ANNUAL·REPORT·89

Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

EUR 12272 EN

I N U A L · R E P O R T · 89

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

LEGAL NOTICE

Cataloguing data can be found at the end of this publication

Luxembourg: Office for Official Publications of the European Communities, 1990

ISBN 92 - 826 - 1329 - 1

Catalogue number: CD - NA - 12272 - EN - C

Printed in the Netherlands

Table of Contents

I Introduction

Introduction

Advanced Materials, Industry and Society

The industrial advancement of societies has always been predicated to the presence of appropriate materials. This is as true today as it has always been, and the advent of advanced materials that is, materials with specific functions as well as materials with improved and reliable structural performance will be rate-determining in the introduction of the new technologies that will in turn influence the shape of future society.

This has been recognised in all the technologically developed countries, most of which have established plans for placing emphasis and resources in materials technologies as part of a general global pattern of competition in the development and trade in science based products.
In turn, the work of the new Institute for Advanced Materials of the Joint Research Centre has been planned in order to contribute, within the capacities of the Institute, to the body of diverse effort required to meet the future challenge of advanced materials and with the broad objective of contributing to the industrial competitivity of Europe.

The Institute Structure of the Joint Research Centre

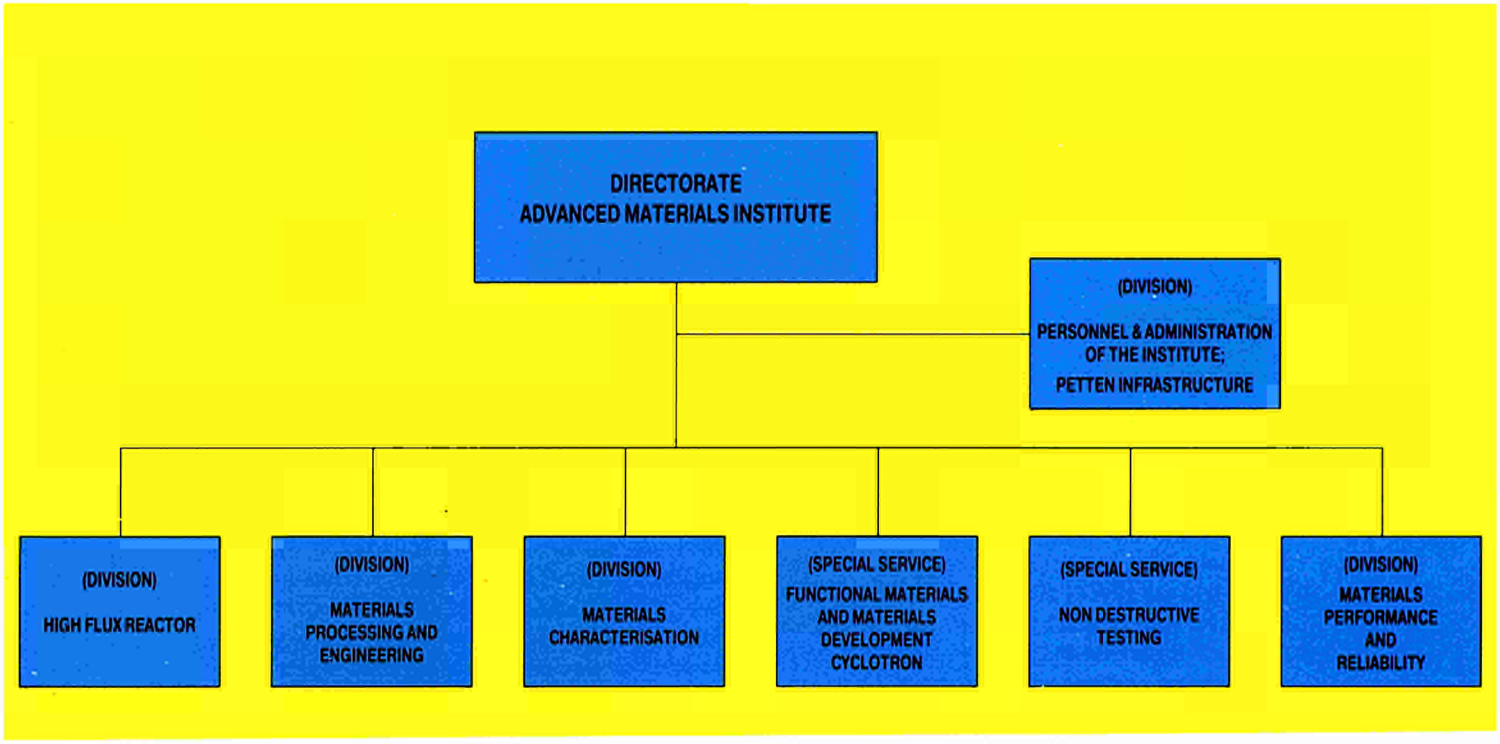
This represents the first Science and Technology Report of the Institute for Advanced Materials of the Joint Research Centre, which was launched at the beginning of 1989, and hence covers progress in work during this period.
The origins of the Institute for Advanced Materials lie in the response of the Commission of the European Communities to the report (Ref.1) of a body of leading industrial experts who reviewed the work of the Joint Research Centre in 1986. They recommended a radical transformation of the Centre with the declared aim of the JRC playing a more direct role in strengthening the industrial technologies and the industrial competitivity of Europe.
This represents a considerable departure from historical practice to such an extent that the Joint Research Centre will have to evolve a new R & D culture within the customer/contractor principle.
Consequently, this Centre has been restructured into a number of Institutes, the first general report (Ref. 2) on which has already been published.

In connection with materials R & D, this type of work does not represent an entirely new direction in the JRC, in fact, this has been carried out in one form or another since its foundation. However, much of the work on materials R & D was geared to the original nuclear role of the JRC, both in nuclear fission and thermonuclear fusion. There has been in addition the industrially oriented materials programme on high temperature materials at the Petten Establishment of the CEC, aimed specifically at strengthening the technologies associated with, for example, power engineering and aeronautical engineering.
In view of the new found role of Advanced Materials in the new format of the Joint Research Centre, with the emphasis on the industrial technologies, the Institute has been structured in order to embrace most of the materials research in the Joint Research Centre, in particular that in Petten and in the Ispra sites. A main reason for this is to obtain the benefit of size and range of expertise available in the two Centres, as well as the complementarity of competences. Hence the essence of the new Institute structure is one of coordination across the two geographical sites, allowing the planning and management of a comprehensive range of activities across the materials spectrum.

The organigram of the Institute shown schematically in the figure on page 4. It is composed of six Units, three in each of the sites. This arrangement responds to the need for a balance among the key materials R & D themes: Synthesis; Characterisation; Processing and Engineering; Materials Testing and Non Destructive Evaluation. Clearly these do not indicate sharp boundaries but a scheme of overlapping activities, and one should expect a vigorous interplay between the various Units as well as between the two sites of the Institute.

