisaged treatment facility is shown in the Figure above.

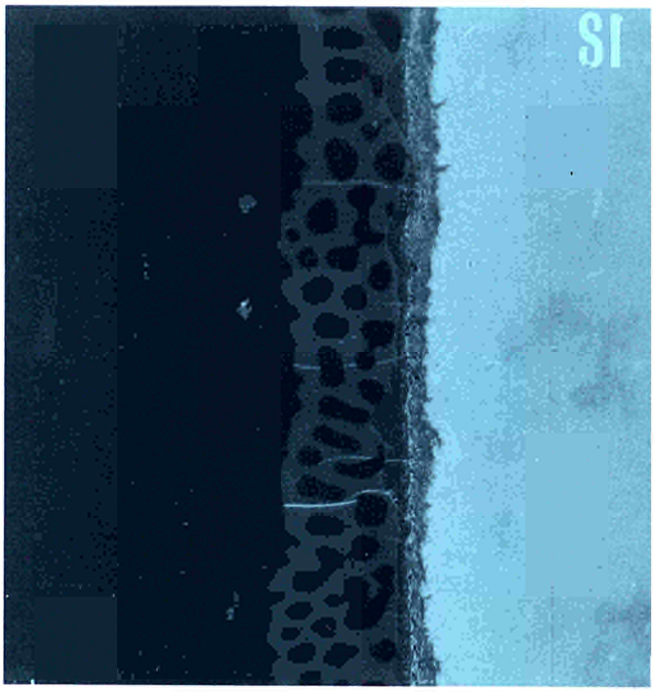
Above: *The Petten BNCT Treatment Facility*

Joining of Ceramics to Metals

Successful exploitation of advanced engineering ceramics such as Si_3N_4 (SN) depends partly on the availability of reliable joining processes.

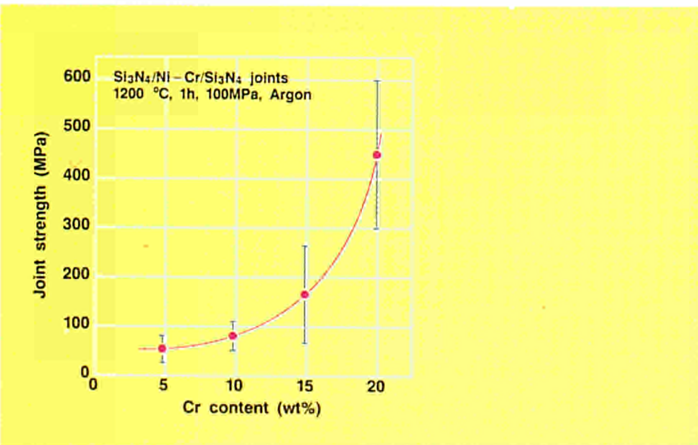
I. Joining of Silicon Nitride Ceramics

An approach that utilizes modification (structural and/or chemical) of the Si_3N_4 surface prior to joining, ductile alloy (Ni-Cr) foils as joining agents, and optimally controlled solid state bonding has resulted in the routine production of strong and reliable joints (400-500 MPa fracture strength and 6-7 Weibull modulus). Having succeeded in producing SN joints that could be of use for a number of structural applications, efforts have been directed towards identifying the materials factors that affect the quality of the bond, thus attempting to correlate workpiece/joining conditions with interface microstructure and joint mechanical properties.
Detailed microstructural analysis has shown that the bridging compounds formed at the interface were mainly Cr-nitrides.

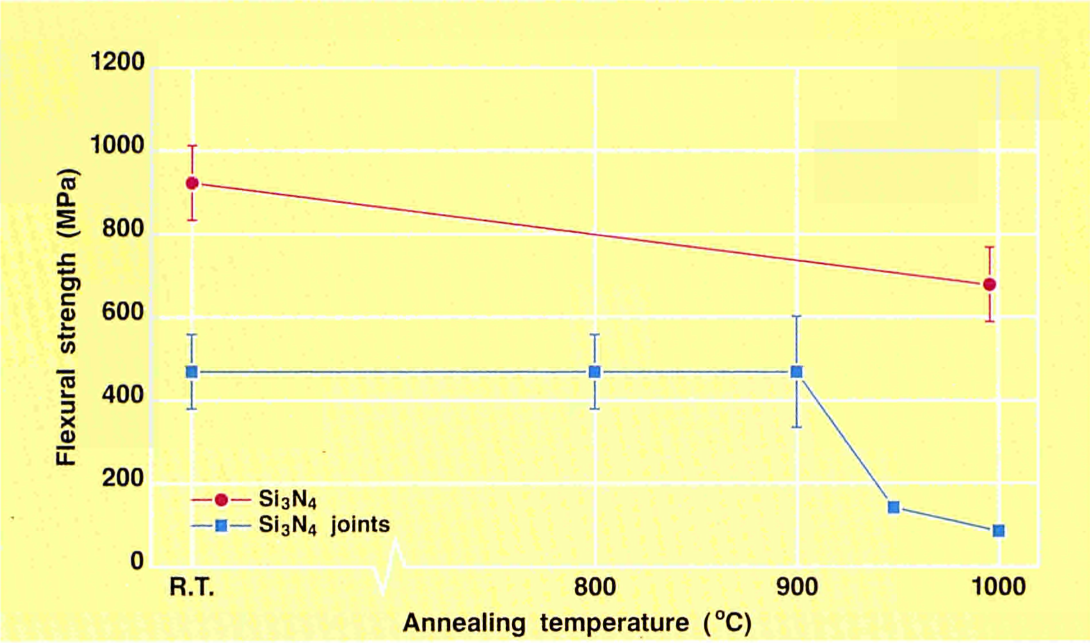


Above: *Optical micrograph of a Si_3N_4/Ni-20%Cr interface bonded in vacuum (marker=20 μm)*

Below: *Fracture strength of SN/Ni-Cr/SN joints as a function of Cr content in the Ni-alloy interlayer*



The reaction proceeds via the dissolution or dissociation of Si_3N_4, the sequential formation of Cr_2N and CrN and, ultimately the formation of silicides.
However, the progress and kinetics of the reaction are strongly influenced by the N_2 and Cr activities in the system during fabrication.
Indeed, changing the environment from Ar to vacuum had a major effect on the interfacial microstructure, as depicted from the Figure above, which was detrimental for strong bonding.
On the other hand, reducing the Cr activity in the Ni-alloy foil also resulted in a weaker joint as shown in the Figure below.



The joint strength was retained upon aging in air for 100 h up to 900°C, as demonstrated in the Figure above, which is clearly of importance for high temperature applications.

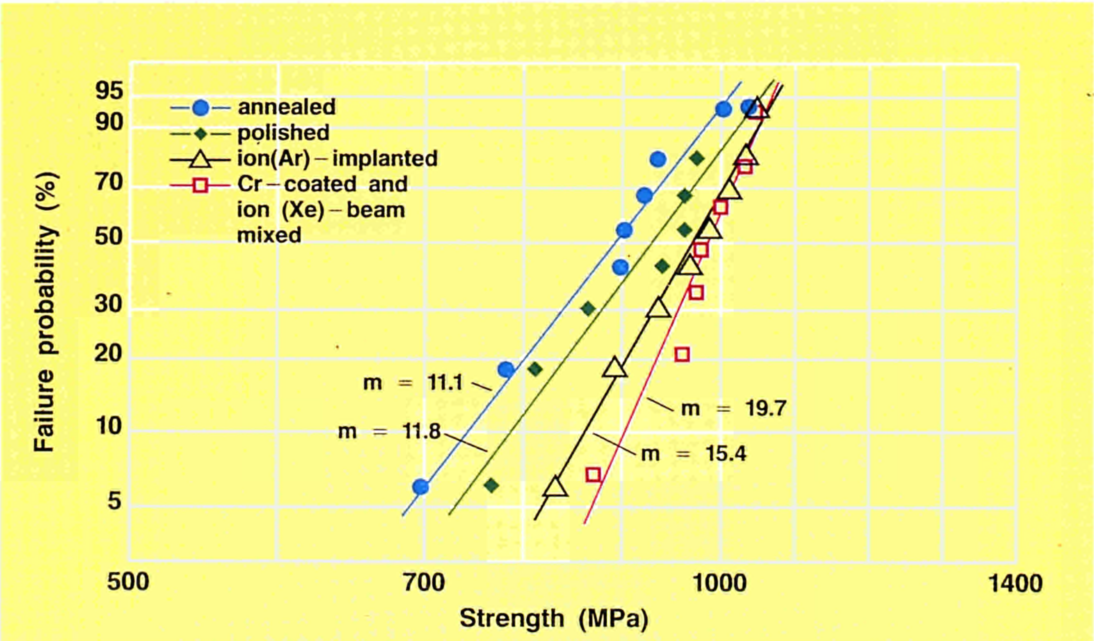
II. Effects of Ion Implantation into SN ceramics andSN/Metal Interfaces.*

It has been demonstrated for a range of ions (Ar, Cr, Si, N) and ion bombardment conditions that ion implantation can improve the surface and bulk mechanical properties of SN, as shown for example in the Figure below.

Ion beam mixing has been observed in the Cr/Si/Si_3N_4 and Si/Si_3N_4 systems, and limited (only of ballistic nature) mixing in the Cr/Si_3N_4 system where ion implantation increased the adhesion of Cr on to Si_3N_4.

Above: *RT flexural strength of Si_3N_4 joints after holding for 100 h in air at the indicated temperatures*

Below: *Fracture strength of ion-beam modified silicon nitride*



* *Work performed jointly with the FOM-Institute (NL).*

Micro-Hydrodynamics of Laser Melted Pools

Local erosion of solids in contact with gas/liquid or with liquid/liquid interfaces is a well known phenomenon.
There are many practical situations, in chemical engineering, and in glass and steel making technologies where two or more fluid phases are in contact at interfaces.

It has long been suggested that interfacial tension flows (Marangoni flows) might play a key role in determining the erosion patterns at the walls of pyrometallurgical furnaces.

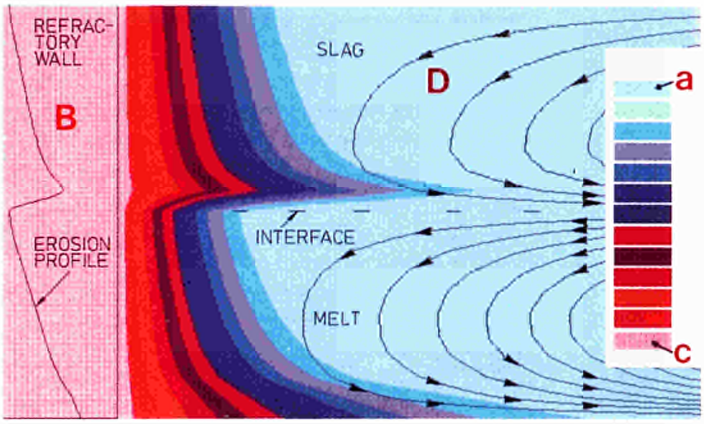
A mathematical model has been developed for this mechanism on the assumption that the wall erosion is caused purely by buoyancy and Marangoni convective flows.

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.
It is therefore of interest to examine the behaviour of systems where the flow field is controlled by Marangoni interfacial flows and where buoyancy flows play a secondary role.

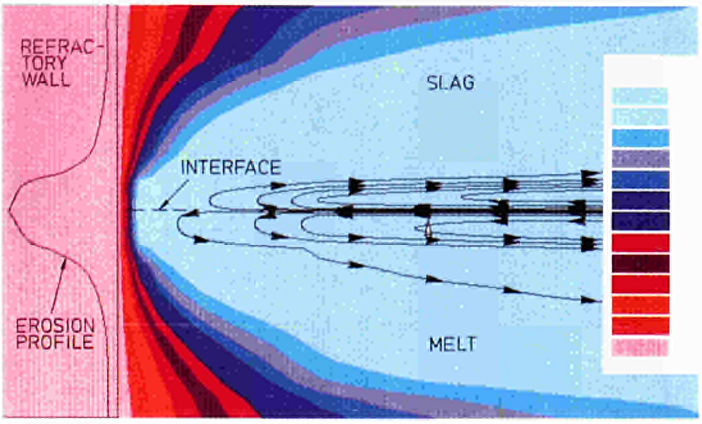
These conditions are depicted in the Figure below. The flow pattern in the slag has been completely reversed and the corresponding erosion profile is more symmetrical around the interface.

Profiles of this form are found in practice.
In conclusion, the computations suggest that optimization of process parameters such as process input, wall temperature gradient and wall material properties could result in reduced erosion rates.



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 konvective 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

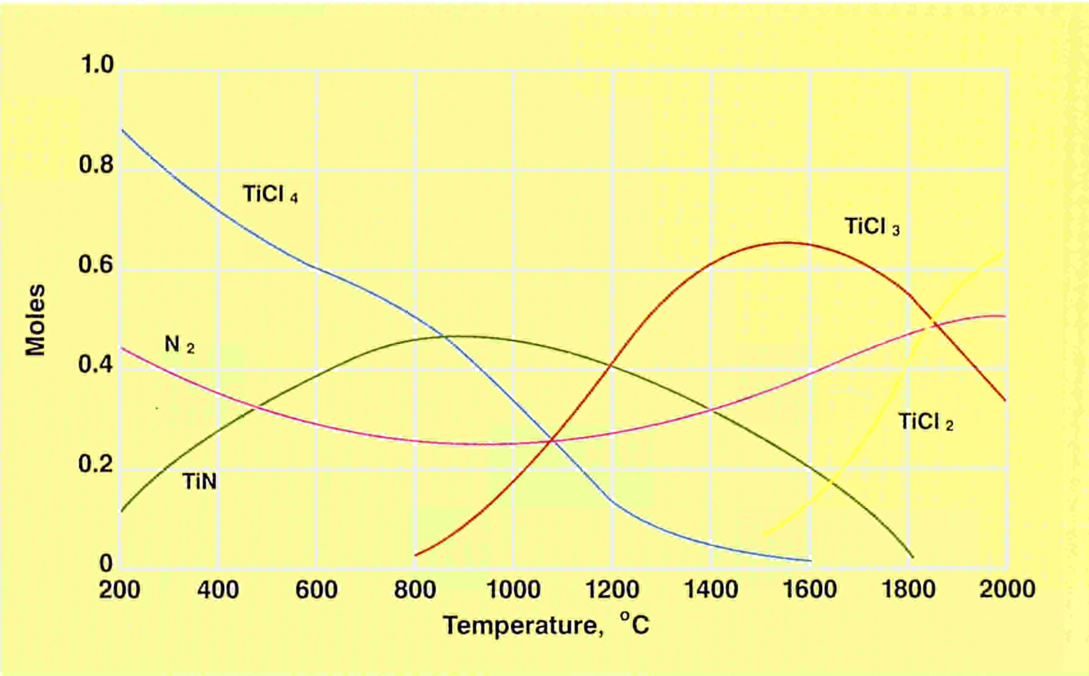
Intelligent processing is a new development in process control. Computers are used to model the response of material properties to changes in process variables, to react to information from sensors which monitor the evolution of materials properties during processing, and to send signals to actuators to control process variables.
The idea is to actively steer the process towards a goal defined in terms of material properties.
The benefits are improved process efficiency and better product quality.

The objectives of the project are: to study the application of "intelligent processing" to chemical vapour deposition (CVD) for the purpose of improving coating properties by reducing grain size; to identify the control strategies necessary to achieve this aim; to identify areas where developments are necessary to implement this aim; to establish an experimental facility to test control systems; to identify other materials processing technologies capable of benefiting from intelligent control.
The achievements in 1990 were:

a) Thermodynamic modelling

Thermodynamics determines the window of operating conditions for the CVD process. It also offers insight into the process. The formation of TiN from $TiCl_4$, H_2, and N_2 was investigated.
The findings were:
1) a broad temperature window for the reaction exists centred on 900^0C, see Figure below;
2) the equilibrium yield depends only weakly on gas pressure (thus pressure can be varied to optimize kinetics);
3) the role of hydrogen is to produce $TiCl_3$ without which TiN formation cannot occur so the process should be run with an excess of H_2.

Below: *Multiphase equilibrium for the production of TiN by CVD at 0.01 bar*



b) Intelligent control

A simple empirical model will be used initially. Later it will be progressively upgraded to contain more rigorous physical descriptions which will enable the model to be used for optimizing the process. An additional level of software is required so that external priorities such as minimizing time, cost etc. are also respected by the model. The model is thus regarded as a core of functional dependencies that operates within a temporary set of boundary conditions. Development is needed on sensors and on kinetic models. Sensors will remain the limiting factor in the application of intelligent control to CVD.

c) Identification of other processes suitable for intelligent control

The synthesis of ceramic powders by precipitation as colloids from solution with further processing by slip-casting to monolithic or composite materials was identified as a suitable process for the application of intelligent control. An activity in this area could also complement the exploratory research project on ceramic fibre composites for which slip casting is being considered for infiltrating fibres.

d) Experimental facility

A computer system was acquired for modelling, data acquisition and process control.
Linear displacement transducers were purchased as sensors with the intention of using a simple example of the spring-back problem in the bending of sheet metal as a test of the computer system. Laboratory space became available to the project during the year.
The project will ultimately use CVD equipment currently being installed at Petten.

Cold Fusion

Research effort in 1990 concentrated on dry cold fusion and in particular on the study of the effect of deuteration of metal shavings.

The effects of deuteration have been examined not only on shavings of titanium but also of zirconium, hafnium and Ti-Zr alloy. Tritium production has been evaluated making by a balance between the tritium content of the deuterium gas and the final tritium present in the metal. An active collaboration, on tritium analysis has been set up with ENEA Frascati. Neutrons emission has been studied using a ^{3}He neutron counter.

A statistical analysis on the tests performed did not reveal a significant neutron emission. Tritium production depends on the metals used in the deuteration and appears to be significant in the case of the Ti-Zr alloy. The results obtained are summarized in the table above.

System	Mean Value	st. deviation	N⁰ of tests
Deuterium gas	10.59	0.051	36
Titanium	10.66	0.121	7
Zirconium	11.51	0.091	12
Hafnium	11.03	0.132	6
Ti-Zr 50%at.	12.02	0.065	14
All metals	11.61	0.045	39

Table above: *Statistical evaluation of Tritium content*

Ⅲ List of Publications

Scientific -Technical Articles and Monographs

J.N. Adami, D. Bolsch, J. Bressers, E. Fenske, M. Steen;
Uniaxial Tension and Cyclic Tension/Compression Testing of Ceramics. Journal of the European Ceramic Society (publication in Feb. 1991)
ART 29588

J. Ahlf, J. Schinkel;
Upgrading and Modernisation of the High Flux Reactor Petten.
Nuclear Engineering and Design (to be published)
ART 29593

J. Barton, S. Fuijne, K. Kanda, G. Matsumoto (Eds.);
Proceedings of the Third World Conference on Neutron Radiography.
ISBN 0-7923-0832-8, July 1990,
EUR 12876

J. Bressers, W. Hessler, U.W. Hildebrandt, H. Willems;
NDT assessment of the residual life in fatigue damaged Alloy 800H.
High Temperature Materials for Power Engineering (1990). Kluwer Academic Publishers, p.629

J. Bressers, M. Steen;
Fatigue and Microstructure in Austenitic High Temperature Alloys.
Int. Journal of Pressure Vessels and Piping (to be published)
ART 29132

R. Conrad;
Irradiation Experiments on Liquid Tritium Breeding Material. Pb-17Li in the HFR Petten.
Fusion Engineering and Design, 13 (1990)
ART 29389

R. Conrad, L. Debarberis;
Irradiation of Liquid Breeder Material Pb-17Li with in-situ Tritium. Release measurements in the LIBRETTO-2 Experiment.
Journal of Nuclear Materials 176, 1990
(to be published)

R. Conrad, L. Debarberis;
Irradiation Facilities for Testing Solid and Liquid Blanket, Breeder Materials with in-situ Tritium Release Measurements in the HFR Petten.
Journal of Nuclear Materials 176 (1990)

S. Crutzen, R. Nichols, K. Kussmaul, Y. Yagawa;
Development in Fracture Mechanics and NDE.
SMIRT 10, Los Angeles, 13-18 August 1989.
Nuclear Engineering and Design (1990)

G. De Beni, R. Friesen;
Impianto Solare Termico Innovativo per i Rifugi Montani. Energie Alternative HTE (to be published)

X. Edelmann, P. Jehenson, M. Gambini, S. Crutzen;
Investigation on Field Removed Pipe Sections in the PISC Hot Laboratories.
CEC Seminar on the Reactor Safety Research, Varese, 20-24 Nov. 1989.
PISC III Report No. 15, EUR 12922 EN (1990)

R.J. Fordham (Ed.);
High Temperature Corrosion of Technical Ceramics. Proceedings of the European Colloquium, Petten 26-28 June 1989
Elsevier Science Publishers.
EUR 13221 EN

W. Gissler, J. Haupt, A. Hoffmann, P.N. Gibson, D.G. Rickerby;
Sputter deposition and characterization of boron nitride films.
Materials and Manufacturing Processes (to be published)

W. Gissler, J. Haupt, A. Hoffman, P.N. Gibson, D.G. Rickerby;
Mixed Phase nanocrystalline boron nitride films: preparation and characterization.
Thin Solid Films (to be published)

V. Guttmann, J. Timm;
On the Influence of the Thermal Pretreatment of Alloy 800H,
Zeitschrift für Metallkunde, H6 (1990) p.p. 428
ART 29104

F. Lanza, R. Feduzi, J. Fuger;
Effects of lithium oxide on the electrical properties of CuO at low temperature.
Journal of Materials Research, MRS, Vol. 5, No. 8, Aug. 1990

J.F.W. Markgraf (Ed.);
Neutron Radiography at the HFR Petten.
Compilation of the HFR Petten Contributions to the Third World Conference on Neutron Radiography and the SITEF Symposium 1989
EUR 12727 EN, March 1990

J.B. Marriott;
Future Materials Requirements for High Temperature Power Engineering Components.
Materials and Design Vol 11, No. 3, June 1990, p.p. 122-128
ART 29498

C. Martins, M. Steen, L. Guerra Rosa;
Theoretical Comparison of Error in KIc-Values Determined by Different Testing Methods of Ceramics.
Journal of Testing and Evaluation (to be published)
ART 29462

S. McAllister, J. Markgraf, D. Kennedy, I. Ruyter;
Development of a two-dimensional Computer Code for the Prediction of Two Fase Heat Transfer in an Experimental Light Water Irradiation Capsule.
Journal of the British Nuclear Energy Society (to be published)
ART 29142

S. McAllister, R.C. Hurst, T.E. Chung;
Modelling the Multiaxial Creep Behaviour of Alloy 800H.
Int. Journal of Pressure Vessels and Piping (to be published)

R.L. Moss, L. Dewit, D. Gabel;
New developments in Neutron Capture Therapy.
European Journal of Cancer, Oct. 1990
ART 29644

R.L. Moss, M. Beers, M. Debarberis, G. Tsotridis;
FBR Fuel Pin Testing at the HFR Petten.
Summary of Current Programme and Future Trends Nuclear Europe (to be published)
ART 29522

P. Nanni, V. Buscaglia, C. Battilana, E. Ruedl;
Air oxidation of a Mn-Cr austenitic steel of potential use for fusion reactor structural applications between 1073 and 1473 K at 10^5 Pa.
Journal of Nuclear Materials (to be published)

J.F. Norton, J.A. Kneeshaw;
The high temperature corrosion behaviour of Fe-Cr-Ni alloys exposed to gaseous oxidising/sulphidising/carburising atmospheres.
Res Mechanica, published by Elsevier Applied Science Publishers (1990)
ART 29191

C. Olagnon, E. Bullock, G. Fantozzi;
Processing of high density sintered SiC whisker re-inforced Si_3N_4 composites.
Ceramics International (to be published)
ART 29463

S.D. Peteves, M. Nakamura;
Joining of Silicon Nitride Ceramics under Nitrogen Gas Pressure.
Journal of Materials Science Letters 8 (1989) p.p. 1228
ART 29129

S.D. Peteves, M. Nakamura;
Solid State Bonding of Silicon Nitride Ceramics with Ni-Cr Alloy Interlayers.
Journal American Ceramic Society 73 (1990) p.p.1221
ART 29130

S.D. Peteves, M. Moulaert, M.G. Nicholas;
Interface Microchemistry of Silicon Nitride/Nickel-Chromium Alloy Joints.
Metallurgical Transactions (to be published)
ART 29773

T. Pradell, C. Olagnon, E. Bullock;
Interphase Stresses in Ceramic Composites.
Journal of Materials Science Letters, 9 (1990) 960 -961
ART 29190

D.G. Rickerby, P.N. Gibson, W. Gissler, J. Haupt;
Structural investigation of reactive sputtered boron nitride films.
Thin Solid Films (to be published)

E. Ruedl, G. Valdrè;
Application of an EM 400T-FEG to some materials problems.
Electron Optics Bulletin, Eindhoven (to be published)

R. Santorelli, J.F. Norton, F. Bregani;
A comparison of the corrosion behaviour of FeCralloy and MA 956 in simulated coal gasification atmospheres.
Werkstoffe und Korrosion, 41, (1990), p.p. 669-677
ART 29526

M. Steen, S. Sinnema, J. Bressers;
Statistical analysis of bend strength data according to different evaluation methods.
Journal of the European Ceramic Society
(to be published)

M. Steen, S. Sinnema, J. Bressers;
Study of the size effect on strength in bend and tensile tests on Al_2O_3 and ZrO_2.
Journal of the European Ceramic Society
(to be published)

M.F. Stroosnijder, V. Guttmann, J.H.W. de Wit;
Corrosion and Creep Behaviour of Alloy 800H in Sulphidising/Oxidising/Carburising Environments at 700^{0}C.
Part I: Corrosion Behaviour in the Stress Free and the Stressed State.
Werkstoffe und Korrosion 41 (1990) p.p. 503-504
ART 29269
Part II: Creep Behaviour.
Werkstoffe und Korrosion, 41 (1990) p.p. 508 - 513
ART 29270

M.F. Stroosnijder, M.J.Bennett, V. Guttmann, J.F. Norton, J.H.W. de Wit;
Influence of Cerium Implantation on the Nucleation and Growth of Corrosion Products on Alloy 800H in a Mixed Sulphidising/Oxidising Environment.
Oxydation of Metals (to be published)
ART 29271

M.F. Stroosnijder, V. Guttmann, J.H.W. de Wit;
Corrosion Behaviour of a 32Ni-27Cr-0,07Ce Steel under simulated Coal Gasification Conditions in stress free and stressed state.
Corrosion Science, 32 (1990), p.p. 151-166
ART 29297

M.F. Stroosnijder, V. Guttmann;
Creep Behaviour of 32Ni-27Cr-0,07Ce Steel in Simulated Coal Gasification Environments.
Steel Research (to be published)
ART 29390

M.F. Stroosnijder, V. Guttmann, H. Buscail;
Effect of Preoxidation and the Influence of Deformation on the Corrosion Behaviour of Two Heat Resistant Steels under Simulated Coal Gasification Conditions.
Werkstoffe und Korrosion (to be published)
ART 29392

M.F. Stroosnijder, M.H. v.d. Voorde;
High Temperature Materials: Their Development and Prospects
Materialwissenschaft und Werkstofftechnik, 21 (1990), p.p. 1-5
ART 29419

S. Yamauchi (Ed.);
Contribution to the Monography: "Chemical Sensor Technology" Vol. IV - "Solid Electrolyte Potentiometric Oxygen Gas Sensors"
"A Chlorine Potentiometric Sensor".
Sensors and Actuators - B (to be published)

A.G. Youtsos, M. Biggio, Y. Crutzen, F. Farfaletti-Casali;
Instability Analysis of First Wall Components of a Tokamak Reactor Subjected to Electromagnetic Transients.
Fusion Engineering and Design, paper NEF 44
(to be published)

A.G. Youtsos, E. Gutiérrez, G. Verzeletti;
Viscoplastic behaviour of stainless steel AISI 316L under cyclic loading conditions.
Acta Mechanica, 84, 109-125 (1990)

Technical EUR Reports

J. Ahlf, A. Gevers (Eds.);
Annual Report 1989,
Operation of the High Flux Reactor.
EUR 12881 EN

D. Bolsch;
Untersuchungen zum Wechselfestigkeitsverhalten von Siliziumnitrid bei hohen Temperaturen.
EUR 13349 DE

M. Cundy, M. Merz (Eds.);
Annual Report 1989,
Institute for Advanced Materials.
EUR 12272 EN

H. Hausen, W. Schüle, M.R. Cundy;
Irradiation creep experiments on fusion reactors candidate structural materials.
EUR 13193 EN

F. Lanza, E. Parnisari, C. Ronsecco;
Tritium analysis in deuterium gas and deuterated metals of the IVb group.
EUR 13367

J.W.F. Markgraf (Ed.);
Neutron Radiography at the HFR Petten.
EUR 12727 EN

C. Olagnon;
Influence des Conditions d'Elaboration sur la Microstructure et ces Proprietes Mechanique du Nitrure de Silicium.
EUR 12760 FR

T. Pradell, P. Glaude, S.D. Peteves, E. Bullock;
The Measurement of Residual Stress in Engineering Ceramics.
EUR 12635 EN

W. Schüle, M. Gieb;
Radiation induced structural changes in alpha-copper-zinc alloys.
EUR (to be published)

N. Swindells, H. Kröckel, N. Waterman (Eds.);
Materials Information for the European Communities.
EUR 13153 EN

Contributions to Conferences

J. Ahlf;
The High Flux Reactor Petten. Present Status and Prospects. 1st Meeting of the International Group on Research Reactors, Oak Ridge, USA,
28 February - 2 March 1990
ORA 35270

J. Ahlf, J. Schinkel;
Upgrading and Modernisation of the High Flux Reactor Petten.
Jahrestagung Kerntechnik, Nürnberg,
15-17 May 1990
ORA 35172

J. Ahlf;
The High Flux Reactor Petten.
Characteristics and Utilization.
XVI Reunion Annual Sociedad Nuclear Española,
Oviedo, 17-19, October 1990
ORA 35590

G. Albertini, F. Rustichelli, G. André, M. Perrin,
R. Matera, P. Miele;
Residue stress in AISI 316L thermally fatigued steel. Conf. Naz. sulle prove non distruttive,
Milano, 28-31 October 1990

G. Albertini, G. André, R. Matera, P. Miele,
M. Merola, M. Perrin, F. Rustichelli;
Deformazioni residue nell'acciaio austenitico AISI 316L sottoposto a cicli di fatica termica.
Convegno Ricerca, Sviluppo e Tecnologie dei Materiali per i reattori a fusione, ENEA, Frascati,
4-6 December 1990

P.G.Avanzini, M. Brossa, U. Guerreschi,
M. Lazzaretti, F. Rosatelli, G. Simbolotti,
V. Zampaglione, V. Vieider, R. Matera;
Technological aspects of the fabrication control and testing of a representative first wall panel for NET. 16th SOFT, London, 3-7 September 1990

D. Besson, B. Michel, J. Archer, B. Libin, R. Matera,
G. Vieider;
NET First Wall Test Section.
16th SOFT, London, 3-7 September 1990

C. Birac, H. Herkenrath, Y. Miyake, G. Maciga;
The Steam Generator Program of the PISC III.
10th International Conference on NDE,
11-14 June 1990, Glasgow, (UK)

J. Bressers, M. Steen, J.N. Adami;
Prestandardization of Aspects of the Uniaxial Mechanical Testing of Ceramics.
Conference on Hamonization of Testing Practice for High Temperature Materials,
Travedona Monate, 18-19 October 1990
ORA 35753

J. Bressers, W. Hessler, U.W. Hildebrandt,
H. Willems;
NDT Assessment of the Residual Life of Fatigue Damaged Alloy 800H.
High Temperature Materials for Power Engineering, Liège, 24-27 September 1990
ORA 35457

F. Brossa, B. Looman, D. Quataert, M. Queriaud;
Ceramic Coatings for Enhancing Thermal Emissivity. 7th CIMTEC World Ceramics Congress,
Montecatini Terme, 24-30 April 1990

F. Brossa;
Giunzioni brasate acciaio-grafite.
Convegno Ricerca, Sviluppo e Tecnologie dei materiali per i Reattori a Fusione, ENEA,
Frascati, 4-6 December 1990

V. Coen, T. Sample;
Pb-17Li: A fully characterised liquid breeder.
16th Symposium on Fusion Tecnology (SOFT),
London, 3-7 September 1990

V. Coen;
L'eutettico piombo-litio, "Pb-17Li":
una scelta Europea quale mantello, liquido, generatore di trizio.
Convegno Ricerca, Sviluppo e Technologie dei Materiali per i reattori a fusione, ENEA, Frascati,
4-6 December 1990
(Invited paper)

F. Costa Oliveira, R.A.H. Edwards, R.J. Fordham;
Effect of Temperature and Gas Composition on the Corrosion Behaviour of Silicon Nitrides Exposed to Sulphidizing Environments of Low-Oxygen Potential.
Workshop High Temperature Corrosion of Advanced Materials,
Frankfurt/Main, 5-6 February 1990
ORA 35267

S. Crutzen, H. Herkenrath, K. Kussmaul, U. Mletzko;
Nondestructive Testing at MPA Full Scale Vessel within PISC III (Status Report and First Results).
16th MPA Seminar, 4-5 October 1990, Stuttgart (D)

S. Crutzen, H. Herkenrath, G. Deuster;
The Importance of Residual Stresses in the PISC Programme. IAEA Specialists Meeting on "Residual Stresses in Structural Materials and Components of Nuclear Power Plants",
17-19 October 1990, Buenos Aires, (Argentina)

S. Crutzen, P. Jehenson;
Inspection Performance with a View to Pressure Vessel Life Management. CSNI Workshop on Pressure Vessel Safety Assessment, VTT, Helsinki, Preprint, 8-9 October 1990

S. Crutzen, P. Jehenson, R. Nichols, N. McDonald;
PISC: Status of the Programme and Evoluation in the Future 5 years.
10th International Conference on NDE,
11-14 June 1990, Glasgow (UK)

S. Crutzen, R. Murgatroyd, H. Seed,
R. van den Bergh;
Human Factor Studies in PISC III.
10th International Conference on NDE,
11-14 June 1990, Glasgow (UK)

F. D'Amato, A. De Ninno, F. Scaramuzzi, F. Lanza;
Search for nuclear phenomena by the interaction between titanium and deuterium.
1st International Conference on Cold Fusion,
Utah Univ. Utah, April 1990

G. De Beni, R. Friesen;
Trasporto spontaneo di calore verso il Basso
- Applicazione ai sistemi solari.
Manifestazione "Sistemi impiantistici per il risparmio energetico" Abano Terme, 21 June 1990

M.I. de Vries, M.R. Cundy;
Effects of 22 Years Neutron Exposure on the Mechanical Properties of a 5154-0 Type Aluminium Alloy.
15th Symposium on Effects of Irradiation,
Nashville, 17-20 June 1990
ORA 35412

L. Dewit, B. Mijnheer, R.L. Moss, D. Gabel;
A Proposal for Clinical Pilot Studies for BNCT.
4th International Symposium on Neutron Capture Therapy for Cancer, Sydney, Australia,
4-7 December 1990

S. Doctor, H. Herkenrath;
Austenitic Steel Piping Testing Assemblies in PISC III.
10th International Conference on NDE,
11-14 June 1990, Glasgow,(UK)

B. Fisher, J. Markgraf, S. McAllister, D. Perry†,
K. Reickardt, I. Ruyter;
The Power Ramp Testing of LWR Fuel Rods at the High Flux Reactor Petten.
Jahrestagung Kerntechnik 1990, Nürnberg,
15-17 May 1990
ORA 35191

R.J. Fordham, J.F. Norton, S. Canetoli, M. Spreij;
Hot Stage Microscope Studies of the Oxidation Behaviour of a Silicon Nitride Ceramic.
Workshop High Temperature Corrosion of Advanced Materials,
Frankfurt/Main, 5-6 February 1990
ORA 35266

J.L. Gonzalez, H. Fattori, V. Guttmann;
Void Formation during High Temperature Oxidation of ODS Alloys MA 6000 and MA 760.
7th Congresso Nacional de Ciencia y Technologia Metallurgicas, Madrid, 3-5 October 1990
ORA 35695

W. Gissler, J. Haupt, T.A. Crabb, P.N. Gibson,
D.G. Rickerby;
Evidence for mixed phase nanocrystalline boron nitride films.
Second International Conference on Surface and Plasma Engineering, Garmisch,
10-14 September 1990,
In press in: Surface and Science Technology

S.F. Garibba, A.C. Lucia, A. Servida;
Multi-expert knowledge acquisition and uncertainty handling in a decision support system for structural reliability assessment.
Conference: A quarter Century of Fuzzinen NAFIPS 90, Toronto, Canada, 5-8 June 1990

V. Guttmann, H. Schönherr;
Improved Creep Strength and Ductility for HP40Nb Steel by Preannealing Rupture Ductibility of Creep Resistant Steels.
Workshop: Rupture Ductibility of Creep Resistant Steels.
York, UK, 4-5 December 1990,
ORA 35883

V. Guttmann;
Important Features for High Temperature Creep Investigations in Corrosive Atmospheres.
The Practicalities of Mechanical Testing at Elevated Temperatures in Controlled Environments.
ERA Workshop: The Practicalities of Mechanical Testing at Elevated Temperature, Surrey (UK),
7 December 1990,
ORA 35884

V. Guttmann;
Untersuchungen zum Einfluss von Unterbrechungen beim Zeitstandversuch
13. Vortragsveranstaltung VDE, Düsseldorf,
30 November 1990

V. Guttmann, M.F. Stroosnijder, H. Buscail;
Some Aspects of High Temperature Corrosion under Stress.
Workshop High Temperature Corrosion of Advanced Materials,
Frankfurt/Main, 5-6 February 1990,
ORA 35287

H. Hausen, W. Schüle, M.R. Cundy;
Irradiation creep experiments on fusion reactor candidate structural materials.
Conference The Effect of Irradiation on Materials of Fusion Reactors, Leningrad, USSR,
September 1990

E.D. Hondros, M. Merz;
Materials Measurements - A European Prospective.
Conference: Harmonisation of Testing Practice for High Temperature Materials, Travedona Monate,
18-19 October 1990

E.D. Hondros, S.D. Peteves;
Interface Microchemical Aspects of Ceramic Joining, Proceedings Japan Fine Ceramics Workshop: New Processing and Properties.
K. Funatani (Ed.), JFCC, Nagoya, Japan,
15-16 March 1990

R. Huiskamp, A.C. Begg, V.G.A. Gregoire, D. Gabel, A. Siefert, R.L. Moss;
Radiobiology Studies at Petten: Status on cell culture, mice and dog experiments.
4th International Symposium on Neutron Capture Therapy for Cancer Sydney, Australia,
4-7 December 1990

C.P. Hunter, N. G. Tailor, R.C. Hurst;
The Influence of Multiaxial Stress and Corrosion on the Creep Duties of Alloy 800H
Rupture Ductibility of Creep Resistant Steels,
York, UK, 4-5 December 1990,
ORA 35568

R.C. Hurst, N.G. Tailor, B.R. Twaddle;
Prediction of Alloy 800H and 2 1/4 G 1 Mo Component Creep Behaviour from Uniaxial Specimen Data.
4th International Conference on Creep and Fracture of Engineering Materials and Structures, Swansea, 1-6 April 1990,
ORA 35265

R.C. Hurst, C.P. Hunter, D.M.R. Taplin;
Determination of the Controlling Multiaxial Stress Rupture Criterion in Tubular Alloy 800H Components.
High Temperature Materials for Power Engineering Liège, 24-27 September 1990,
ORA 35523

R.C. Hurst;
Internal Pressure Testing of Tubular Components at Elevated Temperatures - The Route to Establishing a Testing Standard.
Conference on Harmonization of Testing Practice for High Temperature Materials,
Travedona Monate 18-19 October 1990
ORA 35754

W. Janssens, P. de Meester,
F. Farfaletti-Casali, R. Matera, Y. Crutzen;
The divertor element in future Tokamak-type fusion reactor: technological requirements and alternative solutions.
Annual Meeting on Nuclear Technology 1990, Nürnberg, 21-25 May 1990

M. Jeandin, F. Dos Santos, F. Brossa;
Electron beam surface alloying of copper with aluminium. IV International Conference on Surface Modification Technologies,
Paris, 6-9 November 1990,

H. Kröckel, N. Swindells;
EC Activities on Materials Data Systems.
Computer Applications to Materials Science and Engineering, Tokyo, 28-31 August 1990,
ORA 35782