Nature of the Work of the Institute

Within its single management structure, the Institute for Advanced Materials is aiming to develop a style and a type of activity with a distinctive character, involving materials R&D of a high scientific level, embedded in the needs of industry and within the unique model of materials R&D on an European dimension, with the opportunity to harness the individual energies and backgrounds of scientists



from all member countries in order to procure a heightened scientific creativity.
The nature of the work of the Institute is thus determined by a number of constraints. Among these, there is the need for subsidiarity, that is, the work can be conducted well in the international setting of the Community laboratories and the projects derive an enhanced effectiveness from this Institutional approach; second, the projects must have an aspect of Community cohesion, that is, an awareness of a strong and united European response to the challenge of materials technologies. Among the elements that help forge the programme structure, there is the neutral and objective role of the Institute, which is independent of national frontiers and ideal for setting up projects aimed at the definition of advanced technical standards; there is the need to develop and exploit unique experimental facilities and other capabilities for the benefit of European industry; the need to disseminate freely and widely materials data and information to all Member States; finally, the need to help develop a supply of highly trained specialists for the newly developing materials based industries.

Specialised Materials Research Facilities

In addition to a wide network of advanced surface and bulk analytical techniques which are today the standard armoury of all advanced laboratories, such as Scanning Electron Microscopy, Transmission Electron Microscopy, X-Ray, Micro- Analysis, Rutherford Backscattering Analysis, Auger Electron Spectroscopy and X-Ray Photo-Electron Spectroscopy, the Institute has other unique facilities such as X-Ray Glancing Angle Spectroscopy, particularly suited for the study of the structure of thin surface layers and films.

In addition, there are some major installations, among which:

- the High Flux Reactor in Petten, of demonstrated value as a leading European Materials Test Reactor,
- the Environmental Testing Laboratory, specialising in High Temperature Materials Testing in aggressive environments,
- the Laser-Ion Foundry, recently installed in Ispra in which materials surfaces can be modified by an interplay of electron, ion and laser beams,
- a developing laboratory in Coating Protection Technology, with the latest techniques for plasma vapour deposition and chemical vapour depostion processes.

Programme Structure of the Institute

During the first and later years of its life, the work of the Institute has been planned to cover a range of research areas which respond to the perceived future needs of materials science and of customers demands, and in line with the competences which exist and which are being developed.
These research areas are:

- Functional Materials - synthesis of materials with specific functions, such as sensors,
- Computer Integrated Materials Engineering - in particular processing,
- Surface Modulation and Engineering of Materials - to improve performance of structural and functional materials,
- Materials and Structural Reliability - to predict the long term behaviour and remnant life of materials, structures and plant in operational conditions,

- The Behaviour of Alloys in High Temperature, Aggressive Operational Conditions - the extension to components,
- Advanced Ceramics for high temperature applications - performance in corrosive service environments,
- Data Banks and Materials Informational Services - extension to other materials themes and standards,
- Non-Destructive Evaluation and Testing - of structures, components and materials such as composites and ceramics.

Within the above broad materials research categories, projects have been launched, consistent with the Programme and budgetary sources indicated below.

The Specific Programme on Advanced Materials

Here, projects and research areas are supported by the Specific Programme on Advanced Materials of the Framework Programme of the C.E.C. The following conform to the pattern of Workschedules devised by the Joint Research Centre, which is the principal managerial means of planning and controlling the research of the Institute. These are:

- Properties, Performance, Characteristics and Improvements of Structural Materials

  Alloys

  To study the performance and deterioration of materials in simulated industrial environments with physically based modelling and experimental verification to predict behaviour in service. To develop, test and assess methodologies suitable for industrial needs.

  Engineering Ceramics

  The investigation of engineering ceramics behaviour in simulated industrial situations. The analysis and engineering of microstructural and interfacial factors influencing materials properties. The development of metrology for ceramic materials characterisation and testing.

  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: to support the design for the first wall of NET.

  Operational Defects in Materials and Lifetime Predictions

  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 on non-intrusive methods (coherent light and thermal emission) and acoustic emission for materials and components. Numerical simulation of creep and fatigue experiments.

  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 possibly other processes and by treatment with ion beams.
  Development of evaluation procedures for these coatings.

- Properties, Performance, Characteristics and Innovation of Functional Materials

  Characterisation of High Tc Superconductors

  Critical analysis and development of characterisation methods of high Tc superconductors.
  Development in JRC of a source of well characterised samples, to be made available externally.

  Low Activation Steels

  Development and characterisation of new stainless steels by replacement of nickel and chromium alloying elements by manganese and silicon to produce a) heat resisting steels at lower cost and b) lower activation alloys for H.T. nuclear applications.

  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.

Dependent on the results to improve the properties and the performance in some specific cases.

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. To form an industrial project club around associated labs (JRC, UKAEA, Univ. Tuebingen, Milan and Madrid).

- Modulation of Surface Properties
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.

- 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 an instrument 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 Institutes contributes additionally to the following specific programmes in the research areas of:

- 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 is put on the In-Service Inspection (ISI) of the primary circuit of nuclear reactors.

- Radioactive Waste Management
Safety of Final Storage in Geological Formations
Materials Research Aspects
To describe the interactions between conditioned waste (vitrified high level waste and alphacontaminated waste in concrete) and the surrounding materials in final storage conditions, essentially for the development of risk assessment models. Material research problems in support to other activities of the programme will also be studied.

- Fusion Technology and Safety
Materials Integrity
Provide experimental information on properties and radiation behaviour of AISI 316 steel for NET first wall and for low activation austenitic Mn-Cr steels. Study of effects of plasma disruptions - determination of thermal fatigue behaviour of first wall elements. Collect data on Pb-Li properties and its compatibility with structural materials.

Projects in Support of Commission Services

A number of project areas are directly sponsored by other Services of the Commission in different Directorates Generales. These refer closely to the specific interests of the Services. Currently, the following projects are being supported by other arms of the Commission Services:

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

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 projets for the industrial integration of materials information services,
3) Development of standards for materials databanks.

Exploratory Research

Under this heading, those projects are grouped which are obtained by a process of competition throughout the Joint Research Centre.