H. Küpfer, C. Keller, C. Meier, K. Salama,
V. Selvamanickam, G.P. Tartaglia;
Critical Current and Relaxation of Oriented Grained $YBa_2Cu_3O_7$ after Fast Neutron Irradiation.
Applied Superconductivity Conference, United States, September 1990

K. Kussmaul, U. Mletzko, S. Crutzen, H. Herkenrath;
Status of PISC III Action 2: Full Scale Vessel Testing and First Results.
10th International Conference on NDE,
11-14 June 1990, Glasgow, (UK)

H. Kwast, R. Conrad, S.D. Preston, G. Verstappen,
N. Roux, S. Casadio, H. Werle, J.D. Elen;
Comparison of the Tritium Residence Times of Various Ceramic Breeder Materials Irradiated in EXOTIC Experiments 4 and 5
16th Symposium on Fusion Technology (SOFT), London, 3-7 September 1990,
ORA 35360

G.Y. Lai, J.F. Norton, F.G. Hodge;
The Corrosion Behaviour of a New Sulphidation Resistant Alloy in a Sulphidising/Oxidising/ Carburising Atmosphere.
1st International Conference on Heat Resistant Alloys, Lake Geneva, Wisconsin, 22-26 Sept. 1990,
ORA 35781

F. Lakestani;
Analyse Theorique de la modelisation prevoyant les resultats d'un controle non destructif par ultrasons.
1er Congres COFREND sur les Essais Non Destructifs, Nice, 6-9 November 1990

F. Lakestani, V. Schmitz, J. Coffey;
The Models Validation Work in PISC III.
10th Conference on NDE in the Nuclear and Pressure Vessel Industries, Glasgow, Scotland,
11-14 June 1990

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
A proposed upgrading of the HFR HB-8 neutron radiography facility for commercial application.
First International Topical Meeting on Neutron Radiography System Design and Characterization, Pembroke/Canada, 28-30 August 1990,
ORA 35694

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
Experiences in radiographic unsharpness determination by means of a knife edge object.
First International Topical Meeting on Neutron Radiography System Design and Characterization, Pembroke/Canada, 28-30 August 1990

H.P. Leeflang, J.F.W. Markgraf, S. McAllister,
K.H. van Otterdijk;
Non-destructive testing methods at the HFR Petten for Inspection and Examination of LWR fuel Rods.
KTG-Fachgruppe Brennelemente, Fachtag 1990, Bonn, 19-20 November 1990

Y.Z. Li, M.C. Nguyen, H.H. Over;
Creep and Crack Growth Evaluation within the European High Temperature Materials Databank Petten.
4th International Conference on Creep and Fracture of Engineering materials and Structures, Swansea, 1-6 April 1990,
ORA 35264

A.C. Lucia, A. Servida, J. Bressers;
Expert System for creep damage assessment and headers management.
International Conference on High Temperature Structural Design,
Venice, October 1990

M. Marchionni, D.J. Boerman, G. Piatti;
Fatica oligociclica ad elevata temperatura di un acciaio austenitico al Cr-Mn per reatori a fusione.
Convegno Ricerca, Sviluppo e Tecnologie dei Materiali per i reattori a fusione, ENEA,
Frascati, 4-6 December 1990

J.W.F. Markgraf, S. McAllister, H.P. Leeflang, K.H. v. Otterdijk;
Non-destructive Testing Methods at the HFR for Inspection and Examination of LWR Fuel Rods.
Fachtag 1990, KTG Fachgruppe Brennelemente,
Bonn, 19-20 November 1990,
ORA 35833

J. Marples, N. Godon, F. Lanza, A. Loida, P. van Iseghem;
Radionuclide release from high-level waste forms under repository conditions.
3rd EC Conference on Radioactive Waste Management and Disposal,
Luxembourg, September 1990

R. Matera;
The JRC High Temperature Data Bank.
PCF Development and Testing.
Experimental Simulation of Plasma-Wall Interaction.
IAEA Consultants' Meeting on Thermal response of Plasma Facing Materials and Components,
Vienna, 11-13 June 1990

R. Matera;
Status of First Wall Thermal Cycling Experiments.
IAEA Research Coordination Meeting on Lifetime Predictions for the First Wall of Fusion Machines,
ISPRA, 12-13 September 1990

R. Matera;
Materiali compositi nella tecnologia della fusione.
Convegno Ricerca Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

R. Matera, P. Ruatto, M. Merola;
Comportamento a fatica termica di un modello di prima parete del reattore a fusione ITER/NET.
Convegno Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

R. Matera, P. Miele, M. Merola;
Deformazioni residue nell'acciaio austenitico AISI 316L sottoposto a cicli di fatica termica.
Convegno Ricerca, Sviluppi e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

S. McAllister, A. Carey;
Development of an Improved, Automated NDE Facility for LWR Fuel Rod Testing.
10th International Conference on NDE in the Nuclear & PV Industries,
Glasgow, 11-14 June 1990,
ORA 35273

G. Mercurio, R. Coppola, P. Fenici, O. Francescangeli;
Camera per impiantazione ioni leggeri ad alta temperatura.
Convegno su Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA Frascati, 4-6 December 1990

P. Miele, E. Pizzinato, M. Merola, R. Matera;
Prove di fatica termica su prototipi di prima parete del reattore sperimentale a fusione NET/ITER.
Analisi delle deformazioni mediante il metodo del Moiré geomtrico.
XVIII Convegno Nazionale dell'Ass.
It. per l'Analisi delle Sollecitazioni,
Amalfi, 12-15 September 1990

R.L. Moss, L. Debarberis, M. Beers;
Determinazione in Pila della Potenza Fissile di Combustibile.
Nucleare Utilizzando Metodi Colorimetricci e l'Applicazione di Tecniche di Analisi del Rumore.
VIII Congreso Trasmissione Calore,
Ancona, 28-30 June 1990,
ORA 35460

R.L. Moss, M. Beers, L. Debarberis, G. Tsotridis;
Operational Transient Experiments on FBR Fuel Pins in the High Flux Materials Testing Reactor HFR Petten.
International Fast Reactor Safety Meeting 1990,
Snowbird, Utah, 12-16 August 1990,
ORA 35461

R.L. Moss, F. Stecher-Rasmussen, R. Huiskamp, L. Dewit, B. Mijnheer;
The Petten BNCT Project.
4th International Symposium on Neutron Capture Therapy for Cancer
Sydney, Australia, 4-7 December 1990

J.F. Norton, S. Canetoli, K. Schuster;
Nucleation and Growth Studies of Surface Oxides and Sulphides formed on Fe-Cr-Ni Alloys during Exposure to H_2-CO-H_2S Atmospheres.
Microscopy of Oxidation, Cambridge (UK), 26-28 March 1990,
ORA 35338

J.F. Norton, G.F. Hodge, G.Y. Lai;
A Study of the Corrosion Behaviour of some Fe-Cr-Ni and Advanced Ni-based Alloys Exposed to a Sulphidising/Oxidising/Carburising Atmosphere at 700^{0}C.
High Temperature Materials for Power Engineering, Liège, 24-27 September 1990,
ORA 35432

J.F. Norton, R. Santorelli, S. Canetoli, K. Schuster, F. Bregani;
Corrosion Behaviour of Commercial Alloys Exposed to Sulphidising/Oxidising/Carburising Atmospheres with Relevance to Coal Gasification Conditions.
High Temperature Materials for Power Engineering Liège, 24-27 September 1990,
ORA 35456

S.D. Peteves;
Improved Joining of Surface Modified Silicon Nitride Ceramics.
Proceedings of the 7th CIMTEC, World Ceramics Congress.
P. Vincenzini, (Ed.), Elsevier 1990 (to be pulished)
ORA 35521

S.D. Peteves, H. Jensen, G. Sorensen, A. Vredenberg;
Effects of Ion Implantation on the Mechanical Properties of Silicon Nitride Ceramics.
Proceedings of the 7th CIMTEC, World Ceramics Congress,
P. Vincenzini, (Ed.), Elsevier 1990 (to be published)
ORA 35522

S.D. Pevetes, M.G. Nicholas;
Materials Factors Affecting Joining of Silicon Nitride Ceramics.
Proceedings of the ASM-TMS 1990 Annual Fall Meeting.
P.K. Kumar, (Ed.), TMS, Warrendale, Pa.
(to be published)
ORA 35953

G. Piatti, F. Dos Santos Marques, M. Vedani;
Sviluppo di microstrutture nella lega Fe-10Cr-18Mn-0.2N rifusa al laser.
XXIII Convegno Nazionale AIM, Ancona, 25-27 September 1990,
(Oral presentation + Proceedings)

G. Piatti;
Acciai inossidabili austenitici per la prima parete.
Convegna Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA Frascati, 4-6 December 1990
(Invited Paper + Proceedings)

J.B. Price, M.J. Bennett, J.F. Norton, S. Canetoli;
Real Time Studies of Uranium Dioxide Oxidation.
Microscopy of Oxidation, Cambridge (UK), 26-28 March 1990,
ORA 35564

D.G. Rickerby, G. Piatti;
Influence of thermomechanical processing on martensitic transformation in a chromium-manganese austenitic steel.
Conference: Microstructure and Mechanical Processing, Cambridge, 28-30 March 1990,
Proceedings Mat. Science and Technology
(to be published)

D.G. Rickerby, G. Piatti;
A comparative study of the tensile behaviour of a chromium manganese austenitic steel and AISI 316
4th International Conference on Creep and Fracture of Engineering Materials and Structures,
Swansea, 1-6 April 1990

E. Ruedl, G. Valdrè;
Microscopy and nanoanalysis of a Cr-Mn austenitic steel alphabombarded at room and elevated temperature. International Conference on Microbeam Analysis.

E. Ruedl, G. Valdrè;
Nanoscale compositional changes in Cr-Mn steels alpha-bombarded at 773 K.
12th International Congress for Electron Microscopy, Seattle, August 1990

E. Ruedl, V. Buscaglia, G. Battilana, De Asmundis;
Ossidazione in aria tra 700 e 1200°C di acciai austenitici al Mn di potenziale uso in reattori a fusione.
Convegno Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

E. Ruedl, G. Valdrè;
La microscopia elettronica nello studio del danno da radiazione negli acciai per reattori a fusione.
Convegno Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

R. Santorelli, J.F. Norton, F. Bregani;
A comparison of the corrosion behaviour of FeCralloy and MA 956 in simulated coal gasification atmospheres.
DECHEMA Workshop on "High Temperature Corrosion of Advanced Materials",
Frankfurt am Maim, Germany, February 1990,
ART 29526

R. Santorelli, J.F. Norton. F. Bregani;
High Temperature corrosion of some Fe-Ni-Cr commercial alloys in gaseous atmospheres with relevance to coal conversion processes.
Proceedings of 11th International Corrosion Congress.
published by Associzione Italiana di Metallurgia (1990)
ORA 35089

P. Schiller, P. Fenici;
Problematiche dei Materiali per i reattori a fusione.
Convegno su Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990,
(Invited Paper + Proceedings)

R. Scholz, R. Matera, G. Bergamo, M. Merola;
Sperimentazione ed analisi numerica dell'interazione tra fatica termica e creep da irraggiamento.
Convegno su Ricerca, Sviluppo e Tecnologie dei Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990

W. Schüle;
A new approach to radiation damage.
International Conference on Radiation Materials Science, Alushta (USSR) 25 May 1990

W. Schüle;
A Contribution to the phase diagram of Fe-Mn-Cr alloys.
15th Symposium on Radiation Effects,
Nashville (USA), 17-20 June 1990

H. Stamm, G. Walz;
Numerical calculation of crack tip parameters in a viscoplastic material. 5th International Conference on Numerical Methods in Fracture Mechanics,
Freiburg, April 1990

H. Stamm;
Determination of creep damage in an austenitic steel. IV IUTAM Symposium on Creep in Structures,
Krakow, September 1990

H. Stamm, E. Gariboldi, F. Lakestani,
P. Rimoldi, R. Scholz, U. von Estorff;
Determination of creep damage by ultrasonic velocity measurements. "High Temp. Materials for Power Engineering", Liège, September 1990

H. Stamm, U. von Estorff;
The use of creep damage measurements in combination with material models for residual lifetime assessment. 8th European Conference on Fracture, Torino, 1-5 October 1990

F. Stecher-Rasmussen, J.B.M. de Haas,
W. Freudenreich, W. Voorbraak, B. Mijnheer,
R.L. Moss, M. Konijnenberg;
Dose Distribution Studies for BNCT Optimisation and Treatment Planning.
4th International Symposium on Neutron Capture Therapy for Cancer,
Sydney, Australia, 4-7 December 1990

F. Stecher-Rasmussen, R. Huiskamp,
M. Konijnenberg, V.G.A. Gregoire, B. Mijnheer,
A.C. Begg, R.L. Moss, L. Dewit;
Boron detection for the Petten BNCT project:
prompt-gamma, ICP-AES track etch and ESI.
4th International Symposium on Neutron Capture
Therapy for Cancer,
Sydney, Australia, 4-7 December 1990

D.J. Stephenson, J.R. Nicholls, J.F. Norton,
F. Gandrey, F. Stott, R. Prescott, M.M. Stack,
G. Wouters, W. DeBruyn, W. Engelen;
Modelling of erosion/corrosion processes in coal
conversion systems.
International Conference on High Temperature
Materials for Power Engineering,
Liège, Belgium, September 1990,
ORA 35832

M.F. Stroosnijder, V. Guttmann, J.H.W. de Wit;
Corrosion Behaviour of a Heat Resistant Steel
in the Stress Free and Stressed State under
Simulated Coal Gasification Conditions.
11th International Corrosion Congress,
Florence, Italy, 2-6 April 1990,
ORA 35170

N. Swindells, H. Kröckel;
Standards for Materials Databases:
An International Concern.
12th International CODATA Conference,
Columbus, Ohio, 15-19 July 1990,
ORA 35783

G.P. Tartaglia, G. Piatti, H. Scheurer,
W.V. Witzenburg;
Irradiation Effects on Mechanical Properties of
316 Welded Joints.
16th Symposium on Fusion Technology (SOFT),
London, 3-7 September 1990,
ORA 35361

G.P. Tartaglia, G. Sordon;
Irradiation of Fusion Reactor Materials in the
HFR Petten.
Convegno su Ricerca, Sviluppo e Tecnologie dei
Materiali per i Reattori a Fusione,
ENEA, Frascati, 4-6 December 1990,
ORA 35880

M. Vedani, F. Dos Santos Marques, G. Tuberga,
G. Piatti, V. Wagner;
Microstructure and properties of aluminium alloy
7050 electron beam.
Conference on Microstructure and Properties of
Al 7050 Electron Beam Weldments,
ESTEC, The Netherlands, March 1990

M. Vedani, F. Dos Santos Marques, F. Brossa,
G. Piatti, G. Donzelli;
Particulate reinforced aluminium metal matrix
composites produced through a plasma spray
co-deposition technique: processing and
microstructural aspects.
Advanced Aluminium and Magnesium Alloys,
ASM Europe, Amsterdam, 20-22 June 1990

Y. Yamauchi, M. Steen, J. Bressers;
Subcritical crack growth behaviour of SiC-whisker
reinforced silicon nitride ceramic.
4th International Conference on Creep and
Fracture of Engineering Materials and Structures,
B. Wilshire, R. Evans (Eds.),
The Institute of Metals (1990)

Y. Yukihiko, M. Steen;
Subcritical Crack Growth Behaviour of SiC-Whisker
Reinforced Silicon Nitride Ceramics.
4th International Conference on Creep and
Fracture of Engineering Materials and Structures,
Swansea, 1-6 April 1990,
ORA 35156

M. Zucchetti, D. Boerman, G. Piatti;
Characterization of a titanium alloy for nuclear
fusion applications.
VI Meeting Internazionale sul Titanio,
Torino, 23 November 1990

Special Issues

P. Büttner, H. Kröckel (Eds.)
Report of a VAMAS Workshop on Standards for Materials Databanks.
Petten, November 1988,
VAMAS Technical Report No. 4, November 1989

H. Over, F. Franck;
High Temperature Materials Databank.
Brochure

IV Meetings/ Conferences

Meetings/Conferences

Date	Venue	Subject Area	Type of Meeting	Co-organiser/Co-Sponsor
13/14-2-1990	Petten	Ceramic Materials	Task Group	European Group of Fracture
15-2-1990	Petten	Prenormative R&D for Advanced Ceramics	Ad-hoc Committee Round Table	–
22/23-2-1990	Petten	Standards for Advanced Ceramics	Technical Committee	CEN-TC184
3/6-4-1990	Petten	Designing with Structural Ceramics	Workshop	ECN, NL; European Physical Society
31-5-1990	Petten	High Temperature Fiber Technology	Expert Meeting	Ecole des Mines, F.
5/6-6-1990	Brussels	Intermetallics	Expert Meeting + Workshop	DG XII/C, EGSI
19/21-6-1990	Petten	-Advanced Blading for Gas Turbines -Prediction of Lifetime	Annual Progress Meeting	COST 501
20/21/22-6-1990	Ispra	Classification of Advanced Ceramics	Technical Working Party + Workshop	VAMAS
25/27-6-1990	Petten	User Aspects of Phase Diagrams	Conference	Institute of Metals, U.K.
3-7-1990	Ispra	Structural Integrity and Materials Testings	Seminar	ENEA
27/31-8-1990	Strasbourg	Reactor Dosimetry	Symposium	ASTM, IAEA
12/13-9-1990	Ispra	Lifetime Predictions for First Wall of Fusion Machines	Research Coordination Meeting	IAEA
10/12-10-1990	Ispra	Radio-isotopes	Course	–
1/2/3-10-1990	Ispra	Low Activation Materials	Workshop	DG XII-H
18/19-10-1990	Ispra	High Temperature Materials Testing	Conference	NPL, UK; BCR

V Glossary

Glossary

ABAQUS	Finite Element Code
AES	Auger Electron Spectroscopy
ARTIC	Expert System
ASME	American Society for Mechanical Engineers
ASTM	American Society for Testing and Materials
BCR	Bureau Communautaire de Référence
BNCT	Boron Neutron Capture Therapy
BRITE	Basic Research in Industrial Technologies for Europe
BUMMEL	Bubble Mobility Measurement Level
BWR	Boiling Water Reactor
CAE	Computer Aided Engineering
CCG	Creep Crack Growth
CEC	Commission of the European Communities
CEGB	Central Electricity Generating Board
CEN	Comité Europeen de Normalisation
CISE	Centro Informazioni Studi Esperienze
CMC	Ceramic Matrix Composite
CNR	Centro Nationale di Ricerche
CODATA	Committee for Data on Science and Technology
COST	European Cooperation in the Field of Science and Technical Research
COST 501	Advanced Materials for Power Engineering
CRKPRO	Crack Propagation
CT	Compact Tension
CVD	Chemical Vapor Deposition
DAQ	Decentralized Data Acquisition
DB	Data Bank
DG	Directorate General
DOT	Deterministic Code
EB	Electron Beam
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
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
FRUST	Fusion Reactor Utilisation of Stainless Steel

FTIR	Fourier Transformed Infra-Red
HFR	High Flux Reactor
HPSN	Hot-Pressed Silicon Nitride
HSS	High Speed Steel
HTM	High Temperature Materials
HTMDB	High Temperature Materials - Data Bank
HTR	High Temperature Reactor
IAM	Institute for Advanced Materials
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
IMPACT	Community Programme: Internal Information Service Market
IR	Infra Red
ISE	Institute for System Engineering (JRC Ispra)
ISI	In Service Inspection
ISO	International Organization for Standardization
IIW	International Institute of Welding
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 Coolant Accident
LPPS	Low Pressure Plasma Spray
LWR	Light Water Reactor
MAN	Machienenfabriek Augsburg-Nürnberg
MCNP	Monte Carlo Neutron and Photon Code
MFE	Margin to Failure Evaluation
MOX	Mixed Oxide
MPA	Staatlich Materialprüfungsanstalt (Stuttgart)
MPR	Materials Performance en Reliability
NDE	Non Destructive Evaluation
NDT	Non Destructive Testing
NEL	National Engineering Laboratory
NET	Next European Torus
NILOC	Nitride Fuel, Low Oxygen and Carbon
NPL	National Physical Laboratory
ODS	Oxide Dispersion Strengthened
OECD	Organization for Economic Cooperation and Development
PAS	Positron Annihilation Spectroscopy
PD	Potential Drop
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
PWR	Pressurized Water Reactor
PVSCC	Pressure Vessel Stress Corrosion Cracking
RBS	Rutherford Backscattering
RBSN	Reaction Bonded Silicon Nitride
R&D	Research and Development
REFLEXAFS	Extended X-ray Absorption Fine Structure in Reflexion Mode
RF	Radio Frequency
RLE	Residual Life Evaluation
RRT	Round Robin Test
RT	Radiographic Techniques
RWE	Rheinisch Westphälische Elektrizitätswerke
SAM	Scanning Auger Microscope
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
SOC	Sulphidizing/Oxidizing/Carburizing
SOXAFS	Surface Oriented X-ray Absorption Fine Structure
S/T	Science and Technology
STEP	Science and Technology for Environmental Protection
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
VAMAS	Versailles Agreement on Advanced Materials and Standards
VPS	Vacuum Plasma Spray
WP	Work Package
XANES	X-ray Absorption Near Edge Structures
XPS	X-ray Photo-emission Spectroscopy
XRD	X-ray Diffraction