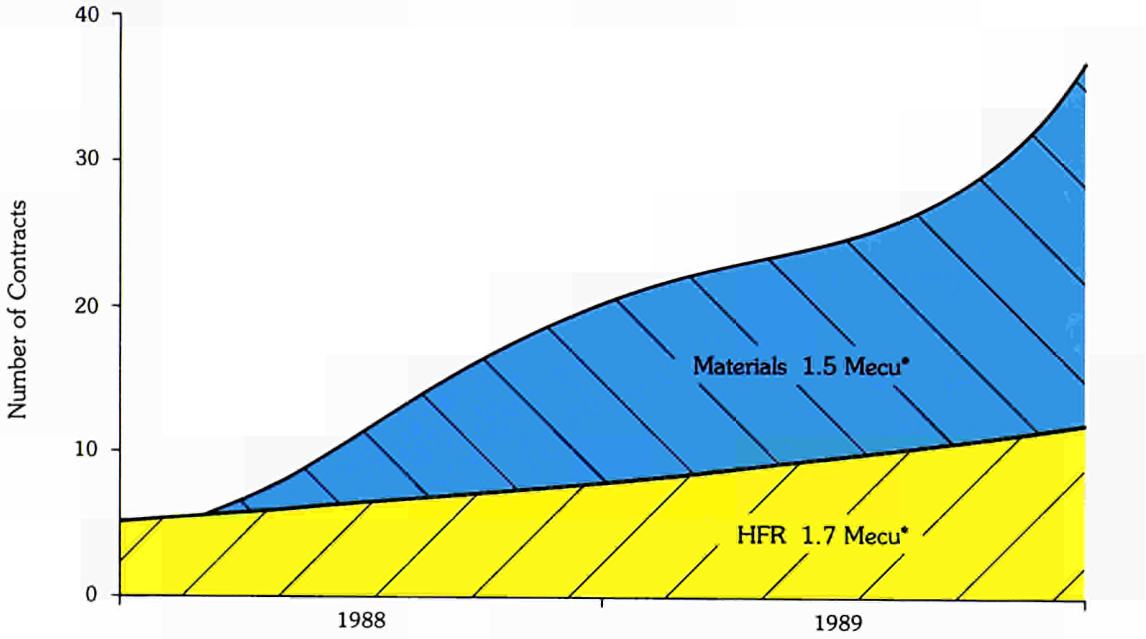
Here, 5% of the value of all the specific programmes have been collected into a general surcharge budget for supporting this type of research, which is aimed to stimulate originality and fresh directions in the scope of programmes of the Institute. During the first year, the Institute has been successful in gaining a number of such projects which are listed 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.

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 have been made available for research contracted by outside bodies. During the first year of the Institute, a number of contracts have been signed, which reflect the range of interest of the Institute, the projects generally deriving from the scientific experience gained in the execution of the Specific Programme. The evolution of this type of R & D is illustrated in figure below.

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



Budget and Resources 1989

The table below lists the budget appropriations and the manpower resources allocated to each of the research areas during the year 1989.

Programme	Research Staff	Research Budget (Kecu)
1. Specific Programme		
Materials	84	2280
Fusion Materials	21	870
Reactor Safety (PISC)	21	530
Other Specific	6	70
2. Support to the Commission	4	100
3. Exploratory	4	60
4. High Flux Reactor		
Complementary	42	850
Common	3	200
Totals	185	4960

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 1989 amounted to 1.700 Kecu.

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

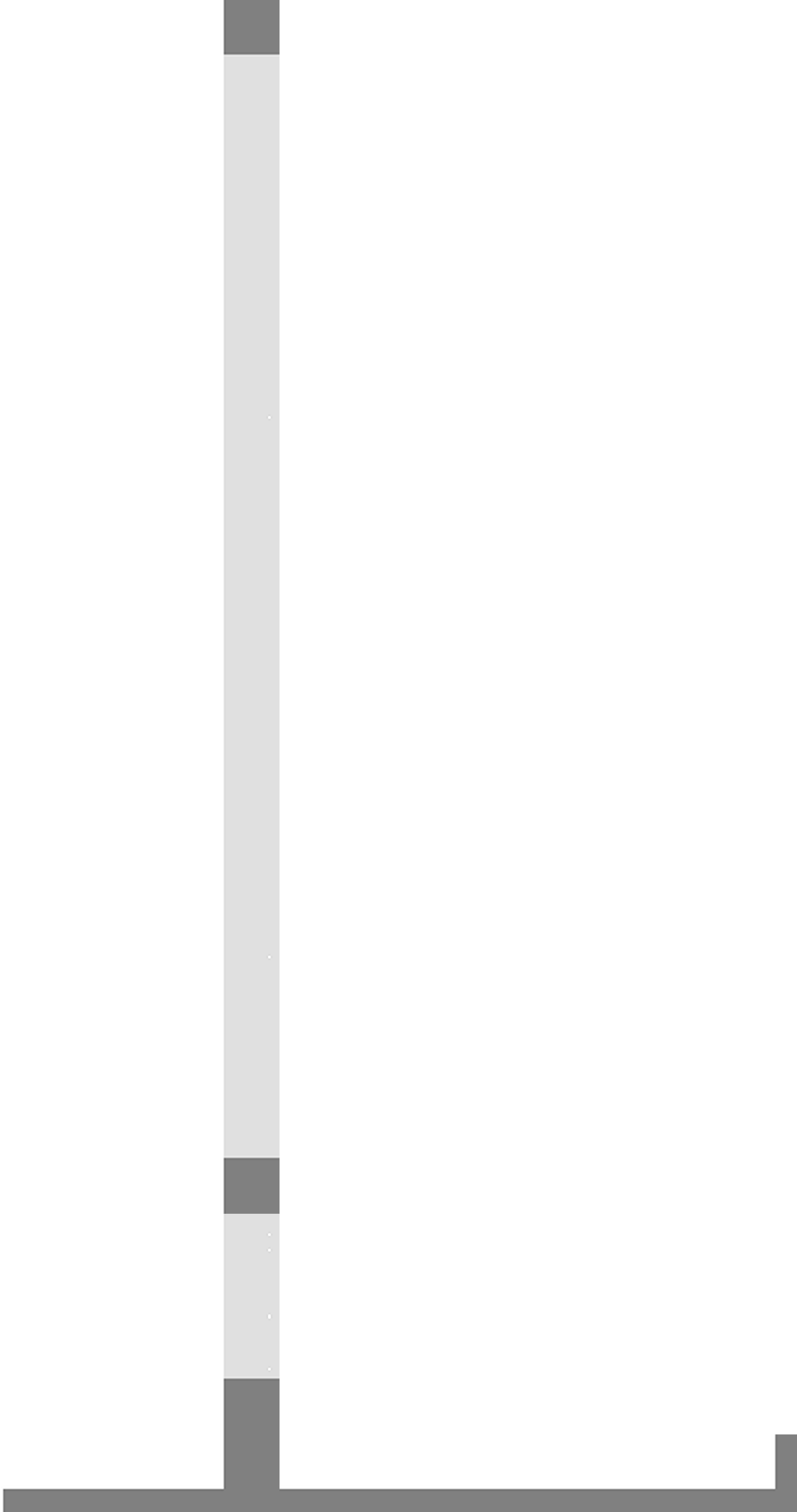
References:

1 JRC Panel of Senior Industrialists: "An Industrial View on the JRC", 6.11.1986
2 JRC Annual Report 1988, EUR 12305 EN

II Scientific - Technical Achievements

1. Specific Programme: Advanced Materials

Properties, Performance, Characteristics and Improvements of STRUCTURAL MATERIALS



Alloys

The aim of this multi-annual project has been 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 requirement is to develop test and assessment methodologies suitable for industrial needs.

Changes in the Institute strategy since the aims were originally proposed have meant that some critical experiments and evaluations required to enable the derivation of a physically based model accounting for corrosion, creep and fatigue cannot now be envisaged. Instead, assistance is being given to collaborative European efforts, which are aimed at physically based modelling or are concerned with pre-competitive research related to components which are critical for various high-temperature applications.

The importance of third party work to the Institute has also resulted in a shift of emphasis between the two areas of scientific work outlined in the objectives. The development of test and assessment methodologies was originally foreseen to be a secondary activity.
However, during 1989 this type of work has consumed a considerable proportion of the effort of the project.