VI List of Authors

List of Authors

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

II **SCIENTIFIC - TECHNICAL ACHIEVEMENTS**

- Alloys: J.B. Marriott, J. Bressers, V. Guttmann, R.C. Hurst, J.F. Norton.
- Engineering Ceramics: M. Van de Voorde, J. Bressers, E. Bullock, R.J. Fordham, J.F. Norton, M. Steen.
- Components and Thermal Fatigue: J. Bressers, G. Tartaglia, R. Hurst, L. Lamain.
- Operational Defects in Materials and Lifetime Predictions: H. Stamm, F. Lakestani, P. Rimoldi, R. Scholz, U. Von Estorff.
- Wear and Corrosion Resistant Coatings: W. Gissler, E. Lang.
- Composite Materials Properties Improvements: G. Piatti, F. Brossa, F. Dos Santos Marques, M. Vedani.
- Chemical Sensors: L. Manes, G.B. Bardi, M.D. Giardina, A. Manara.
- Surface Treatments for Improved Performance: P. Schiller, F. Brossa, F. Coen, O. Gautsch, F. Geiger, N. Gibson, A. Manara, L. Manes, D. Quataert, P. Weisgerber.
- Databanks: F. Franck, H. Over, Y. Li.
- Information Centre: M. Merz.
- Project on the Integrity of Steel Components (PISC): P. Jehenson.
- Safety of Final Storage in Geological Formation: Materials Research Aspects: F. Lanza.
- Materials Integrity: P. Schiller, D. Boerman, F. Brossa, V. Coen, P. Fenici, O. Gautsch, G. Piatti, D. Quataert, D.G. Rickerby, T. Sample, R. Scholz, W. Schüle.
- Nuclear Fuels and Actinide Research: R. Moss.
- Operation of the High Flux Reactor: J. Ahlf.
- Standards for Advanced Ceramics: M. Merz, J. Bressers, P. Jehenson, J.F. Norton, M. Steen.
- Standardization of Quality Control Protocols for Cyclotron Produced Radio-Pharmaceuticals: M. Castiglioni, B. Weckermann.
- Materials Data Banks: H. Kröckel, N. Swindells.
- Valorization of JRC Research Results: Spontaneous Downward Heat Transport: G. De Beni; Oxygen Sensors: L. Manes.

- Boron Neutron Capture Therapy (BNCT): R. Moss.
- Joining of Ceramics to Metals: S. Peteves.
- Micro-Hydrodynamics of Laser Melted Pools: G. Tsotridis.
- Development of Intelligent Processing for Sub-Micron Ceramic Structures: S. Pickering.
- Cold Fusion: F. Lanza.

ANNEX

INSTITUTE COMPETENCES AND FACILITIES

- Materials Characterization: J. Bressers, W. Bullock, R.J. Fordham, V. Guttmann, J.F. Norton.
- Materials Performance and Reliability: P. Schiller.
- Functional Materials: L. Manes, E. Diana, M. Forte, J. Haupt.
- Non Destructive Testing: P. Jehenson.
- Advanced Materials Informative Systems: H. Kröckel, F. Franck, H. Over, A.G. Youtsos.
- High Flux Reactor Petten: J. Ahlf.
- Cyclotron: L. Manes.
- Environmental Testing Laboratory: J.B. Marriott.
- Surface Modification Centre: P. Schiller.

Editing and Coordination: M. Merz.

Sub-Editing: M.R. Cundy, A. Gevers, P. Gibson, R. Hurst, J.B. Marriott, S. Pickering, D. Rickerby

Annex: Competences and Facilities

1. Skill Pools and Competence Areas

Materials Characterisations

Corrosion Properties

The facilities are primarily housed in the purpose-built Environmental Test Laboratory (ETL) and have been extended during the past year to include the following;

* a further two high-temperature multi-specimen autoclaves have been commissioned and are fully operational (temperature capability = 1500°C),
* a thermobalance capable of enabling continuous mass-change measurements to be made at temperatures up to 1700°C has been installed and is being used for studies concerned with establishing the behaviour of advanced engineering ceramics,
* a laboratory burner rig is presently being commissioned for research investigations concentrating on the influence of hot (molten) salts on the degradation of alloys, ceramics and coatings.

These additional facilities complement those already existing, as summarised in previous Annual Reports. In summary, a total of;

‡ 5 vertical top-loading autoclaves having a temperature capability of 1100°C,
‡ 4 high-temperature (1500°C) multi-specimen autoclaves,
‡ 2 horizontal muffle-furnaces designed to operate at temperatures of up to 1500°C,
‡ 4 thermobalances for continuous thermogravimetric analysis at temperatures ranging from 1000°C to 1700°C,
‡ 1 hot-stage microscope with environmental chamber for in- situ studies of corrosion as it occurs with video camera attachment for recording the events on a real-time base,
‡ laboratory burner rig for hot corrosion studies at temperatures of up to 1200°C.

All of these facilities have been designed to enable high temperature corrosion research investigations to be carried out using any of the following;

- gas mixtures containing a range of toxic and/or explosive species including H_2, CO, CO_2, CH_4, H_2O, H_2S, SO_2, SO_3, N_2 air etc.,
- solid deposits e.g., real or synthetic fly-ash containing mixed sulphates, carbonates, chlorides, etc.,
- molten salt deposits of the types found in boilers and gas- turbines, etc.,
- liquid metals, e.g. aluminium, zinc, etc.

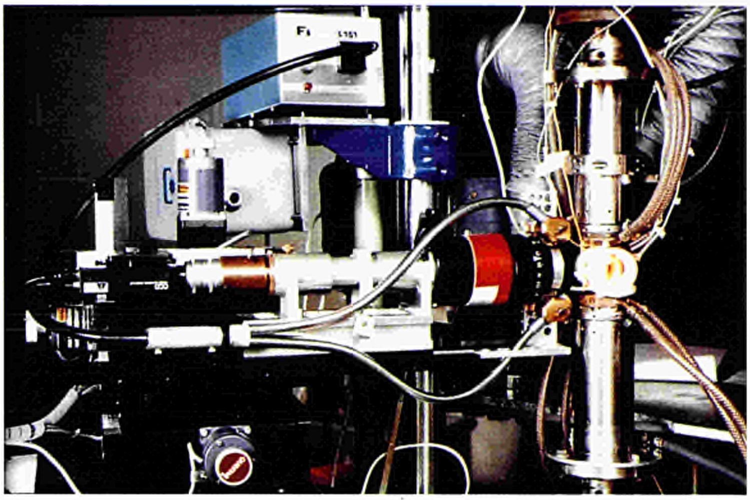
Mechanical Properties/Environment Interaction

The facilities, which are mostly located in the E.T.L. have been extended during the past year with the addition of:

- 1 cyclic creep rig capable of programmed variations in tensile loading and temperatures up to 1100°C;
- 1 cyclic thermal gradient rig for crack growth studies in tubular components internally cooled with water and externally induction heated. Crack growth from machined internal and external defects is continuously monitored using a DC potential drop technique;
- 2 conventional creep machines have been adapted to allow creep crack growth testing of small compact tension test pieces with DC PD used for creep crack growth;
- conversion of one of the tubular component test cells for testing up to 1200°C.

These additional facilities complement those already existing, which have been described in previous reports. In summary, there are in addition:

* 12 uniaxial creep rigs for testing in air to maximum temperatures between 1000°C and 1250°C;
* 17 uniaxial creep rigs for testing in controlled (aggressive) environments; 4 allowing salt additions to be made to the sample during to test. The maximum test temperature lies between 1000°C and 1050°C;
* 1 cyclic creep rig capable of programmed variations in tensile loading and temperature in corrosive atmosphere up to 1050°C;
* 4 tubular component test cells capable of tests to 300 bar pressure and 1050°C in air or controlled environment; 2 with facilities for additional axial loading;
* creep crack growth facilities for incorporation in the above test cells, followed by fracture mechanics analysis.
* 1 rig for uniaxial creep testing in a system pressure of aggressive gas up to 200 bar.



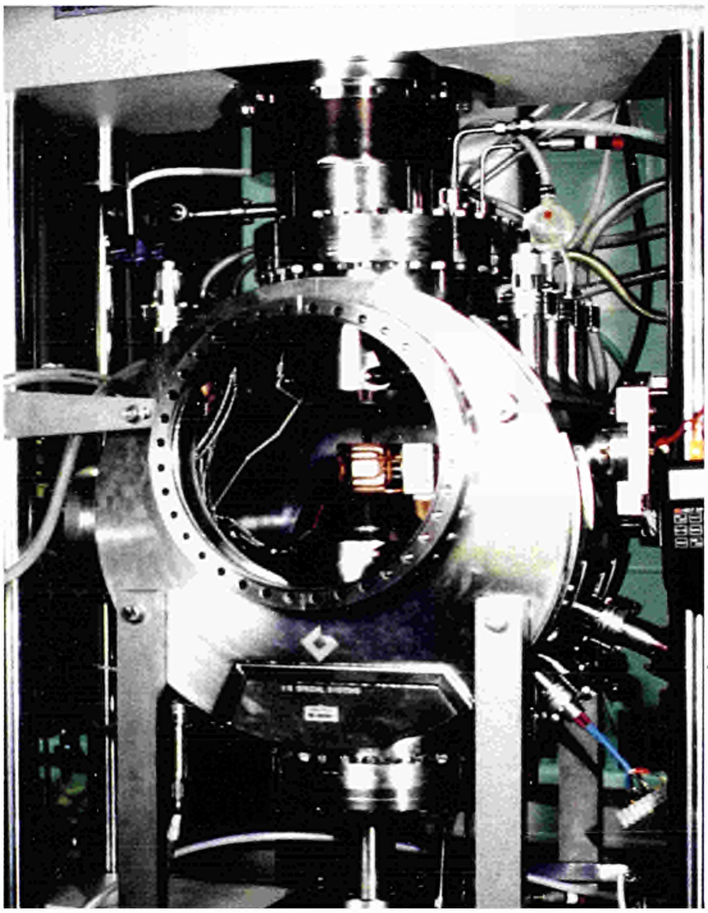
Mechanical Properties

The group's competence essentially concentrates on the measurement of the mechanical behaviour of advanced materials under complex or extreme conditions, on their relationship with the microstructure and on performance prediction.

The range of materials covered includes metallic alloys, monolithic ceramics and continuous fibre ceramic matrix composites. The focus on advanced materials and on extreme testing conditions almost automatically triggers a spin-off into another area of research to which the group is heavily committed, i.e. the prestandardisation aspect of mechanical testing.
Round robin testing, participation in two working groups of technical committee TC 184 of CEN and experimental work constituted the main prestandards activities in the past year.
The experimental work aims at establishing the testing methodology for the uniaxial testing of monolithic ceramics and of CMC's and at analyzing and evaluating the effect of testing variables in order to help defining acceptable tolerance limits to these variables for inclusion in the test standards.

Above: *In-situ monitoring of microcracking during fatigue by means of a computer vision system*

Below: *Uniaxial tension/creep testing of ceramics facility under environmentally controlled conditions*



The group operates eight universal testing machines for the measurement of a range of mechanical properties up to temperatures of 1450°C and a series of in-house developed rigs for 4-point bend testing of ceramics in air and in corrosive environments up to the same temperature level.

Most of the rigs, housed in a temperature and humidity controlled testing laboratory (ETL-S), are modified for special purpose testing.
Several rigs, for example, are adapted for thermomechanical fatigue testing with fast heating and cooling cycles. One of the rigs is equipped with a computer vision system (figure above), for the in-situ monitoring of the initiation and growth of microcracks on the surface of the test sample, achieving a resolution of 1.5 µm.

The images are digitized and stored for subsequent analysis of the growth characteristics of the cracks. Tension, creep and fatigue testing of ceramics in the uniaxial mode has been developed on two rigs, one of which is equipped with an environmental chamber for tests in inert gas or vacuum (figure below). In 1990 a major effort was devoted to the implementation of a decentralized data acquisition system for the digital acquisition, analysis and presentation of test data. Some of the testing rigs in the ETL-S laboratory are now equipped with DAQ-systems.

Interfacial Engineering

Research into Interfacial Engineering of "composite" materials is covered by activities within the Specific Research programme, the Exploratory Research programme and Work for Third Parties. The Group also contributes to collaborative projects in the Eureka and CEC Science programmes.

Founded on a Ceramics Processing remit the Group provides facilities and expertise for the characterisation of powder with respect to particle size and size distribution, particle morphology, surface topography etc. Green forming of powder compacts can be achieved by cold isostatic pressing or slip casting, with a range of facilities to characterise rheology, viscometry and sedimentation in slips.
Densification in controlled environments is available by low pressure sintering or uniaxial hot pressing, or by reaction sintering of ceramic precursors.

Joining of ceramics to ceramics or to metals and engineering of interface chemistry and microstructure are performed by brazing or diffusion bonding. Characterisation of the physical properties of ceramics such as density, hardness, porosity etc., and mechanical testing, is backed up by a machining capability with in-situ measurement of triaxial grinding forces and continuous monitoring of test piece dimensions. Quantification of machining damage is achieved by measurement of rugocity, hardness and residual stress in subsurface layers.

Materials Performance and Reliability

The Division contributes to those programmes of the JRC in which materials have to be selected and characterized with respect to their performance and behaviour under different and difficult operating conditions. Currently it participates in the programmes: Advanced Materials, Fusion Technology and Safety, Waste Management, Reactor Safety and it executes on request work for third parties. The main emphasis is presently on the first two of these programmes. In the frame of these activities it is necessary to determine physical, mechanical and physicochemical properties of selected materials, to relate them to the existing microstructures and to investigate the modifications which the microstructure and the properties undergo, due to the environment of the specific operating conditions under which the materials are used.

In nuclear environments radiation damage and its effect on the mechanical properties, stability, lifetime and corrosion behaviour are determined, described and explained.

For materials for non-nuclear energy conversion, there are requirements to determine the corrosion behaviour at high temperatures and means to protect the materials against agressive environments; it is also required to investigate damage which develops as a result of long term loads at high temperatures, to develop damage laws and to apply this knowledge for lifetime predictions.

Participating in these and other programmes within the Division, a number of techniques are being used and developed.

1. 1. Electron Microscopy has developed to one of the most powerful means for research in Materials Science. Within the MPR Division, this technique has been used widely. The dislocation structure in deformed metals and alloys has been investigated intensively. The interaction of dislocations with dispersed particles for ODS materials has been identified as reason both for their strength as well as for their low ductility. In austenitic steels, the stacking fault energy of dislocations split in partials has been determined. Deformation of metals generates too the formation of second phases which can best be determined by TEM.

   For the development of fusion reactor materials, the study of the different radiation damage features as dislocation loops, voids and He-bubbles is important and is a subject of our activities.

   The attachments to modern microscopes as EDS and EELS systems allow the determination of the chemical composition on a very small scale. The knowledge of the composition of corrosion layers of different origin and the changes of composition of the underlying material are important information to understand the corrosion processes and are being studied for different systems.

2. Radiation Damage is studied in the MPR Division as one of the most important problems for fusion reactor materials.
   A number of devices has been designed and constructed for the study of radiation damage phenomena on the Ispra cyclotron.
   Due to the fact that a light ion cyclotron is particularly apted for in-beam measurements, most of the devices are designed for the observation of dose rate dependent effects.

   Radiation creep can be observed with the JRC installation in a wide temperature and stress range. It was possible to show that irradiation creep occurs at temperatures as low as 80^{0}C. The equipment has now been modified in such a way that it is possible to reverse the stress and to do experiments with hold times.
   This allows to study the interaction between radiation creep and fatigue.

   The laws of propagation of fatigue cracks under irradiation are not known.
   An installation has been constructed to execute measurements on the phenomena.
   Even at rather low doses, comparable to dose rates in a fusion reactor, the crack growth velocity is reduced.
   The changes in mechanical properties and the dose rate effects depend on the microstructure which develops under irradiation. A global information on the development of the microstructure can be obtained by in-beam electrical measurements.

3. Compatibility of Liquid Metals. Liquid metals are used in a number of advanced energy generating systems. MPR Division has developed competences in the field of the compatibility of liquid metals with structural materials. The corrosion by pure Li and Li 17-Pb on austenitic steels has been determined. These studies have been extended to the influence of impurities in the liquid metals on their corrosion behaviour. Special equipments have been developed to maintain a constant hydrogen content in the liquid metal during the corrosion tests.

   Another installation has been designed and constructed which allows to detemine the influence of liquid metals on the mechanical properties of structural materials. Creep and tensile tests in liquid Li 17-Pb are being executed for the determination of liquid metal embrittlement.

4. Mechanical Testing is the starting point for the characterisation of structural materials. Besides the well-known tensile testing of standard specimens and the determination of the thermal creep behaviour which are both normally used by the MPR Division, methods for special cases have been developed. For the determination of the influence of light ion irradiations on the mechanical properties, the development of miniaturised specimens was necessary. This technique is also used to determine the local variation of the mechanical properties in large metallic pieces. The best example is the mapping of flow stress, ultimate tensile stress, ductility and Young's modulus near and within welds. This knowledge gives the possibility to determine in a better way the behaviour of welds in large sections and to calculate the overall properties of the component.

5. Thermal Fatigue. The first wall in a fusion reactor is exposed to a pulsed heat flux. As a consequence, variable thermal stresses are generated in the material. Similar problems appear in other installations. The resulting fatigue problems cannot be described adequately by the well-known mechanical fatigue data. A combination of experimental facilities which allow to generate heat fluxes in relatively simple components as tubes and cooled panels with finite element calculations on temperature distribution, stress fields and strains is applied to establish the lifetime of these components.
   Complex experimental problems as heat flux measurements, surface temperatures in a radiation field and crack detection, have to be solved. The careful elaboration of the measured values allow a verification of the results obtained by the FE calculations. At present, several prototypes of first wall elements for NET are under investigation.

6. Welds. Metallic materials joined by different welding procedures are an essential part of all structures. The Division has developed methods which allow to characterise welds in thick structures with respect to their metallurgical, metallographic and mechanical properties. Advanced analysis methods are applied for the verification of the compositional homogeniety inside the welds, miniaturised tensile testing gives information on the thermomechanical history of the single weld passes, while the metallographic observations give indications on the cooling rate of the melt.

7. Surface Treatments.
   Protection of metallic surfaces is a long standing activity in the Division. The experience in the past had been concentrated on plasma spray coatings in air and under low gas pressure. Experience has been accumulated on coatings for protection of metallic materials against corrosion by sulphuric acids at high temperatures. Other coatings in the field of chromium compounds have been developed, in order to increase the optical absorptivity of surfaces in solar plants. Low Z coatings of the titanium-carbide type have been produced for the surfaces in plasma physics devices. This field has been substantially enlarged in the last year.
   The Division has added the possibility to remelt systematically original or coated surfaces and to generate dense surface layers of different chemical compositions or with fine grained structure. Extensive investigations have allowed to relate melt depth and cooling velocity to the thermophysical properties of the materials and to the parameters of the heating process, i.e. power density and heating time.

Finally, the modification of solid surfaces by ion implantation has been introduced in the procedures for surface modifications. The implantation of a large number of ions is being studied in an extended field of temperatures and at energies between 50 and 200 KeV. The structures generated are close to those of high dose neutron radiation but frequently with the improving effects of the additional atoms.

8. Physical Chemistry is crucial for many problems in Materials Science. Within the Division, corrosion problems are studied in many ways. Experience in high temperature corrosion of nickel alloys by oxidising atmospheres has been accumulated in the past programmes on thermochemical hydrogen production. Corrosion, due to industrial atmospheres in the temperature range below 100^{0}C, has been an argument for solar collectors. The transport of corrosion products in cooling circuits built with stainless steel tubes has been the subject of an intensive study. The migration of corrosion products of different nature in the earthcrust and in different geological formations are being investigated for the programme on nuclear waste.

9. Damage Mechanics. The properties of structural materials are strongly influenced by the defect structure on a microscale which develops during production or during service.
   For the determination of the lifetime of highly stressed components, the knowledge of the defect structure is an important detail.
   On the other hand, this knowledge has to be translated in the values used by the mechanical calculations. It is one of the major tasks of the MPR Division to assemble the information obtained by different techniques on the defect structure, with the notions of solid state mechanics for a reliable prediction of the lifetime of the materials.

   Techniques for the determination of defects are destructive methods as metallography, electron microscopy, X-ray techniques, mechanical testing and non-destructive methods as ultrasonic measurements and X-ray transmission inspection.
   These are guided and evaluated by applied mechanics considerations. Actually, a focus of the activity is the damage developing under creep conditions in steels.

Functional Materials

In the JRC "Advanced Materials" Programme, the Specialized Service "Functional Materials and Cyclotron" has the task of developing new materials which can be employed for their specific properties. In addition the group promotes the adoption of new and advanced characterization methodologies for industrial applications.
The areas of research of the group include:

Chemical sensors

The development of simple, reliable, long-lived, continuously monitoring sensors for the quantitative detection of components in gaseous atmospheres is of importance for both environmental as well as industrial purposes.
The Service has licensed an emf oxygen-sensing cell, with an innovative electrode, which has the advantage of considerable precision, stability of signal, and long-duration.
This oxygen-sensor is being commercialized by the Firm CIFER in Seregno (Milan - Italy).

The Service is at present considering the extension of its sensors activity into two R&D lines:
- development of an NO_x sensor;
- development of an array-sensor (array of micro-processed selective sensor units) for detection of complex atmospheres.

In order to further this work an association is being formed with other European Laboratories.

High Tc superconductors

Structural, thermodynamic and theoretical work is being performed with the purpose of establishing possible correlations between structural, thermodynamic and superconduction parameters.

Network of surface/interface analytical methods

A considerable effort is being devoted to establishing a network of surface/interface analytical methods.
By focusing on the complementarity of these methods an integrated solution for specific problems can be offered to industrial users.

Coatings

In this field, new methods of deposition and mixing of protective and functional coatings are developed. In particular, attention is focussed on:
- production of amorphous coatings
- mixing of insoluble multilayered films
- increase of film/support adherence.

Research is being performed on:
- special types of protective coatings (TiN, BN, carbon-diamond layers)
- ceramics/metal bonding (e.g. amorphous TiN on steel)
- metal/metal mixing (e.g. Ag/Cu).

Ion assisted deposition is currently used, and in the near future the Surface Modification Centre's Facilities (ion implanter, laser and electron beam heating etc.) will also be used.

Facilities

The most relevant facilities directly available or accessible through co-operation contracts are described below.

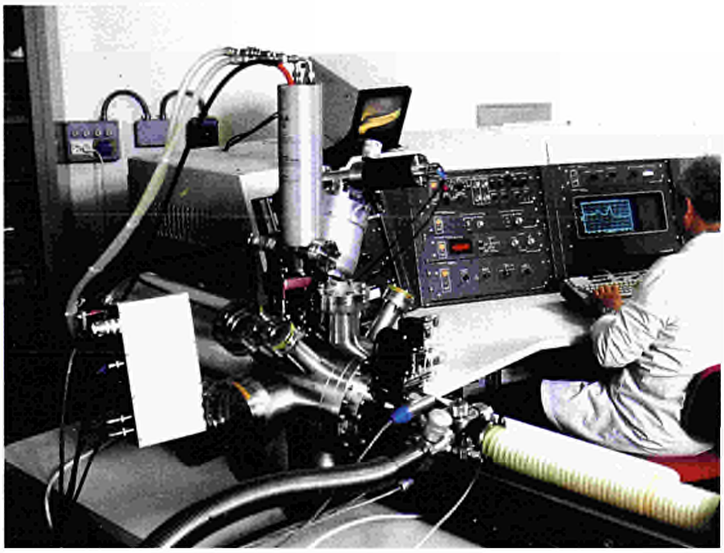
Surface and interface analytical methods.

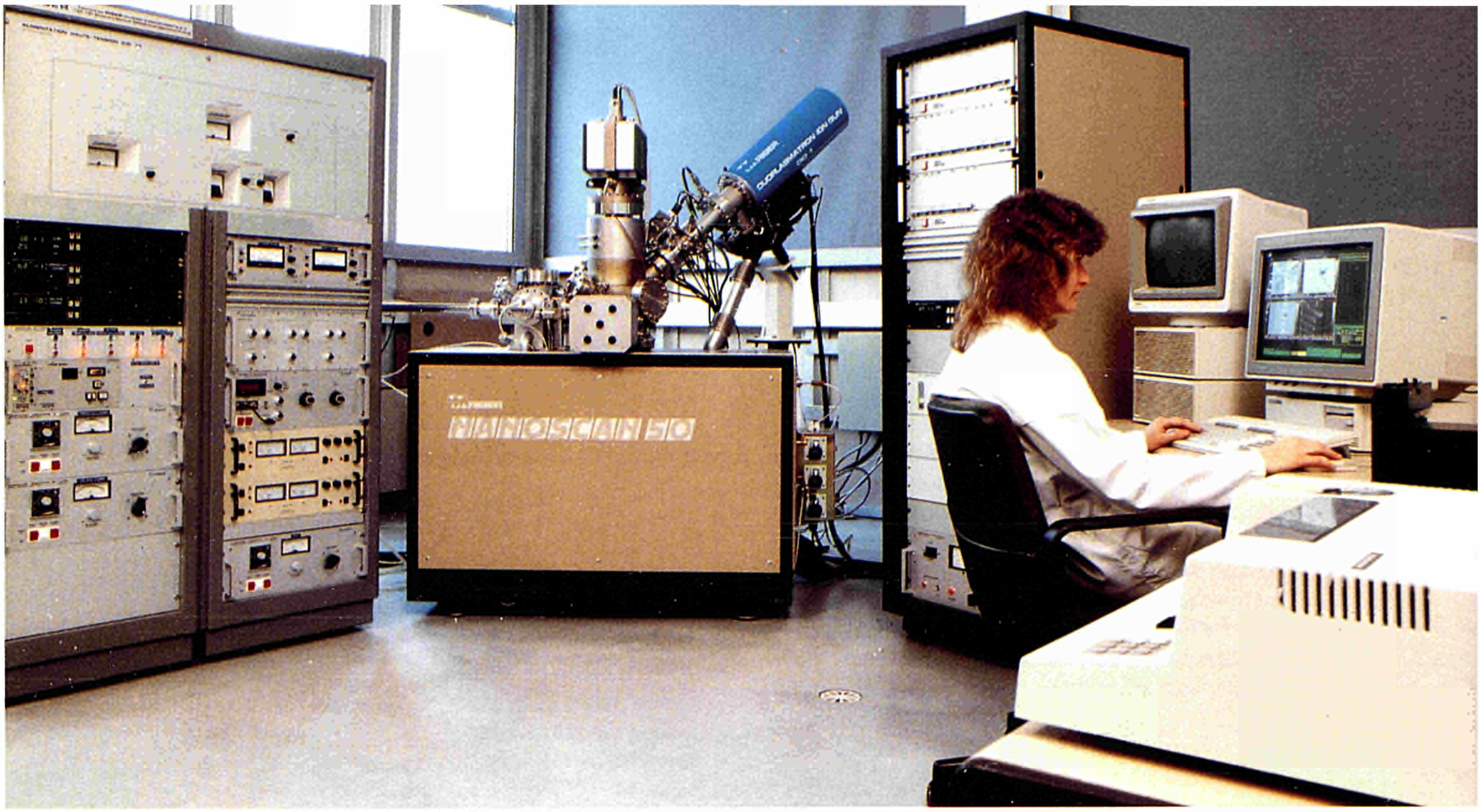
Chemical analysis

a) Perkin-Elmer Scanning Auger/XPS Spectrometer:

XPS high sensitivity (700.000 c/s at 2 eV resolution, 45000 c/s at 0.9 eV resolution), Mg and Al anods. High Auger sensitivity (100.000 c/s at 0.7% resolution with incident current of 15µA), lateral resolution 1µm. (Figure below).

Below: *Surface analysis system employed for the study of radioactive waste glass and metal corrosion*





b) RIBER Scanning Auger Microscope:

High lateral resolution (ca.350 A), high sensitivity 2.10^5 cps (Cu-LMM) at energy resolution of 3 eV, data treatment (peak area, normalization, topological correction on lines and on maps) (Figures above and below).

c) Ellipsometer:

Laser source, microspot (25 µm) high sensitivity for film thickness determination.

Access to:
- Rutherford Backscattering (RBS)
- SIMS
- Nuclear Reactions
- ERD (Elastic Recoil Detection).

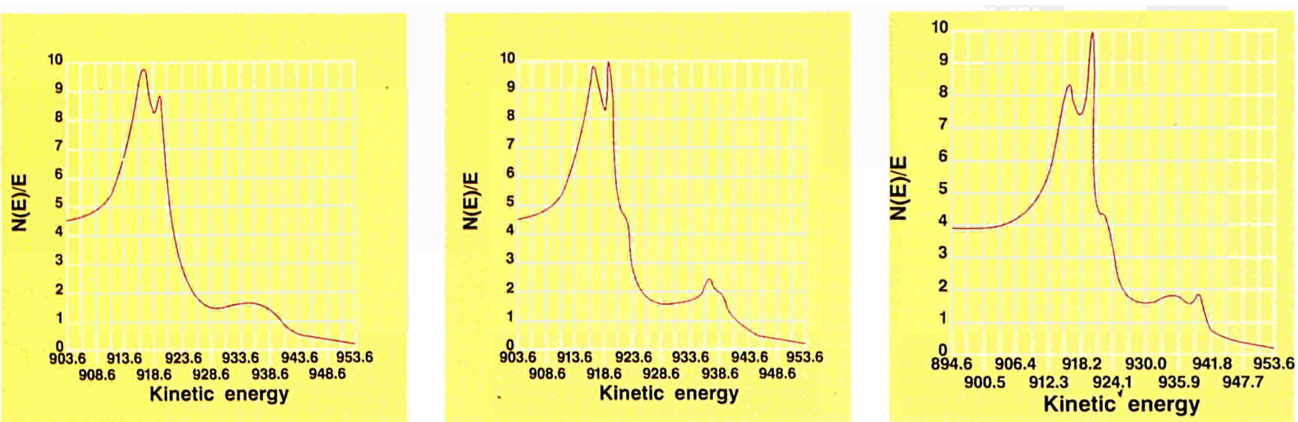
Above: *Riber Scanning Auger Microscope*

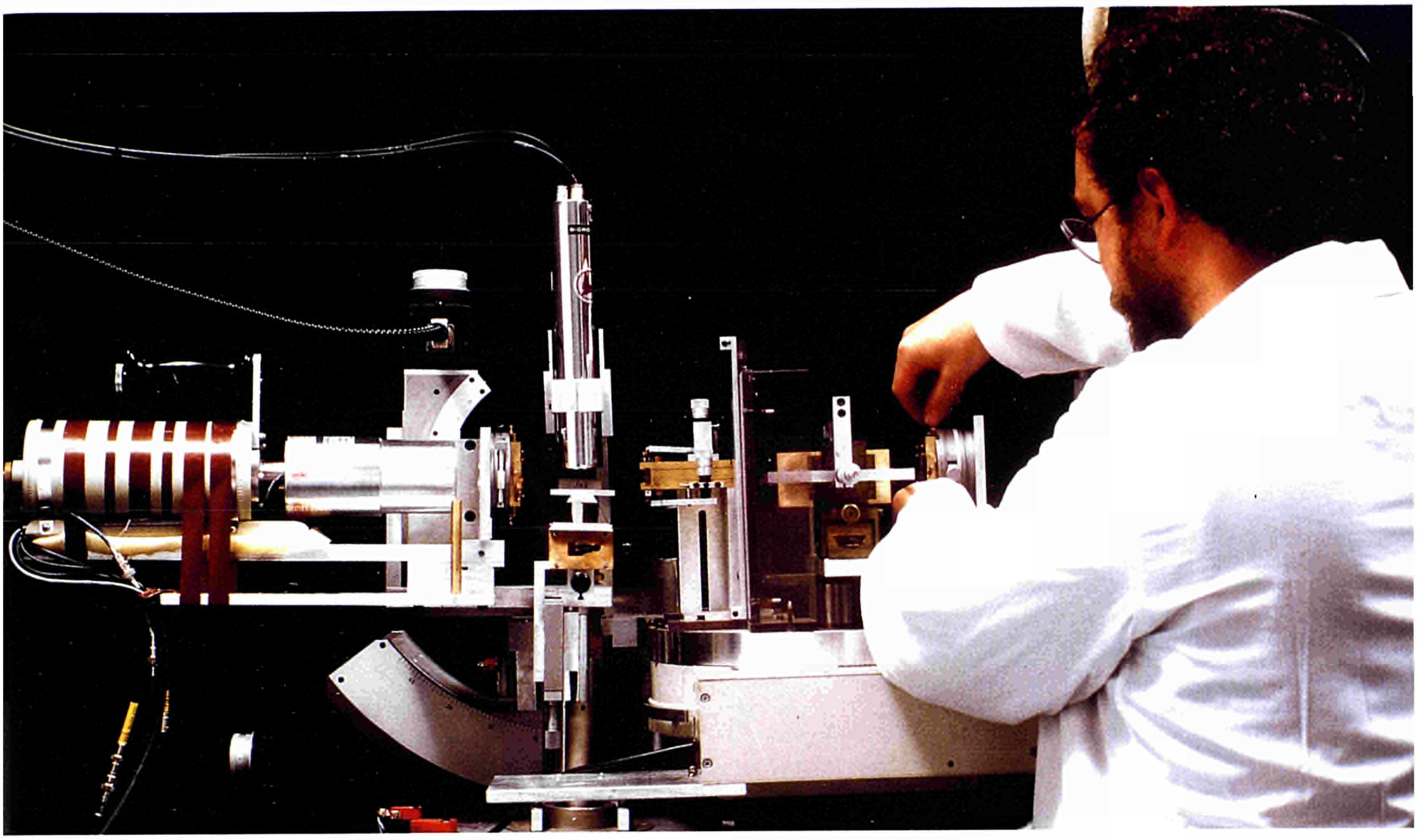
Below: *Auger LVV spectra of COPPER in the Printed CIRCUIT BOARD treated in vapour phase at 220°C for welding process*

1 *Auger LVV spectrum of copper after treatment in vapour phase at 220°C after ≈ 10 Å of sputtering*

2 *Auger LVV spectrum of copper treated in vapour phase and sputtered for ≈ 20 Å*

3 *Auger LVV spetrum of copper treated in vapour phase and sputtered for ≈ 50 Å*





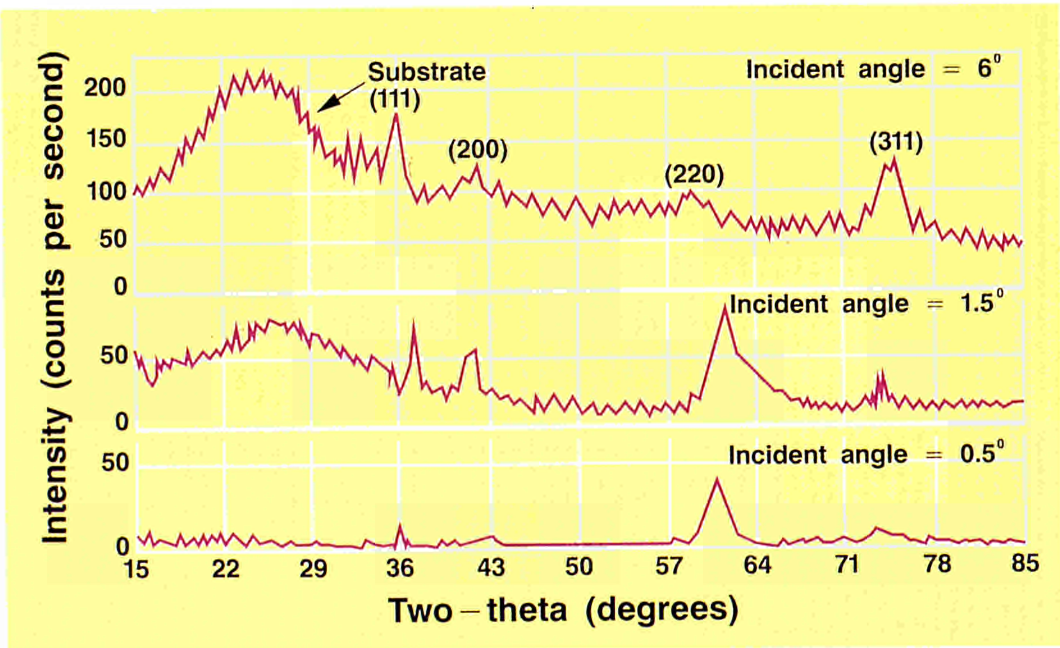
Structural analysis

a) X-ray glancing angle diffractometer and reflectometer:

Parallel beam geometry, surface sensitivity 1 µm, angular resolution 0.3^0 (diffraction), incident angle 0.5^0, angular resolution 0.01^0 (reflection) (Figures above and below).

Above: *Aligning the prototype glancing angle X-ray spectrometer recently contructed in the Physics Division at Ispra for surface studies of advanced materials*

Below: *Glancing angle X-ray diffraction spectra of a thin film of titanium nitride deposited on a glass substrate (Film thickness = 3000 angstroms)*



b) X-ray absorption spectroscopy:

- High resolution XANES, better than 1 eV energy resolution, range 5 KeV to 20 KeV
- High intensity source
- Attachment for surface XANES measurements

Access to:
- Synchroton Radiation (methods: EXAFS, XANES, REFLEXAFS, SOXAFS) Daresbury, UK.

c) Positron annihilation spectroscopy:

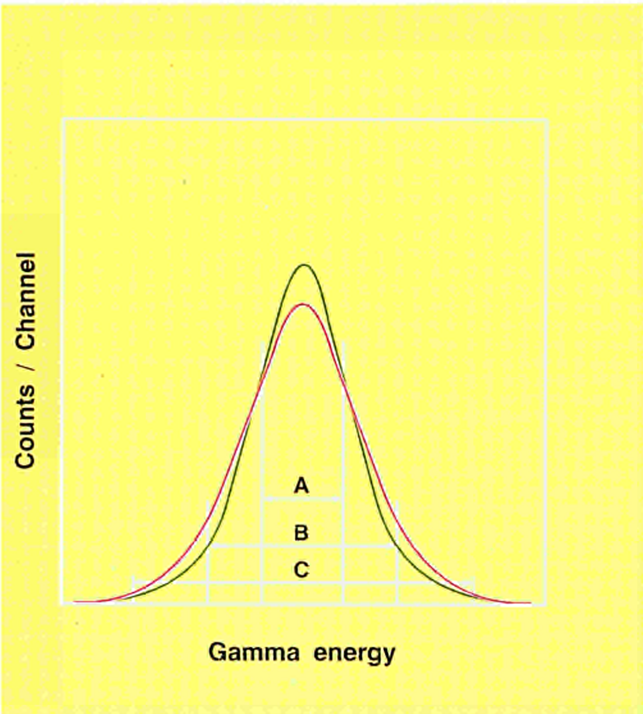
Positron Annihilation Spectroscopy (PAS), based on a Ge-gamma-spectrometer, after completing the facility and the analytical software, has been used for the investigation of defects in micrometric coatings of TiN on glass and metal substrates.

The Doppler effect in the positron annihilation gamma line (depending on the electron momentum distribution in the material) is characterized by standard parameters (Figure below).

In this application, most positrons from ordinary beta sources (Na 22) annihilate deeply in the substrate; however, a very good measuring accuracy and reproducibility allows us to separate the contributions related to defects present in the coating (TiN stoichiometry defect and other defects) as well as introduced by the surface treatment of the substrate (e.g. mechanical polishing, sputtering, etching, etc.). With TiN coatings of 8.5 μ, typical differences in the PAS parameters, related to different stoichiometry, or consequent to annealing, could be determined with an accuracy of 10%.