Much of the scientific activity of the project has been carried out in cooperation with industrial and research institutes throughout Europe, particularly in the frame of COST 501 Round II, but including also the Non-Nuclear Energy Programme and BRITE-I.

As such the investigations are geared to respond to the specific objectives of these various integrated programmes. It is for this reason that some coherency between the various test programmes which contribute to the project is less than ideal. Again, for this reason it is necessary to describe the project activities in terms of the groups concerned.

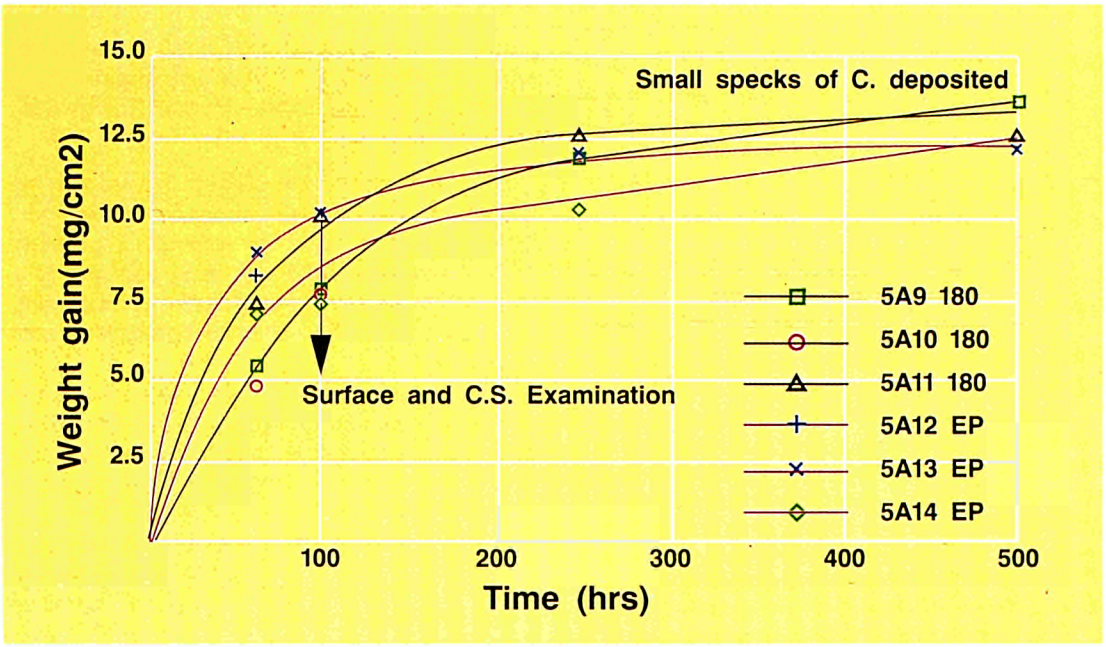
a. Corrosion

Much of this group's activity fell within the cooperative actions such as COST 501/Round II and the Community sponsored Non-Nuclear Energy R&D programme.

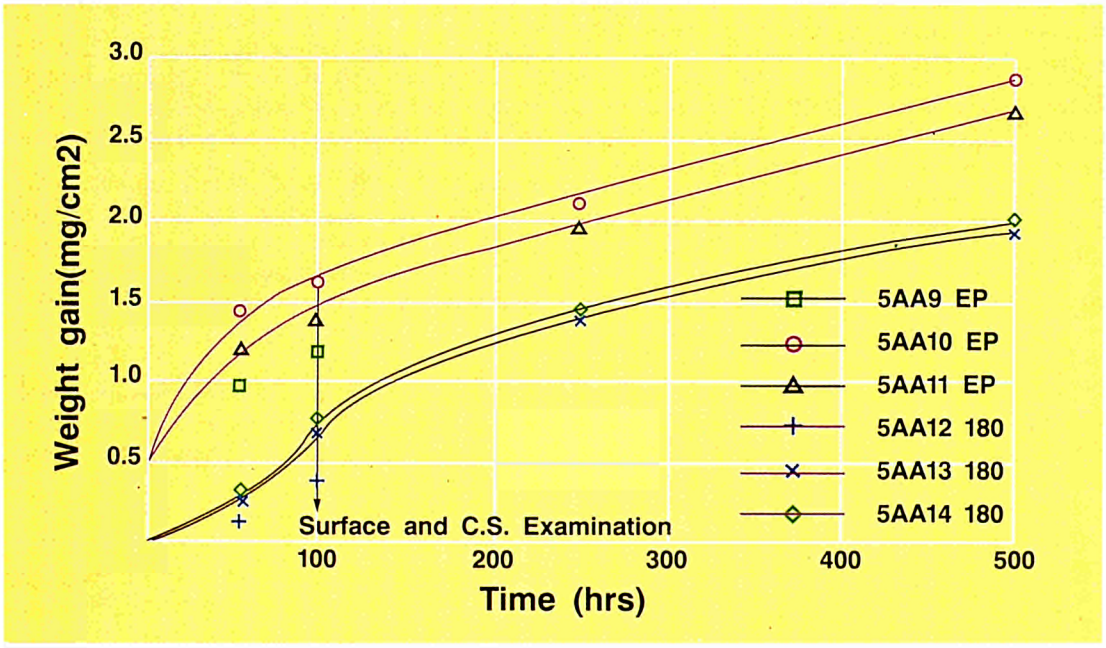
Experimental work in the COST 501/II programme was concerned with materials for heat exchangers (Work Package 4) and commenced in September 1988. Candidate alloys are being exposed to a range of mixed sulphidising/oxidising/carburising atmospheres of relevance to coal gasification processes.
One of the primary objectives is to correlate the corrosion behaviour of these materials after exposure in accurately defined and closely controlled laboratory conditions with that observed in plant situations.
The alloys chosen for this investigation were AISI 310, Alloy 800H, Fecralloy (a ferritic alloy containing 5% Al + Y for enhanced corrosion resistance) and an oxide-dispersion strengthened alloy, MA 956.

During 1989, laboratory testing of the 4 alloys started, after an initial delay in the procurement of the materials.
Tests are being carried out at 600^0C using a H_2 - 7% CO - 1.5% H_2O - 0.6% H_2S gas mixture which, assuming equilibration, has a
$pO_2 = 10^{-26}$ bar, $pS_2 = 10^{-10}$ bar and $a_c = 0.15$ to 0.2.