This example shows the potential of the method for the characterization of surfaces and the study of surface and near-surface properties. The technique is also being tested on ceramic surfaces.

A new system for the production of a slow positron beam (up to 30 KeV), for the characterization of submicrometric surfaces, has been installed, but not yet completed.



Below: *Positron annihilation lineshape parameters are defined by the fraction of gamma-ray counts in fixed energy intervals:*

$$s = A/C \approx 0,5 \qquad W = (C-B)/C \approx 0,2$$

W and S are, respectively, related to annihilation events with high momentum (core electrons) and low momentum (conduction electrons, positron trapped by defects)

Depositing Units

a) MRC Plasma Sputtering Unit.
3 targets, allowing co-sputtering in modes DC and AF (both diode and magnetron cathode type), 200 mm diameter target, equipped with a loadlock system, cryogenic pumping.
b) Plasma Sputtering Unit combined with electron-gun evaporator, target diameter 76 mm, turbomolecular pumping.
c) Deposition unit consisting of two ion guns for ion beam sputtering and ion-beam assisted deposition.
This facility can be used in conjunction with an evaporation/deposition source.
d) Evaporation/deposition source for small samples
e) Several characterization methods for deposited films: scratch-testing, residual stress determination, indentation tests, etc.

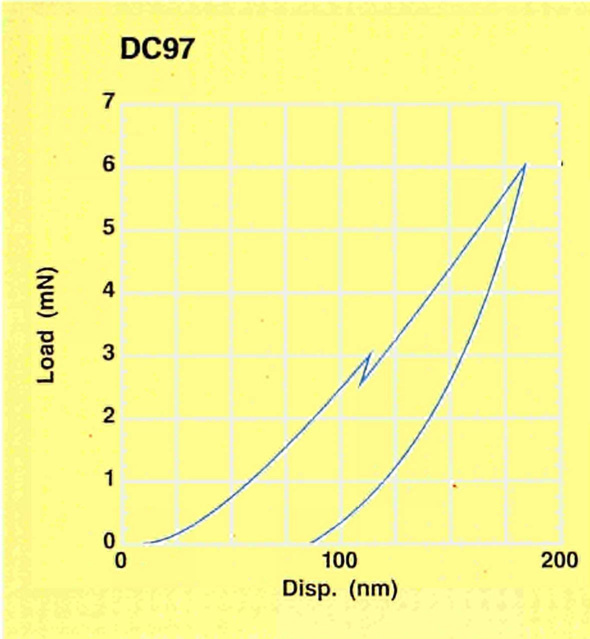
Nanoindenter II

A state of the art mechanical properties microprobe has been available in the IAM-Ispra since May 1990. This instrument allows information to be measured or deduced in the submicron thickness range on the following quantities:
- hardness
- elastic modulus
- yield strength
- creep resistance
- stress relaxation.

It is applied in the field of thin films (wear resistant coatings, protective coatings, optical films, etc.) ion implanted materials, multiphase structures, rapidly solidified alloys, corrosion / cracking / adhesion studies.

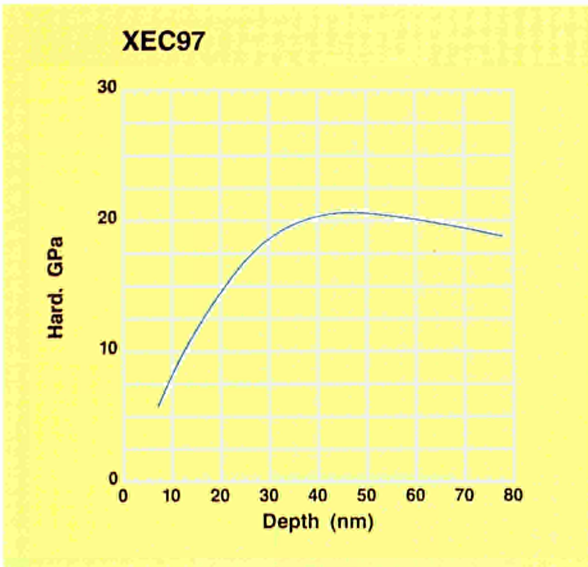
The measuring principle is based on the repeated subnanometer z-axis displacement measurement of a Berkovich diamond tip under the application of a slowly increasing force (via the current setting through a coil, which is attached to the diamond tip support).

Specifications of the instrument:
- displacement resolution ± 0.04 nm
- force resolution ± 75 nN
- four load ranges 0 - 4, 20, 120, 600 mN
- x-y-positioning accuracy 400 nm.



Above: *Load / displacement curve with a 20 % unload at 3 mN of a diamondlike carbon film.*

Below: *Hardness as function of displacement as deduced from the data of figure above*



Non Destructive Testing

Facilities

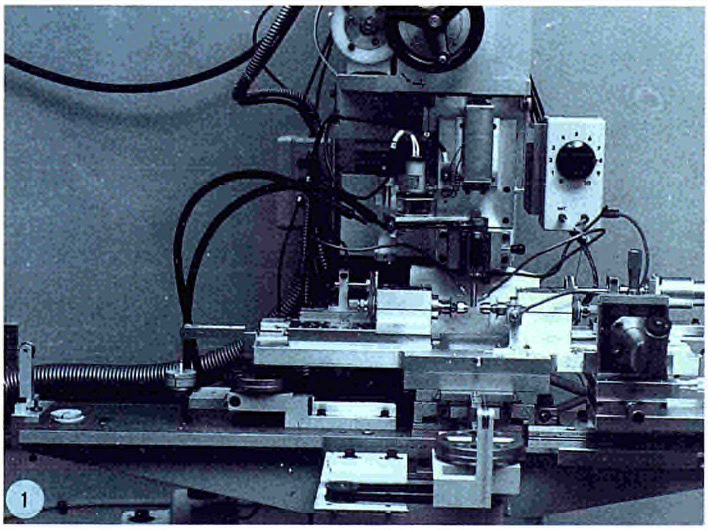
- {} Laboratory for the conception and fabrication of reference defects, using micro spark erosion, milling, punching and drilling techniques (Figure 1 and 2).
- {} Laboratory for the conception and fabrication of special ultrasonic transducers (Figure 3).
- {} Laboratory for the characterization of the ultrasonic instrumentation, using particular techniques (Schlieren, liquid crystals, reference transducers,...) (Figure 4).
- {} Laboratory for basic studies on ultrasound propagation in materials, using highly sophisticated ultrasonic benches (Figure 5).
- {} Laboratory for Applications of ultrasonic techniques, using advanced techniques and automatized scanners.
- {} Laboratory for the characterization of the X-rays instrumentation.
- {} Laboratory for the Applications of X-rays techniques, using 50, 150, 300 and 400 KV equipments; 200 KV microfocus equipment; 1 and 2 MeV linear accelerator (Figure 6).
- {} Laboratory for NDE on contaminated pieces (up to 4 tons), using X-ray, ultrasound remote controlled techniques; also remote controlled destructive examination (Figure 7).
- {} Laboratory for NDE data analysis and evaluation.

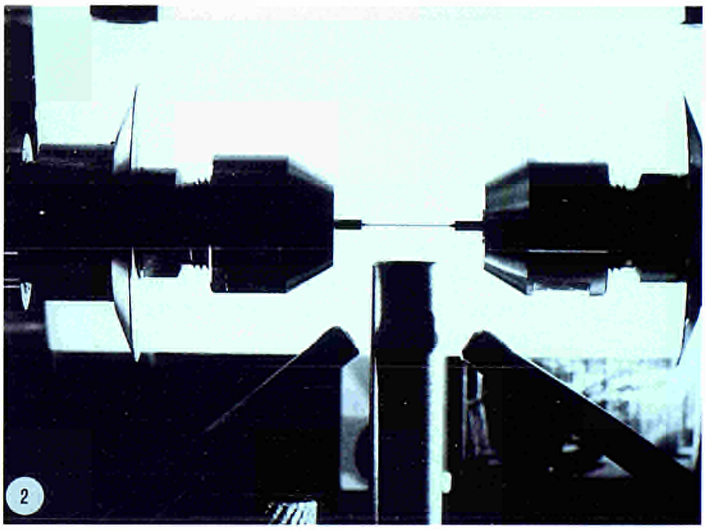
Expertises and technical services:

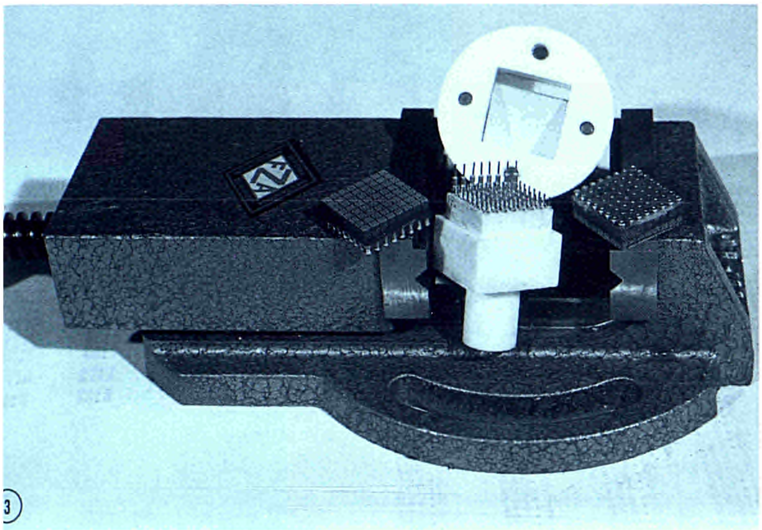
- {} Conception, fabrication and validation of reference defects (micro and macro) and of reference blocks.
- {} Conception and realization of special ultrasonic instrumentation for particular uses.
- {} Characterization of X-rays and ultrasonic instrumentation for particular applications.
- {} Studies on ultrasonic propagation in materials as stainless steels, composites and ceramics.
- {} Critical analysis of Non Destructive control procedures: capability, reliability.
- {} Modelling of defects; modelling of ultrasonic instrumentation.
- {} Role of Reference laboratories for NDE at the national and international levels (non destructive and destructive evaluation).
- {} NDE Data recording, illustration and analysis (engineering and statistical evaluation, uses of software packages).
- {} Support to Codes and Standards bodies.

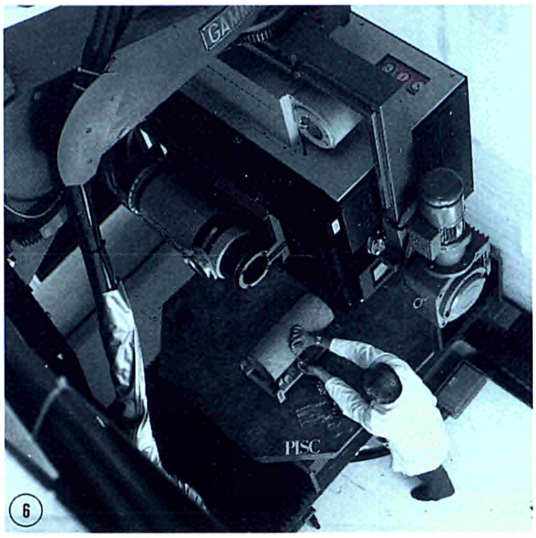
Figure 1: *Micro-spark erosion*

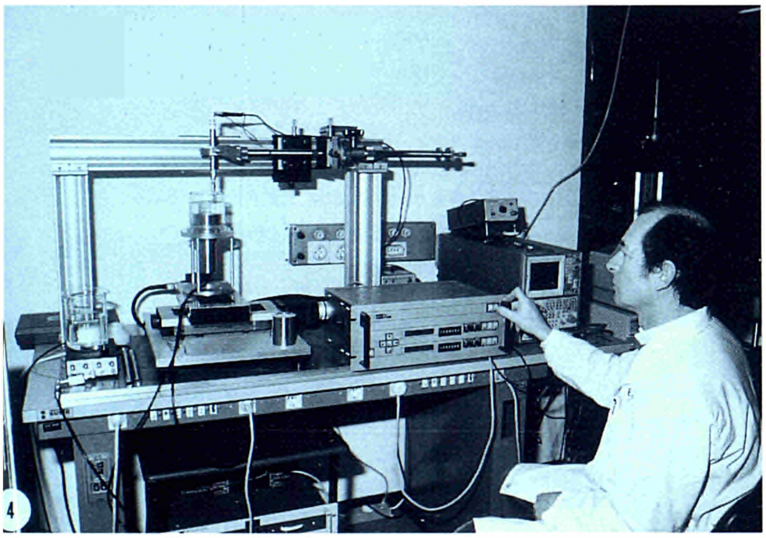
Figure 2: *Fabrication of a micro test-piece in austenic steel (diameter: 15µm, length: 10 mm)*











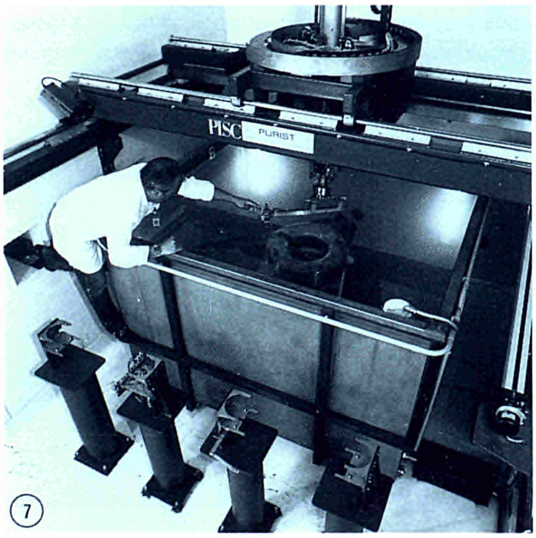


Figure 3: *Examples of a transducers array with 64 elements (NDE laboratories patent); each square element is an ultrasonic transducer, having 2.5 mm size and a nominal frequency of 5 MHz.*

Figure 4: *Calibration of hydrophones, using an ultrasonic reference transducer (NDE Laboratories patent).*

Figure 5: *Characterization of Materials. Measurement of acoustic properties.*

Figure 6: *1 and 2 MeV Linear Accelerator*

Figure 7: *High sophisticated and remote controlled ultrasonic bench.*

Advanced Materials Information Systems

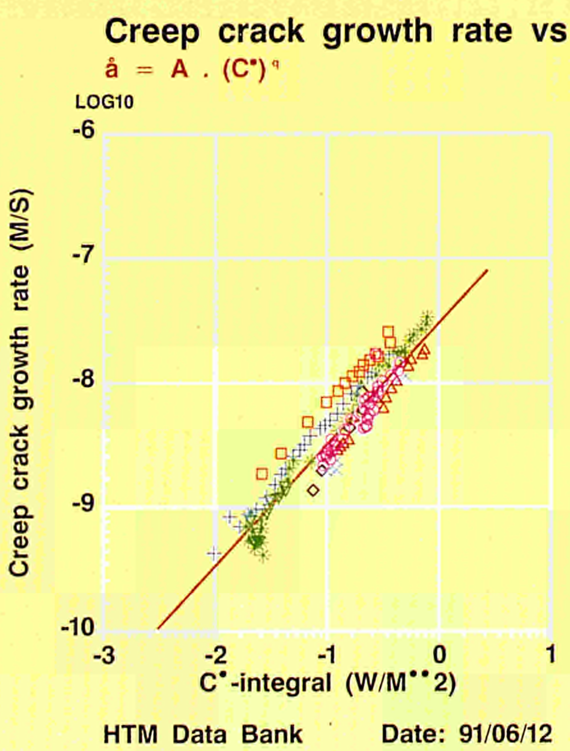
The Institute for Advanced Materials has some ten years experience in research and development for materials databanks.
Based on this experience it can provide a central knowledge pool for this field to the services of the Commission.
This experience has its principal basis in the research and development activity carried out since 1980 for the High Temperature Materials Databank (HTM-DB). The HTM-DB was one of the early projects in the EC which demonstrated the computerization of materials data for engineering in a system providing remote access via public digital data networks.
R&D activities in this field have enabled the Institute to initiate and contribute to Community projects and international collaboration.
The following areas of particular knowledge and experience are available in the two Institute sites at Petten and Ispra who are sharing tasks in this R&D.

1) The High Temperature Materials Databank

The High Temperature Materials Databank (HTM-DB) is an online computer databank of mechanical property test results for metallic materials combined with a program library for data modelling and evaluation relevant to high temperature engineering. The data are from engineering alloys used in components of conventional and nuclear power plants, steam and gas turbines and petrochemical plants. Data can be stored for the following types of mechanical tests:

- tensile
- creep
- relaxation
- fatigue
- fracture toughness
- impact
- creep crack growth
- fatigue crack growth

Below: *An example for a HTM-DB evaluation programme display*



PDOL. No.	Symbol	Ref. No.	Temp. (C)	Material
1	△	6030301	550.0	ALLOY 800
1	▽	6030401	550.0	ALLOY 800
1	+	6030501	550.0	ALLOY 800
1	×	6030701	550.0	ALLOY 800
1	□	6031101	550.0	ALLOY 800
1	◇	6031201	550.0	ALLOY 800
1	○	6030201	550.0	ALLOY 800
1	✳	6030601	550.0	ALLOY 800

PDOL. No.	Line style	A	q
1	——	3.045E-08	0.987

These data have been collected from open literature and publications, from tests in our laboratories, from tests of partner laboratories and from European and world wide joint projects such as COST 50, COST 501, COST 505, BRITE 1209 and VAMAS. The access to some of these data pools is restricted to the collaborating parties who generated the data. The databank is hosted in the Amdahl mainframe of the Joint Research Centre in Ispra and accessed via the public packet switched network (X25). It has a three-level data protection and selective user authorization system.

Searching is menu-assisted and linked to the menu-driven programme library which offers options for evaluation and modelling of the retrieved data. A special PC-based interface which adds extra user-friendliness to the system is available to online users. Output is obtained immediately in the form of reports, tables and graphs. The HTM-DB uses an interactive data input programme operating on user PCs and comprising checking routines and thesaurus control. The HTM-DB offers the following technical services:

- Access to data and evaluation methods of the HTM-DB;
- Data information and evaluation services;
- Storage and evaluation of customer data (private and restricted access files);
- Data management for cooperative projects;
- Help desk functionality.