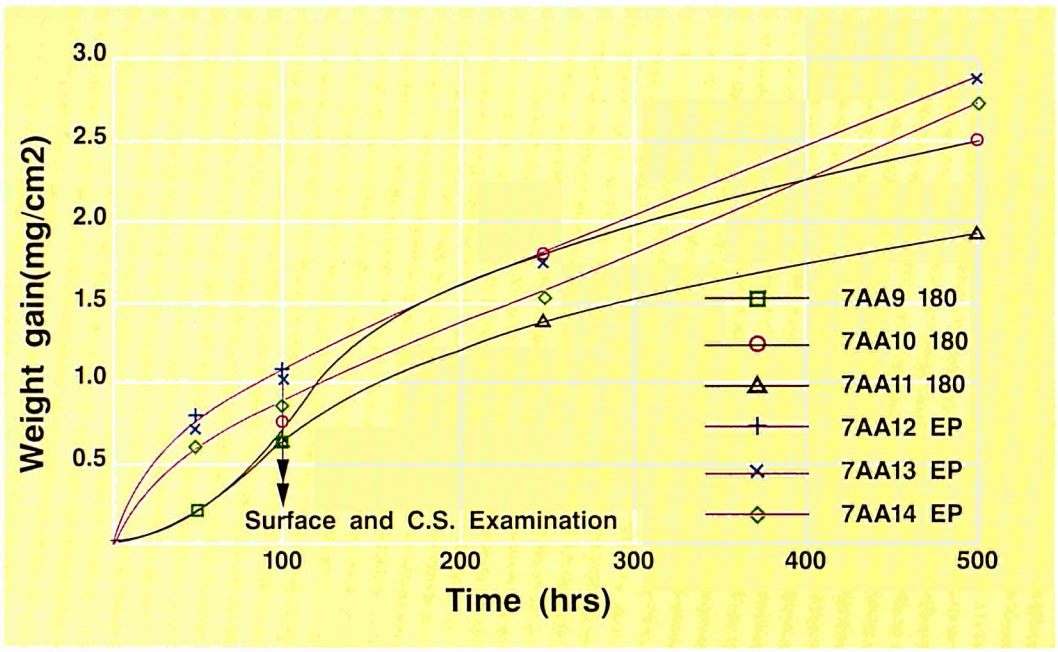
Exposures of 1000 hours have been completed with intermittent examinations at 50, 100, 250 and 500 hours. The kinetics of the corrosion reactions are illustrated in the figures page 14 and page 15, which show that the performances of the MA 956 and Fecralloy materials are much better than either the AISI 310 or Alloy 800H.
The ground specimens of both MA 956 and Fecralloy materials appear to be more corrosion resistant than companion specimens prepared to an electropolished finish.



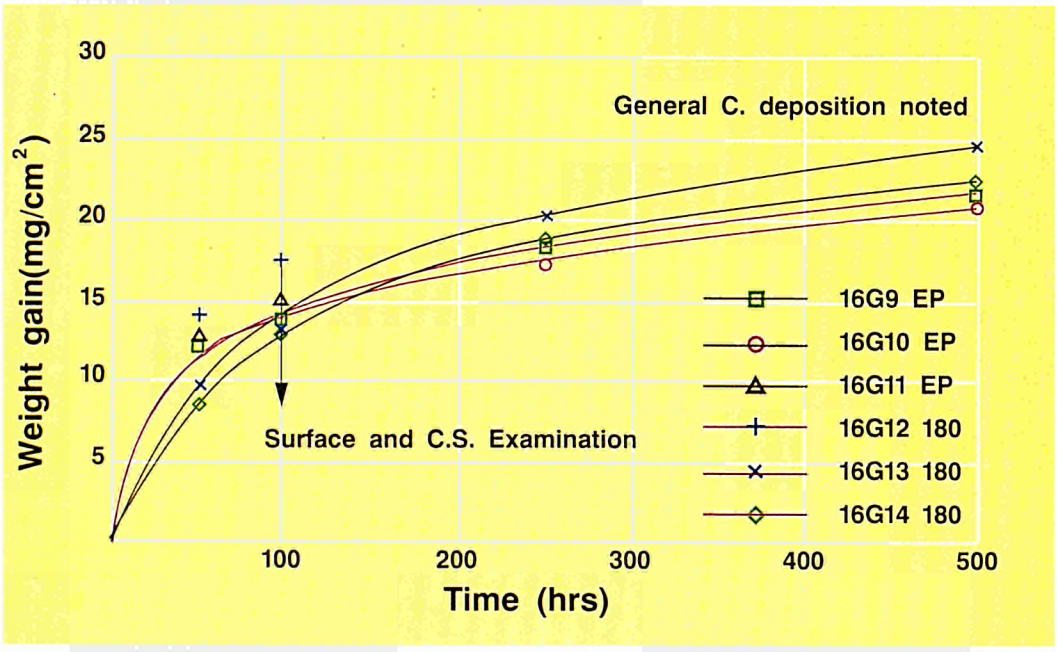
Corrosion kinetics at 600°C/0.6%H_2S - A.I.S.I. 310



Corrosion kinetics at 600°C/0.6%H_2S - M.A. 956



Corrosion kinetics at 600°C/0.6%H_2S - Fecralloy



Corrosion kinetics at 600°C/0.6%H_2S - Alloy 800H

As part of this COST 501/II cooperative action, a gasifier operated by British Coal is being utilised for in-plant exposures: Since it is obviously desirable first of all to characterise the atmosphere of the gasifier, a series of elements and compounds were exposed in the plant prior to the exposure of corrosion coupons made from the collaborative alloys. Samples of 10 elements and compounds, i.e. Co, Fe, Mo, Cr, Nb, Fe_2O_3, Cr_2O_3, $MnCr_2O_4$, MnO and NbC were exposed to the product gas for 550 hours in three different temperature regions (top - 700 to 840^{0}C, middle - 500 to 670^{0}C and bottom - 350 to 530^{0}C).

After exposure these samples were returned to J.R.C.-Petten for identification using an X-ray diffraction technique. These studies have enabled reasonable indications of the activity "windows" for the reactants S and O to be derived.
Figure below is an example of a phase stability diagram for several elements, i.e. Cr, Fe, Co, Ni, Nb and Al and the reactants S and O calculated from thermodynamic principles at 650^{0}C.

The "window" derived from analysis of the elements exposed in the middle region of the gasifier is included on this figure. Coupons from the collaborative alloys have subsequently been exposed in the three positions of the gasifier and the extent and nature of the corrosive attack experienced by these is currently being critically compared with that observed on similar specimens exposed in the laboratory autoclaves.

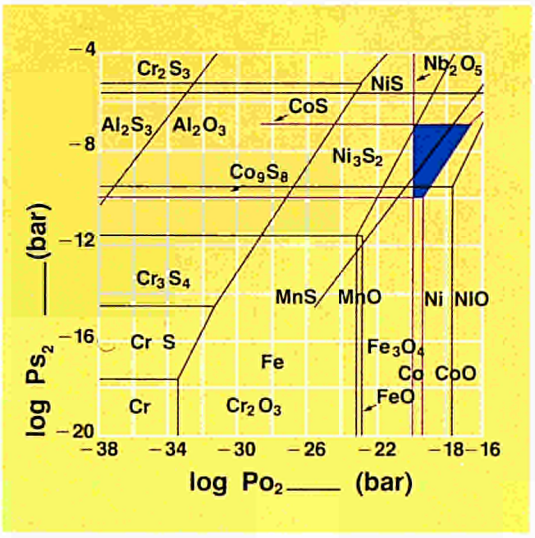
Experimental work in the Non-nuclear R&D Programme: Basic studies have been carried out on an agreed range of commercial alloys to determine the kinetics and mechanisms governing corrosive degradation. These investigations concentrated on the exposure of samples in gaseous atmospheres in the absence of impacting particles whereas parallel investigations being carried out by European collaborators have been designed to assess the interactive effects of erosion and corrosion in similar aggressive environments.

Using these laboratory studies an erosion/corrosion model has been developed and is being validated by comparing the predictions with actual performance of test coupons exposed in commercial gasification and FBC plant.

The programme started in December 1986 and was for an initial 3-year period.

During 1989 all the outstanding corrosion tests were completed which rounded off the series of matrix experiments being carried out in H_2 - CO - H_2O gas mixtures containing varying levels of sulphur, i.e. 0.2% and 0.6% H_2S with selective testing also being carried out in 2.0% H_2S, at 500^{0}C and 700^{0}C. The influence of pre-oxidation treatments upon corrosion resistance was also studied for all 4 materials covered by this programme, i.e. AISI 310, Alloy 800H, Fecralloy AB and a Ni-based gas turbine alloy IN 738 LC.

Below: *Thermodynamic phase stability diagram for selected M-S-O system at 650^{0}C*

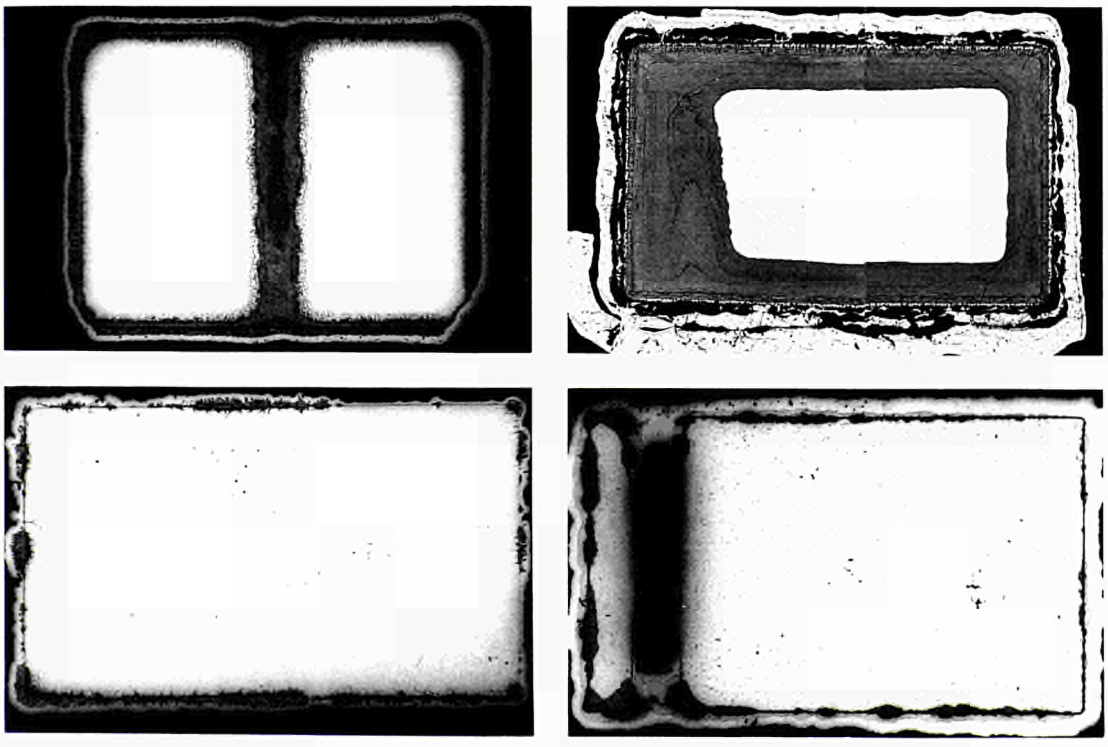


The last series of tests carried out in the 2.0% H_2S-containing atmosphere, have concentrated on establishing whether pre-oxidation treatments given to the Fecralloy AB and IN 738 specimens continued to confer the extremely good protection observed in the 0.2% and 0.6% H_2S-containing gas mixtures. Surface and cross-sectional examinations of specimens have shown that after 1000 hours exposure in the highest H_2S atmosphere, sulphide scales have started to form.

This casts doubt on the long-term benefits of such treatments, at least for use in such an aggressive atmosphere. Of the alloys tested, the Fecralloy AB alloy in the ground and pre-oxidised condition was significantly more corrosion resistant than any of the other alloys tested. Figure below compares the cross-sectional macrostructures of the best two alloys, Fecralloy AB and IN 738 LC, in the pre-oxidised and non-pre-oxidised conditions after being exposed to the 2.0% H_2S-containing atmosphere at 700°C for 1000 hours.

Experimental work involving Corrosion under Deposits: A method has been developed in which powders are pressed onto a specimen inside a closed jig, enabling tests to be conducted without the need for a controlled atmosphere.
The deposits may be synthetic or ground flue ash and use of this technique has enabled the features of corrosion observed under deposits in FBC's to be reproduced more faithfully than by conventional crucible tests. It appears that of the compounds present in FBC deposits only $CaSO_4$ is active in causing sulphidation.

Below: *Cross-sectional macrostructures of Fecralloy AB and IN 738 LC*



b. Creep

This group's activity during 1989 has been concerned with rig development, the completion of long-term testing and conduct of additional investigations arising from COST 501, Round I.
Following the delivery of the collaborative material testing could start in the integrated programme for COST 501, Round II.

Environment - Mechanical Property Interaction in Heat Exchanger Alloys:
Investigations into the influence of S-O-C bearing gases on creep-corrosion and corrosion-creep interaction in the heat resistant steels Alloy 800H and the 32Ni-27Cr-0,07 Ce alloy AC66 (developed by Mannesmann Steel Co.) have been terminated.

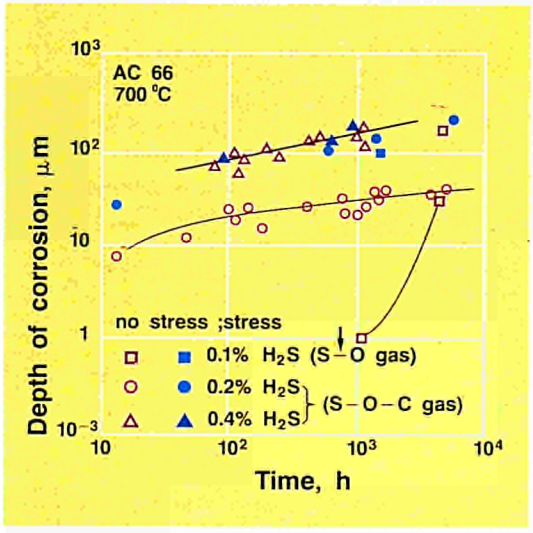
Concerning the corrosion behaviour, it was found that AC66 behaves better only in the pre-oxidised state. However, in the stressed condition, this benefit is eliminated for relatively high deformation. Moreover, for the blank and the pre-oxidised state of both materials it was found, that a superimposed deformation leads to stronger corrosion than is observed for the stress free state, figures below.