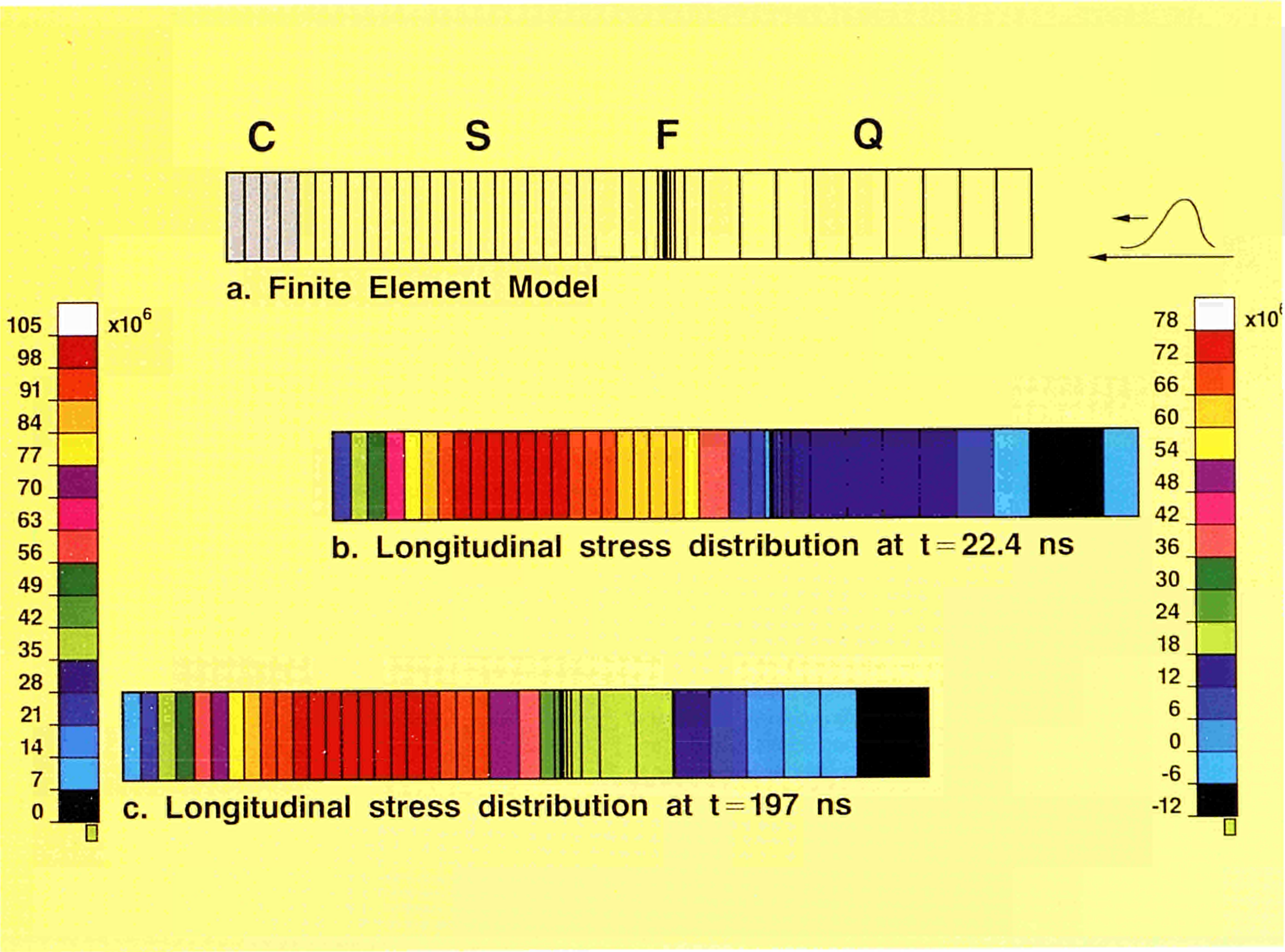
2) Data Evaluation

Data on the mechanical properties of engineering alloys are determined by testing specimens of materials in the laboratory. Unlike physical properties they are not defined in relation to universal physical concepts but by reference to their testing methods and standards. Such data from test results need evaluation by numerical modelling of constitutive assumptions and statistical methods to yield material property parameters directly usable in CAE analysis and design. Data evaluation can provide a basis for reliable interpolation, and in certain cases extrapolation, for the prediction of material data under conditions for which no test results are available. There are currently numerous evaluation techniques and statistical methods used for the determination of a wide scope of high temperature mechanical properties.

The evaluation programme library of the HTM-DB contains some 70 methods which can be accessed interactively, in a user friendly manner, from a menu. The output obtained from the evaluation programmes is a combination of reports on numerically predicted values in tabular or colour graphical form. Some of the routines are spline or linear regression programmes, whereas other modules enable the user to calibrate constitutive relations. The following list shows the nature and number of routines available in the HTM-DB.

Computerized Evaluation Methods, Number of HTM-DB Routines

Tensile Properties	
- tensile curve	(1)
- temperature dependence	(5)
- strain rate dependence	(4)
- load rate dependence	(4)
Creep, Stress Rupture	
- stress/strain/time relations	(6)
- time/temperature relations	(5)
- creep curves	(6)
- constitutive equations	(6)
Fatigue	
- cyclic stress-strain diagrams	(3)
- fatigue correlations	(10)
Creep-Fatigue Interaction	
- damage methods	(3)
- strain rate partitioning	(1)
Fracture Toughness	
- fracture toughness curves	(3)
Creep Crack Growth	
- creep crack growth curve	(4)
- creep crack growth rate	(3)
Fatigue Crack Growth	
- fatigue crack growth curve	(1)
- crack propagation models	(2)
Physical Properties	
- temperature dependence	(8)
Statistical Methods	



Above: *Numerical analysis of transient heat transfer and associated elastic wave propagation of a laser pulse through a quartz-absorbing film-substrate-coating compound*

3) Mathematical Modelling of Advanced Materials for Engineering Analysis

The development of material behaviour subroutines based on the contents of modern materials information systems requires more advanced mathematical modelling of nonlinear constitutive equations in the context of transient finite element analysis methods. State of the art constitutive theories must be employed for the behaviour of structural materials under complex conditions such as viscoplastic behaviour under combined creep-fatigue at high temperatures and conditions of irradiation.
A subroutine for the viscoplastic behaviour of materials under high strain rate has been recently linked with the general purpose finite element method code ABAQUS.

Plans have been made to enrich the HTM-DB with fusion material data and their evaluation will require major numerical work. In addition systems will be developed to host interface properties.

In fact the numerical technique is already under development using ABAQUS, for the simulation of an experimental method for measurement of interface strength based on a laser induced spallation concept. The development of this experimental technique is under preparation and requires good estimates of mechanical and thermal properties of thin films in situ.

4) Know-how Related to Materials Data Systems and Standards

The IAM has acquired extensive competence in R&D on materials data systems and standards during the past ten years of activity. It has been readily recognized and is now well established that internationally harmonized standards in a number of areas are needed in order that an unimpeded flow of materials information can be developed in the media provided by computer and telecommunication technologies. The areas concerned comprise, on the one hand, the description of materials and properties, the methods and conditions of testing, the methods of analysis and evaluation, i.e. standards related to the contents of the data systems, and, on the other hand, the features of architecture, operation and interaction of these systems.
Relevant joint activities with IAM participation have been organized by ISO, ASTM, CODATA, VAMAS and European institutions.

The IAM has contributed to, and is currently involved in, some of these collaborations. The first general review of the need for standards for materials data systems by VAMAS was strongly based on Petten work. The problem of materials data interchange was investigated in cooperation with ASTM and VAMAS. Methods and models for computerized data evaluation are being investigated and catalogued in cooperation with VAMAS and the Japanese NRIM.

In the Materials Database Demonstrator Programme of the CEC contributions were made to the development of a Code of Practice for the operation of data systems, and a Common Reference Vocabulary. The observation that user interfaces of materials data systems are sensitive issues by which the acceptance of these systems can be conditioned, led the IAM to design the "PCInterface" which has meanwhile become the standard interface for the HTM-DB. This remote shell requires minimal user training, performs automatic logon and logoff and uses advanced windowing techniques to assist the user in formulating queries. Typing mistakes and non-relevant queries are avoided as the user selects from lists of allowed terms. The interface has been designed in such way that the user can store on his PC all commands and all retrieved information, both text and graphics, of a session for later use. The interface can be readily tailored to fit other databases as well.

High Flux Reactor Petten

The High Flux Reactor (HFR) at Petten is a 45 MW pool type materials testing reactor with light water cooling and moderation. Within the core 17 positions are available for irradiation experiments and a further two are located in the reflector inside the reactor tank. Two pool side facilities, both with provisions for moving irradiation experiments in the flux gradient facilitate the execution of fuel irradiations under transient conditions. In addition, twelve radial beam tubes of different cross sections are available.

The programme executed in the HFR addresses fuel and structural materials irradiations for LWR's, HTR's, FBR's as well as fusion devices, fundamental and applied research with neutrons at the beam tubes, production of radioisotopes and processing with neutrons, activation analysis and neutron radiography. Experimental equipment has been developed to meet these programme requirements and has been continuously adapted and improved in order to satisfy market demands.

For irradiation testing of water reactor fuel rods a variety of different types of irradiation capsules are available. The capsules provide typical water reactor conditions at the fuel rod surface (e.g. temperature, system pressure and coolant) and are equipped with a variety of instrumentation for dimensional-, temperature- and pressure- measurement or axial load simulation. The irradiation devices are designed for in-core or pool side facility operation. Power control to simulate transient conditions is performed by adjustable absorber screens using BF_3 in the in-core devices and position adjustment in the pool side facility.

Most of the devices are reloadable and, thus, allow intermediate inspections of the fuel rods by HFR pool-based NDT equipment (e.g. eddy current and profilometry system, neutron radiography). For the investigation of fuel rods or fission product behaviour under hypothetical accident conditions of a LOCA a new irradiation device became available. Re-instrumentation and refabrication capabilities are introduced at the Petten hot cells for pre-irradiated water reactor fuel.

For the investigation of irradiation induced changes of physical and mechanical properties of HTR graphites, standardized rigs are available for irradiating unstressed samples in the temperature range from 300°C to 1100°C with the option of re-irradiating samples after intermediate measurements. Rigs to study creep under tensile and compressive stress cover the temperature range from 300°C to 900°C. Recent design improvements have led to easier loading and unloading and to substantial reductions in manufacturing costs.

Left: *BWFC installation*

Middle: *Gas mixing station*

Right: *Tritium measuring station*

Above: *Unloading of irradiated U 235 samples from a core position*

Irradiation testing of coated fuel particles and fuel elements, either spherical or segments for HTR's is performed in rigs which can be operated between 600°C and 1500°C. The capsules are fully instrumented and temperature controlled and attached to a sweep gas system for the quantitative assessment of volatile fission products release. As an option, the sweep gas can be doped with controlled quantities of impurities, for example water vapour.

The programmes related to radiation damage studies and mechanical property changes of the materials used for structural components of FBR's and fusion machines make use of standard irradiation devices, where the specimens are submerged into sodium for close temperature control in the range from 400°C to 700°C.

For FBR fuel testing rigs are available for in-core and pool side facility positions. They have the potential for high linear heat generation rates up to 750 W/cm to study power-to-melt and overpower steady-state performance. The option of flux tailoring by means of a cadmium screen is of advantage for specific spectrum hardening. In the pool side facility power cycling, start-up and shut-down ramps and other transient experiments on fresh and pre-irradiated test fuel rods with the option of intermittent measurement of fuel to clad interaction and on-line measurement of fuel to clad axial elongation during transients is possible. Fuel to cladding gap conductance measurement by noise analysis is under development. The available α-tight EUROS cell, used to encapsulate pre-irradiated fuel pins, has been re-designed to accept longer (> 2.1 m) pins.

Irradiation rigs have been developed for the study of creep of metals under constant load. Measurement is either intermittent in-pile or after intervals of irradiation. Current applications are for first wall fusion materials where the temperature is in the range 300°C to 500°C. A device for studies on thermal fatigue combined with irradiation creep on first wall materials is under development. Special capsules are available to reach a He/dpa ratio typical of fusion reactors. They can operate in the range 180°C to 500°C (for stainless steel samples).
Devices to irradiate first wall coating materials are available. Samples can be irradiated in the range 300°C to 800°C. Special sample holders have been conceived to irradiate divertor materials (80°C to 700°C) and brazed samples at low temperature (80°C).

Tritium release kinetics of potential blanket breeder materials for fusion devices can be investigated by means of swept capsules. Devices for both lithium based ceramics and liquid metal (Pb-17Li) breeder concepts are available. Tritium release is measured in-situ and on-line.

The beam tube HB11 has been re-configured to deliver a predominantly, parallel, epithermal neutron beam in order to study the feasibility of implementing clinical trials on cancer patients using the method of boron neutron capture therapy. At HB7, a prompt gamma-ray spectroscopy system has been installed to determine the boron content of radiobiological specimens irradiated at HB11. A nuclear polarization facility is available at the polarized thermal neutron beam HB2.

Five beam tubes - HB1, 3, 4, 5 and 9 - are in permanent use for neutron scattering investigations. Spectrometers using neutron diffraction, critical scattering, diffuse scattering, inelastic scattering and small-angle scattering are in operation.

The high thermal neutron flux density in the HFR of up to $5 \times 10^{18} m^{-2} s^{-1}$ is ideal for the production of radioisotopes. General purposes devices are available mainly for the production of Ir-192 and Y-90, Xe-133, etc. Dedicated devices are used for cobalt irradiations and for fissile targets to produce Tc-99m. A rotating device giving a uniform neutron fluence distribution in the target is also available.

Four irradiation facilities for neutron activation analysis are routinely in operation. Two of these are located in the pool side facility, one stationary and the other rotating, the remaining two systems are pneumatic shuttle systems.

For non-destructive testing of components and materials neutron radiography is providing a unique and complementary inspection method. Services and studies are offered by the Petten Neutron Radiography Services, PNRS, a joint service of JRC and ECN. PNRS's facilities include the HFR underwater camera for radioactive objects, a neutron radiography facility with filtered neutrons in the HFR containment building and a neutron radiography facility with a thermal neutron beam at the Low Flux Reactor (LFR), both for non-nuclear applications.

New systems for dynamic neutron radiography and image analysis are important additional tools of the PNRS. Development activities addressing tomographic imaging are in progress.

The HFR itself as well as the ancillary and experimental equipment have been continuously upgraded and modernized. In combination with the services from JRC and ECN in support of preparing, performing and evaluating large size irradiation programmes, a full service package is at the disposal of HFR users.

The main characteristics of the HFR and the experimental facilities are described in detail in ref. /1/. The programme achievements are regularly reported in a separate annual report /2/.

References

/1/ H. Röttger, A. Tas, P. von der Hardt, W.P. Voorbraak
High Flux materials testing reactor HFR Petten, characteristics of facilities and standard irradiation devices
EUR-report 5700, 1986

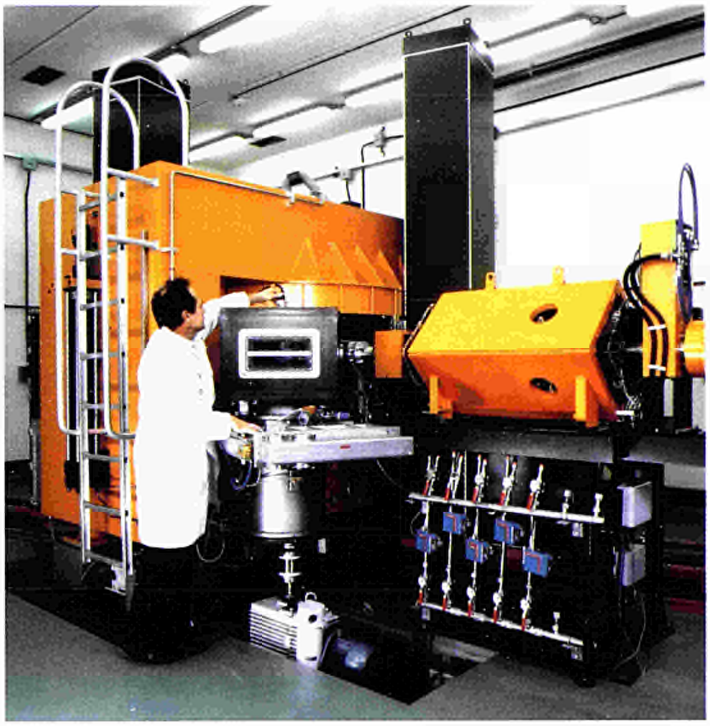
/2/ Annual Report 1990,
Operation of the High Flux Reactor
EUR-report 13590 EN

Cyclotron Laboratory

The Cyclotron Laboratory project at the JRC-Ispra was elaborated in 1976, and approved by the Council of Ministers in July 1977.
The industrial contract called for the supply of a special version of a 40 MeV cyclotron.

The Accelerator

MC-40 is a variable energy light ion cyclotron. The accelerator system consists of two separate indentical RF cavities with RF power amplifiers placed on each side of the cyclotron magnet. The cavities are λ/4 type with 90^0 dees. The stems pass through the vacuum chamber via a flange with pipe supporting insulators (Al_2O_3) and are bent vertically in order to reduce the horizontal extension of the system.
The whole acceleration structure inside the vacuum chamber can easily be reached when the upper yoke of the magnet is raised by means of a hydraulic lifting system. The RF power amplifiers are directly connected to the cavities and can easily be removed.
The fundamental technical and physical data are summarised in Tables I and II. In figure above a view of the cyclotron with the first q-pole triplet is shown.



Above: *MC-40 accelerator*

Table I: Characteristics of the Ispra MC-40 Cyclotron

Pole diameter	115 cm
Magnet weight	60 tons
Main coils max. current	850 A
Sectors	3
Max. magnetic field	2.1 Tesla
Extraction radius	50 cm
Dees	2, 90^0
RF range	12.5 - 27 MHz
Frequency stability	$1x10^{-4}$
Amplitude stability	$1x10^{-3}$
Ion source	PIG Type
Cathode lifetime	2 weeks (p,d)
	50 hours (alfa)
Current stability	~ 1.5% (short time)

Table II: Particle beam intensities

Particles	E-range (MeV)	Max extr. beam current (µA)	Energy Spread ΔE/E/
Protons	10-38	65	0.005
Deuterons	5-19	65	0.01
Helium-4	10-38	30 at max energy	0.01

Auxiliary Systems

The accelerator is supported by a number of service systems, viz.:
- ion source;
  PIG cold cathode discharge with easily exchangeable cathodes;
- beam diagnostics
  . two radial beam probes for controlling the extraction efficiency;
  . profile monitors and Faraday cups in the individual beam lines;
- control, safety (interlock) and alarm circuits.

Instruments and controls are centralised in the control room.

Experimental Facilities

The extraction of the beam is obtained through an electrostatic deflector. A steering magnet adjusts the horizontal position of the extracted beam at the center of the exit port.
Extracted efficiencies measured at the target typically range from 50 to 80% and depend on the type of ions and on their energy.
The specific irradiation equipment presently in use comprises:

- irradiation chambers for proton damage or helium implantation in material specimens, some with connected in-beam creep or fatigue crack growth apparatus;
- radioisotope production station.

Specimens can be cooled directly by jets of purified helium from a closed loop system of indirectly by water-cooled support plates.
Temperature measurement is accomplished by means of thermocouples and pyrometers.

Access to Facility

The Ispra Cyclotron Laboratory is available for:

- experiments performed in the framework of the European Fusion Programme,
- Experiments performed in collaboration with outside laboratories,
- Third Parties Work under contract.

Staff members of the Cyclotron Laboratory provide the following services:

- Operation the Cyclotron,
- Obtaining beam spots on the target of the desired shape,
- Control of beam profile in order to obtain the desired homogeneity,
- Irradiation of samples.

Support to Experimenters

Experimenters can use all existing facilities. Simple irradiation chambers are built for new applications, whereas more sophisticated equipment is usually supplied and operated by the experimenter, assisted by laboratory staff. Support is also available from a machine shop and electrical/electronic engineers. As far as software support is concerned the laboratory supplies all required beam energy and measurement calculations, beam profile control, and experiment safety analysis.

Building

The cyclotron building (Figure below) is divided into the laboratory wing which is a radiological controlled area and an office area.
The accelerator cubicle and the irradiation cells are shielded by 2 m of high density concrete.

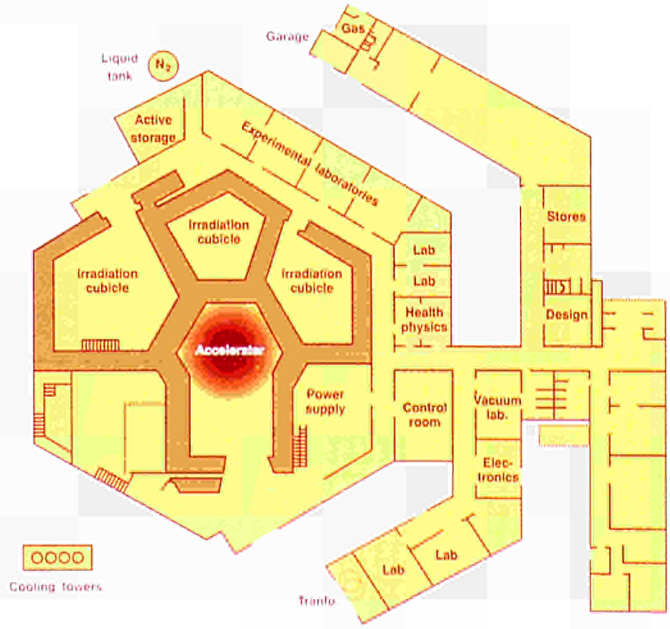
Utilisation

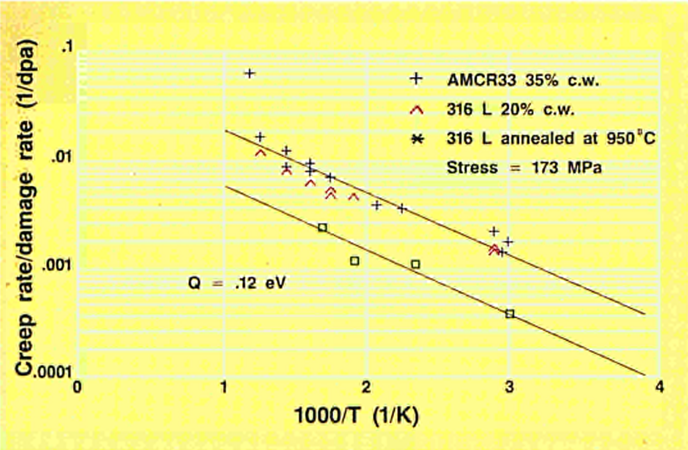
Fusion Reactor Materials:
The Ispra Cyclotron has been employed mostly as a facility for studies of radiation damage in fusion reactor materials, both as displacement and gas (hydrogen and helium) production damage. A large number of material tests in the scope of the European Fusion Technology Programme are presently under way (see also page 71).

They include:

- Basic studies on the kinetics of displacement damage,
- Irradiation of AISI 316 and AMCR samples for post-irradiation measurements of mechanical properties,
- Deuteron irradiation creep of AMCR-0033 and AISI 316L stainless steel.

Below: *Building D50-D50a Cyclotron Laboratory*





- The temperature dependence of the irradiation creep rate has been determined for type 316L stainless steel in both 20% cold worked condition and after annealing at 950°C. The AMCR sample material was 35% cold worked. The results are shown in figure above which shows:
  - for all three materials a linear relationship exists between log (β) and 1/T with a slope which corresponds to an activation energy of 0.12 eV assuming an Arrhenius type law for the irradiation creep process,
  - the AMCR sample and the 20% c.w. 316 L sample shows a very similar irradiation creep deformation for equal experimental conditions,
  - the annealed 316 L sample deforms considerably slower than the two other materials which are cold worked.
- Fundamental radiation studies on SAP, ceramics, vanadium, vanadium alloys and numerous other materials.
- Irradiation of copper and tungsten samples for post-irradiation examination of induced damage.
- In-beam fatigue crack propagation experiments on AMCR steel samples; following the NET requirements a series of in-beam fatigue crack growth experiments on AISI type 316 have been started.

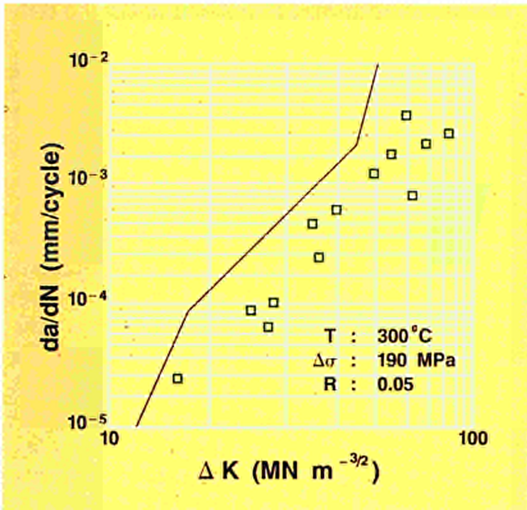
  The irradiation tests will be conducted at 300, 200 and 100°C with 18 MeV protons producing a damage rate of the order of 10^{-7} dpa s^{-1}.

  In figure below a typical result of tests conducted under irradiation at 300°C is presented. The average crack growth per cycle da/dN has been plotted against the stress intensity factor range ΔK. The results are not different from those obtained at 500°C, where at increasing growth rates a gradual deviation of the data has been observed for the irradiated specimens. Evidence of this may also be seen in figure opposite below in the crack growth curves which shows that fatigue lives under irradiation at 300 and 500°C are not very different.

  This preliminary result indicates a slightly shorter fatigue life at 300°C in AISI type 316 steel. Also in the present case it appears that about 20 or 30% of the crack extension occurred in the first 80% of the total cycles.
- Helium implantation into vanadium and vanadium alloy samples for subsequent neutron irradiation in a fission reactor.
- Cross section determination using high energy neutrons produced through the (p,n) reaction in ^{7}Li.

Above: *Temperature dependence of the irradiation creep rate*

Below: *Typical result of tests conducted under irradiation at 300°C*



Environment Studies

Another field of Cyclotron activities concerns the production of isotopes, mainly used as tracers in biological research by the Ispra Environment Programme and for other scientific/technical applications.

In particular a number of radioisotopes are produced mostly via (p,xn) reactions using solid or liquid targets. The principal radioisotopes produced are 201-Tl, 48-V and 206-Bi.

Radioisotopes for nuclear medicine and biology:

A new activity in the laboratory is the production of radioisotopes for use in nuclear medicine.

Research has been performed for the production of the radioisotope 68-Ge and the development of a generator system 68-Ge/68-Ga.

This generator can be used in medical diagnostics as well as for the calibration of Positron Emission Tomography (PET) facilities.

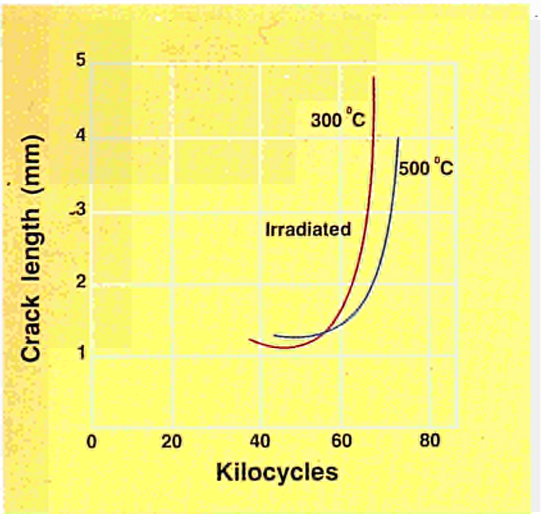
After the necessary studies for production and chemical separation of 68-Ge, irradiation experiments have been carried out to produce the 68-Ge isotope. Quality control by gamma-spectrometry of the irradiated targets is made to check the samples for radionuclide impurities and for the relative intensity of 68Ga.

The main development for the coming three to four years is the set-up of radioisotope production for medical and scientific purposes.
In this context the production of high purity 123-I is foreseen in collaboration with a radiopharmaceutical firm. This new line of activity is important not only for its commercial aspect but also (and principally) for its social aspect.

Proton Nuclear Activation Analysis (PNA)

New applications in the field of proton nuclear activation analysis (PNA) are being developed:

1) Experiments were performed (in collaboration with the University of Milan) to determine elements with low concentrations, for example, iron and molybdenum in the biological field, especially in the human organism. An irradiation chamber with a rotating target holder which allows simultaneous irradiation of 29 samples is used.
2) The thin layer activation method is a powerful tool for the sensitive and precise determination of wear in lubricated mechanical components. Measurements in vital parts of engines in the aeronautical sector (helicopters) is studied in cooperation with an external firm.
3) The Proton Induced X-Ray Emission (PIXE) analytical method was studied and first measurements with an experimental facility were performed in collaboration with the University of Milan and the CNR - Milan.

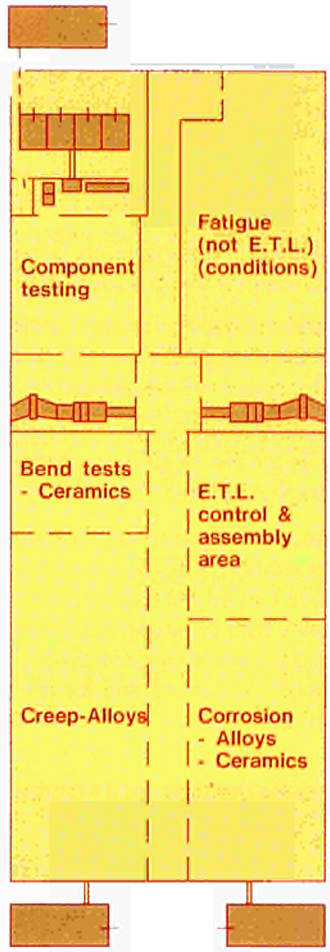


Below: *Fatigue life under irradiation at 300°C and 500°C*

Environmental Testing Laboratory

The Environmental Testing Laboratory was conceived and constructed to enable materials testing in all parts of the programme which involved aggressive and potentially hazardous atmospheres to be conducted under conditions of maximum safety and security.
It is designed in such a way that all experimental rigs can be attached to a pier system which provides all the supplies necessary to run the experiments.
Thus from a common source each experiment has at its disposal a supply of test gas, inert blanket gas, 380V 3 phase, 220V AC and 24V DC electrical supplies, cooling water and an exhaust for the test gas after passing through the rig.
All of these supplies are passed to the experiment through a local control cabinet which ensures safety both for the personnel and for the experiment.
In the past year new uninterruptible power sources have been provided for the control and safety circuits.
The test gases are supplied from batteries of premixed gas held in external gas stations and piped around the ETL.
Because most of these gases contain explosive and/or toxic components (H_2, CO, H_2S, SO_2) the rig areas and the general laboratory space are monitored continuously for any leakage.
The space in the ETL is currently allocated between various test disciplines as shown in figure above.
A total of 51 test points are available which are used for work within the Specific Programme and for third-party contract research.
Associated and linked to the ETL is the Component Testing Facility. There, a similar philosophy applies to all safety related matters.
This unit, however, requires a very close integration of facility and experiment and is dedicated to the work of the Component Testing sub-group.

Above: *Distribution of test areas in E.T.L. during 1990*



Surface Modification Centre

Following a decision of the European Parliament, the IAM has installed in its Ispra site a Surface Modification Center. The center is intended to assemble in a single laboratory the most important techniques for the treatment of solid surfaces in order to study not only the possibilities of each single method, but to investigate the technological potential of the combination of these techniques. It is evident that in addition to the equipment for the preparation of new surface structures, the center needs to install and to develop methods for investigation and characterisation of the different surface properties.

The funds made available by the European Parliament have been used to buy new equipment, which has been combined with existing installations. Currently the SMC has the following configuration.

A. Installations for Surface Modification

1. Ion Implanter
   200 kV, 1 mA, magnetic mass separator, ion sources: gas, high temperature and sputter source which facilitate the implantation of all elements, surfaces 40 x 40 cm.
2. Carbon dioxide Laser
   5 kW output, x-y table, surfaces 50 x 50 cm, for surface melting and alloying.
3. Electron Gun
   30 kW electron beam, electromagnetic deflection, for surface melting and alloying, surface 4 x 4 cm.
4. Plasma Sputter Coating Device
   RFD, DCM, RFM sputtering, 3 targets allow to produce sandwich layers of different elements without breaking the vacuum, surfaces 15 cm diameter.
5. Vacuum Plasma Spray Unit
   for the production of surface coatings from powder material of different compositions, surfaces 50 x 50 cm.

B. Installations for Surface Analysis and Characterisation

1. AUGER Scanning Microscope
   RIBER Nanoscan 50
   High spatial resolution (50 nm) duoplasmatron for depth profile (~3 µm Ø).
2. PERKIN ELMER - ESCA-SAM 560
   High sensitivity and reliability for XPS measurements. Auger and XPS depth profiles by ion sputtering.
3. Transmission Electron Microscope
   JEOL 200 CX with EDS and EEls analysis systems.
4. Scanning Electron Microscopes
   PHILIPS 505 with EDS System
   JEOL 6400 F high resolution microscope (ordered).
5. Glancing Angle X-ray analysis
   Surface structure (range 100-10,000 Å, surface density and thickness).
6. Metallographic Laboratory.
7. Surface Hardness Measurements
   Nano indenter.
8. HT oxidation test loop for corrosion in gases.
9. Laboratory for Electrochemical Tests
   (in preparation).
10. Laboratory for Wear and Friction Measurements
    (in preparation).
11. GAERTNER Ellipsometer
    (Thickness measurement of thin films, optical constant).

European Communities - Commission

EUR 13801 EN — Institute for Advanced Materials, Annual Report 1990

M. Merz, editor

Luxembourg: Office for Oficial Publications of the European Communities

1990 - 169 pages - 21.0 x 29.7 cm

Series: Physical Sciences

EN

Catalogue number: CD-NA-13801-EN-C

Abstract

The Annual Report 1990 of the Institute for Advanced Materials of the JRC highlights the Scientific Technical Achievements and presents in the Annex the Institute's Competence and Facilities available to industry for services and research under contract.
The Institute executed in 1990 the R & D programme on advanced materials of the JRC and contributed to the programmes: reactor safety, radio-active waste management, fusion technology and safety, nuclear fuel and actinide research.
The supplementary programme: Operation of the High Flux Reactor is presented in condensed form. A full report is published separately.

Acknowledgement is made of the following:

Coordination: **B. Seysener** and **F. van der Smaal**
Text Pre-processing: **Y. Hey**
Lay-out and Photosetting: **J. Manten**
Printing: **Casparie-Almere BV,** Almere, NL
Cover Design: **J. Wells**

Venta y suscripciones • Salg og abonnement • Verkauf und Abonnement • Πωλήσεις και συνδρομές
Sales and subscriptions • Vente et abonnements • Vendita e abbonamenti
Verkoop en abonnementen • Venda e assinaturas

BELGIQUE / BELGIË

Moniteur belge / Belgisch Staatsblad
Rue de Louvain 42 / Leuvenseweg 42
1000 Bruxelles / 1000 Brussel
Tél. (02) 512 00 26
Fax 511 01 84
CCP / Postrekening 000-2005502-27

Autres distributeurs / Overige verkooppunten

Librairie européenne/ Europese Boekhandel
Avenue Albert Jonnart 50 /
Albert Jonnartlaan 50
1200 Bruxelles / 1200 Brussel
Tél. (02) 734 02 81
Fax 735 08 60

Jean De Lannoy
Avenue du Roi 202 /Koningslaan 202
1060 Bruxelles / 1060 Brussel
Tél. (02) 538 51 69
Télex 63220 UNBOOK B

CREDOC
Rue de la Montagne 34 / Bergstraat 34
Bte 11 / Bus 11
1000 Bruxelles / 1000 Brussel

DANMARK

J. H. Schultz Information A/S
EF-Publikationer
Ottiliavej 18
2500 Valby
Tlf. 36 44 22 66
Fax 36 44 01 41
Girokonto 6 00 08 86

BR DEUTSCHLAND

Bundesanzeiger Verlag
Breite Straße
Postfach 10 80 06
5000 Köln 1
Tel. (02 21) 20 29-0
Fernschreiber:
ANZEIGER BONN 8 882 595
Fax 20 29 278

GREECE

G.C. Eleftheroudakis SA
International Bookstore
Nikis Street 4
10563 Athens
Tel. (01) 322 63 23
Telex 219410 ELEF
Fax 323 98 21

ESPAÑA

Boletín Oficial del Estado
Trafalgar, 27
28010 Madrid
Tel. (91) 446 60 00

Mundi-Prensa Libros, S.A.
Castelló, 37
28001 Madrid
Tel. (91) 431 33 99 (Libros)
431 32 22 (Suscripciones)
435 36 37 (Dirección)
Télex 49370-MPLI-E
Fax (91) 575 39 98

Sucursal:

Librería Internacional AEDOS
Consejo de Ciento, 391
08009 Barcelona
Tel. (93) 301 86 15
Fax (93) 317 01 41

Generalitat de Catalunya:

Llibreria Rambla dels estudis
Rambla, 118 (Palau Moja)
08002 Barcelona
Tel. (93) 302 68 35
302 64 62

FRANCE

Journal officiel
Service des publications des Communautés européennes
26, rue Desaix
75727 Paris Cedex 15
Tél. (1) 40 58 75 00
Fax (1) 40 58 75 74

IRELAND

Government Publications Sales Office
Sun Alliance House
Molesworth Street
Dublin 2
Tel. 71 03 09

or by post

Government Stationery Office
EEC Section
6th floor
Bishop Street
Dublin 8
Tel. 78 16 66
Fax 78 06 45

ITALIA

Licosa Spa
Via Benedetto Fortini, 120/10
Casella postale 552
50125 Firenze
Tel. (055) 64 54 15
Fax 64 12 57
Telex 570466 LICOSA I
CCP 343 509

Subagenti:

Libreria scientifica Lucio de Biasio - AEIOU
Via Meravigli, 16
20123 Milano
Tel. (02) 80 76 79

Herder Editrice e Libreria
Piazza Montecitorio, 117-120
00186 Roma
Tel. (06) 679 46 28/679 53 04

Libreria giuridica
Via XII Ottobre, 172/R
16121 Genova
Tel. (010) 59 56 93

GRAND-DUCHÉ DE LUXEMBOURG

Abonnements seulement
Subscriptions only
Nur für Abonnements

Messageries Paul Kraus
11, rue Christophe Plantin
2339 Luxembourg
Tél. 499 88 88
Télex 2515
CCP 49242-63

NEDERLAND

SDU Uitgeverij
Christoffel Plantijnstraat 2
Postbus 20014
2500 EA 's-Gravenhage
Tel. (070) 378 98 80 (bestellingen)
Fax (070) 347 63 51
Telex 32486 stdru nl

PORTUGAL

Imprensa Nacional
Casa da Moeda, EP
Rua D. Francisco Manuel de Melo, 5
P-1092 Lisboa Codex
Tel. (01) 69 34 14

Distribuidora de Livros Bertrand, Ld.ª
Grupo Bertrand, SA
Rua das Terras dos Vales, 4-A
Apartado 37
P-2700 Amadora Codex
Tel. (01) 493 90 50 - 494 87 88
Telex 15798 BERDIS
Fax 491 02 55

UNITED KINGDOM

HMSO Books (PC 16)
HMSO Publications Centre
51 Nine Elms Lane
London SW8 5DR
Tel. (071) 873 9090
Fax GP3 873 8463
Telex 29 71 138

Sub-agent:

Alan Armstrong Ltd
2 Arkwright Road
Reading, Berks RG2 0SQ
Tel. (0734) 75 18 55
Telex 849937 AAALTD G
Fax (0734) 75 51 64

CANADA

Renouf Publishing Co. Ltd
Mail orders — Head Office:
1294 Algoma Road
Ottawa, Ontario K1B 3W8
Tel. (613) 741 43 33
Fax (613) 741 54 39
Telex 0534783

Ottawa Store:
61 Sparks Street
Tel. (613) 238 89 85

Toronto Store:
211 Yonge Street
Tel. (416) 363 31 71

JAPAN

Kinokuniya Company Ltd
17-7 Shinjuku 3-Chome
Shinjuku-ku
Tokyo 160-91
Tel. (03) 354 01 31

Journal Department
PO Box 55 Chitose
Tokyo 156
Tel. (03) 439 01 24

MAGYARORSZÁG

Agroinform
Központ:
Budapest I., Attila út 93. H-1012
Levélcím:
Budapest, Pf.: 15 H-1253
Tel. 36 (1) 56 82 11
Telex (22) 4717 AGINF H-61

ÖSTERREICH

Manz'sche Verlags- und Universitätsbuchhandlung
Kohlmarkt 16
1014 Wien
Tel. (0222) 531 61-0
Telex 11 25 00 BOX A
Fax (0222) 531 61-81

SCHWEIZ / SUISSE / SVIZZERA

OSEC
Stampfenbachstraße 85
8035 Zürich
Tel. (01) 365 51 51
Fax (01) 365 54 11

SVERIGE

BTJ
Box 200
22100 Lund
Tel. (046) 18 00 00
Fax (046) 18 01 25

TÜRKIYE

Dünya Süper Dagitim Ticaret ve sanayi A.Ş.
Narlibahçe Sokak No. 15
Cağaloğlu
Istanbul
Tel. 512 01 90
Telex 23822 DSVO-TR

UNITED STATES OF AMERICA

UNIPUB
4611-F Assembly Drive
Lanham, MD 20706-4391
Tel. Toll Free (800) 274 4888
Fax (301) 459 0056
Telex 7108260418

YUGOSLAVIA

Privredni Vjesnik
Bulevar Lenjina 171/XIV
11070 - Beograd
Yougoslavie

AUTRES PAYS
OTHER COUNTRIES
ANDERE LÄNDER

Office des publications officielles des Communautés européennes
2, rue Mercier
L-2985 Luxembourg
Tél. 49 92 81
Télex PUBOF LU 1324 b
Fax 48 85 73
CC bancaire BIL 8-109/6003/700

10/90

16 CD-NA-13801-EN-C

NOTICE TO THE READER

All scientific and technical reports published by the Commission of the European Communities are announced in the monthly periodical **'euro abstracts'.** For subscription (1 year: ECU 84) please write to the address below.

OFFICE FOR OFFICIAL PUBLICATIONS
OF THE EUROPEAN COMMUNITIES

L-2985 Luxembourg