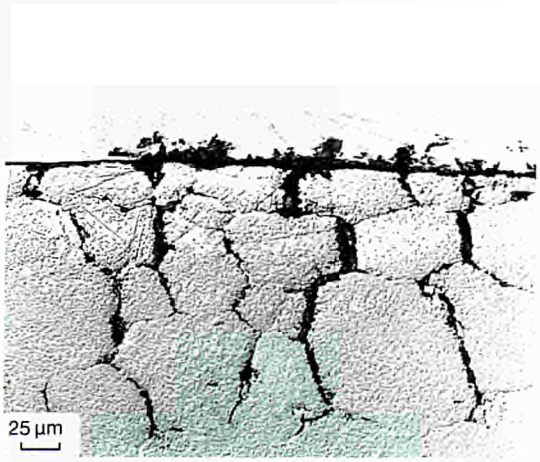
The corrosion pattern was characterised by preferential attack along the transverse grain boundaries, foto below. Regarding the influence of corrosion on creep, for both alloys the creep strength properties were degraded only if a high pS_2/pO_2 level of the environment led to a severe uniform corrosion with the consequence of a reduction of load bearing cross section. On the other hand the creep ductility was reduced considerably for both alloys when the exposure took place in sulphidising environments independently of the pS_2/pO_2 level. This was explained by a reduced cohesion at the matrix-corrosion product interface.

Alloy 800H had been proposed for use in coal gasification and FBC applications and AC66 was developed specifically for such conditions. From the results of the investigation which ran for three years, it can be concluded, that Alloy 800H, in particular, seems to be suitable only if the sulphur activity can be kept to very low levels.
The first experiments, using the COST 501-II collaborative material (ODS alloy MA 956) have shown a much higher corrosion resistance than AC 66 and Alloy 800H as might be expected from the possibility of Al_2O_3 scale formation.

Left: *Depth of internal corrosion versus exposure time for stress free and stressed AC 66 at 700°C in various S-O-C bearing gases*

Right: *Corrosion pattern of AC 66 at 700°C after deformation in 0.2 v/o H_2S gas, t_f = 4983 h*





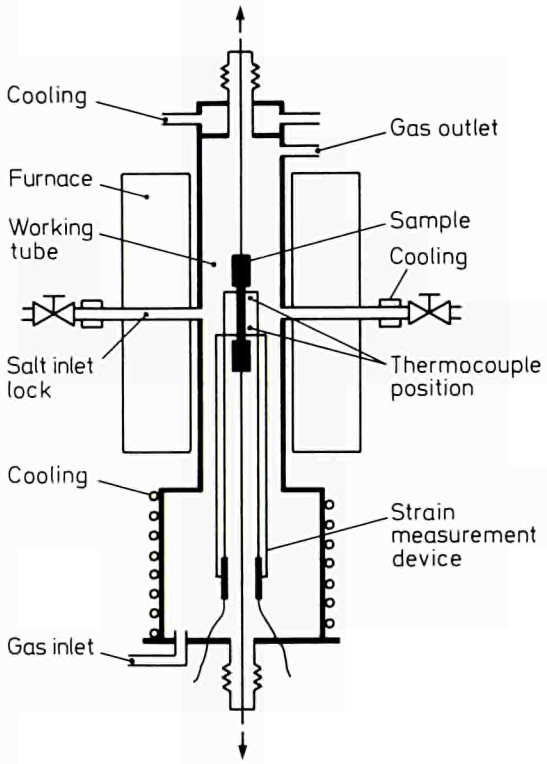
Developmental Gas Turbine Vane Alloys:

In the frame of the COST 501/II programme, creep investigations have been started on the oxide dispersion strengthened alloy MA 760 at 1050°C in air. This work is carried out in collaboration with external laboratories with the aim of obtaining a comprehensive characterization of the material and its application potential for land based gas turbines. The group is contributing in particular with studies on the relationship between creep and microstructure. The stringent material specification, prepared by our industrial partners resulted in manufacturing difficulties and a consequent delay in the delivery of the material. Nevertheless, a few long term tests have been started. The first structural examinations have shown that the formation of surface porosity, will pose a significant structural problem, comparable to that studied on alloy MA 6000, in a COST 501-I test programme which was completed in 1989.

The foto below shows the stress free coupon exposed along with the creep test with the highest temperature/longest duration in the series. Prelimilarly results on creep strength of MA 760 have indicated a behaviour which is similar to that observed for MA 6000.

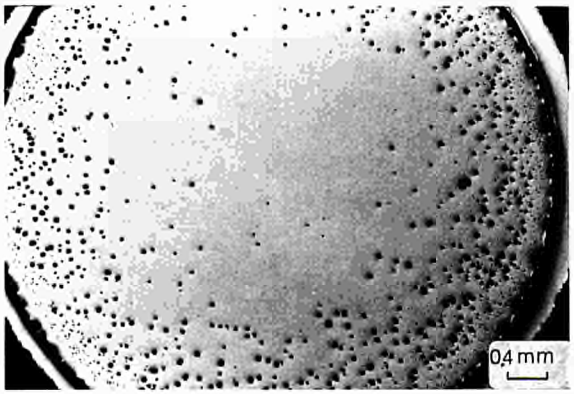
Specific experimental requirements concerning corrosion and creep studies on IN 625 and Hastelloy X in an air/SO_2 mixture with simultaneous addition of seasalt arose in the context of a contract research project. A complicated modification of existing creep facilities became necessary. To enable the additions to be made in a controlled manner, without interruption to the test.

Figure above shows a schematic view of a modified creep rig, which allows a sea salt solution to be injected directly onto the specimen at regular intervals by means of two horizontally mounted tubes.



Above: *Equipment for creep testing in controlled atmospheres with independent salt addition*

Below: *MA 6000 showing severe porosity after 11035h annealing at 1050°C*



c. Component Behaviour

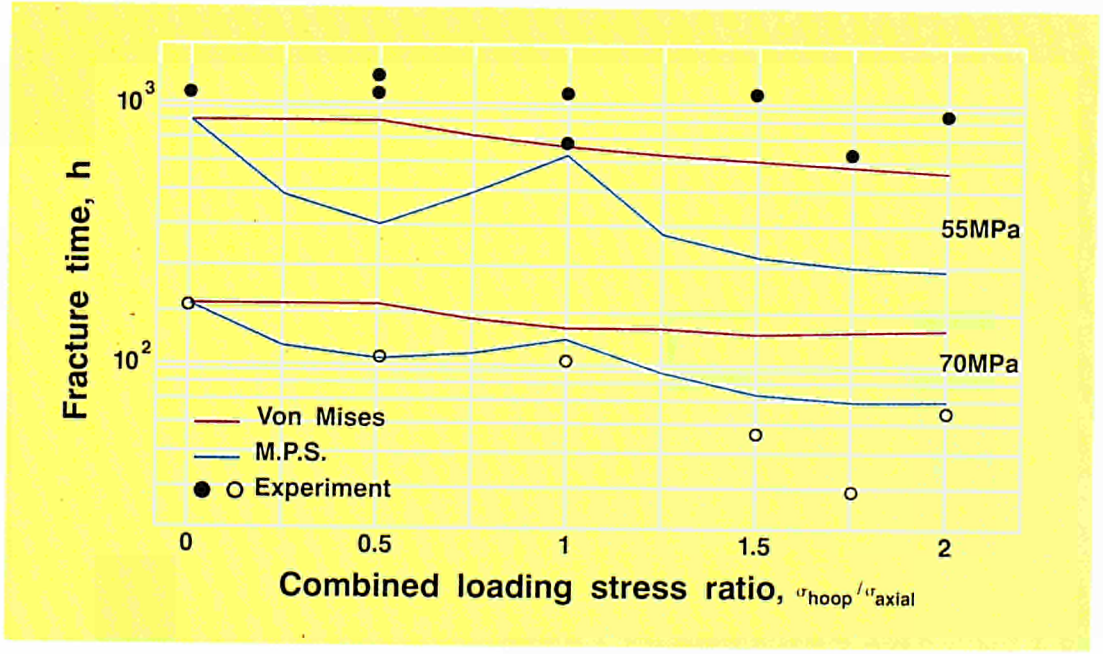
Cooperation within the frame of a BRITE-I contract and in COST 501, Round II required most of this group's experimental effort during 1989.

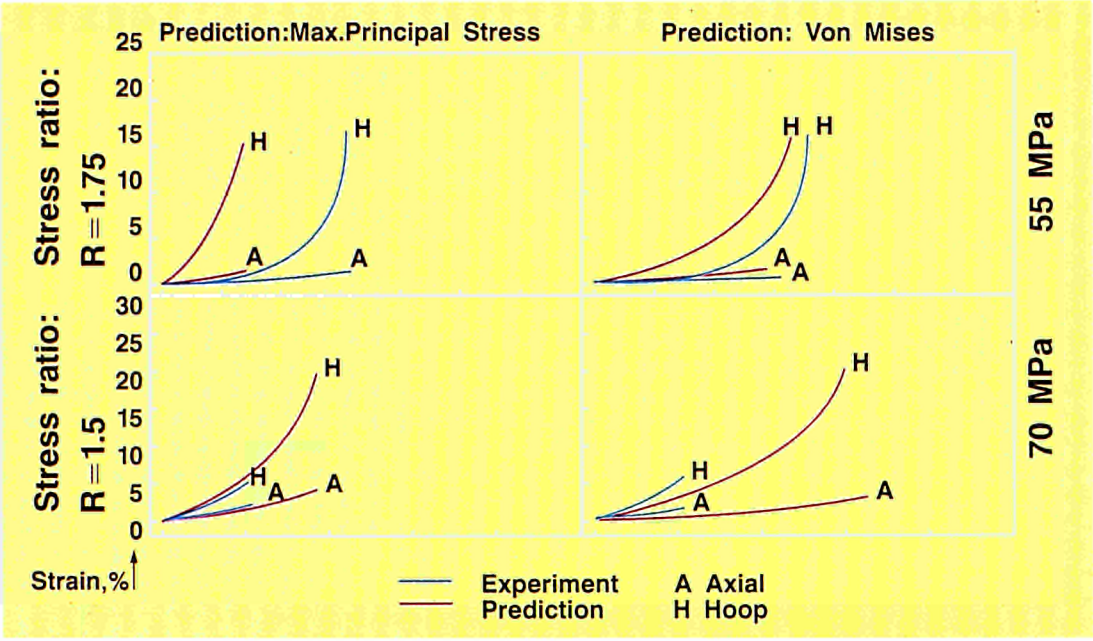
Multiaxial creep behaviour and modelling of Alloy 800H at 800°C.

This activity, which terminated at the end of the year, contained work originally commenced under the auspices of COST 501 Round I.
Earlier experiments, investigating the uniaxial and internal pressure testing of tubular components under a wide range of creep stresses, highlighted an apparent change in creep mechanism below a certain stress which caused difficulties when predicting long term behaviour from relatively short term test data.
In the final year, the research has concentrated on testing at two stress levels (ostensibly above and below the transition stress) over a range of multiaxial stresses ranging from tension (σ_H/σ_A = 0) through various biaxial ratios to internal pressure with σ_H/σ_A = 2. In order that the experiments could contribute as verification tests to creep deformation prediction models, all test pieces were fully instrumented for both hoop (diametral) and axial strain measurements apart from the pure internal pressure tests where no axial strain is manifested.

The baseline uniaxial creep data used in the analysis method comprises constant stress and load tests carried out by partners within the COST programme. Although a number of creep models were applied to the data, the most successful was found to be a continuum damage mechanics model based on the Rabotnov-Kachanov damage equations. These types of models are loosely based on physical mechanisms and, of the many types investigated, the model based on linear strain softening was found to be most suitable. The modelling procedure analyses fitted creep curves which can then regenerate predicted uniaxial or multiaxial curves at the appropriate interpolated or even extrapolated test stresses. Examples of the multiaxial predictions compared with experimental results shown in figure below support a trend for von Mises control over deformation at 55MPa, whilst at the higher stress of 70MPa, Maximum Principal Stress is clearly the governing criterion. The predictions, although confirming the different deformation criteria, nevertheless are much less exact than their uniaxial specimen counterparts, largely due to geometrical considerations.

Below: *Comparison of modell predictions with experimental data for multiaxial creep of Alloy 800H tubes at 800°C*





Above: *Comparison of model predictions with experimental data for creep rupture of Alloy 800H tubes at 800°C*

The stress rupture behaviour under multiaxial stresses better reflects the predictive ability of the model.
Figures above show the predicted stress rupture Locii dependence on stress ratio compared with the actual experimental results. A clear support for von Mises control at 55MPa and MPS control at 70MPa is portrayed.

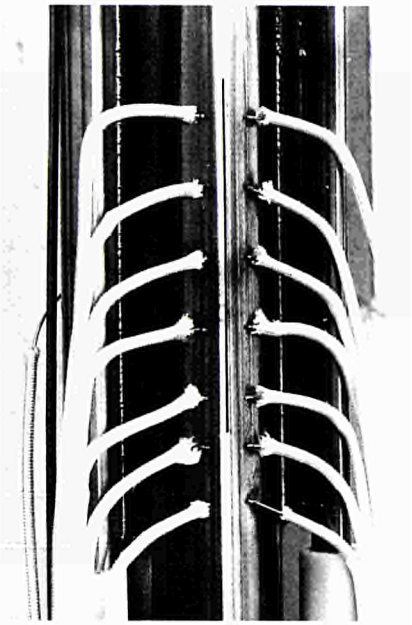
Uniaxial and multiaxial creep of Alloy 800H at 850°C and 2¼Cr1Mo at 550°C.
Within the framework of a BRITE programme aimed at life time prediction for heat exchanger materials, the JRC has contributed experimental results. The work sharing envisaged within the project and the confidentiality imposed precludes a coherent presentation of the results. However, the scope of the work in 1989 included a number of constant load and constant stress creep tests on both alloys.

To this data and that obtained by the partners, creep modelling such as θ-projection and Continuum Damage Mechanics have been applied in order to develop a methodology for component life time prediction. As a first test of the models, tubular tests using internal pressure have been carried out on both alloys as simplified benchmark tests.

This modelling of component creep behaviour, will be linked to parallel creep-fatigue and LCF models being applied by the partners and the results fed to the JRC Databank in such a way that they can be extracted and evaluated for component design using finite element methods.

Internal pressure testing of 2¼Cr1Mo ferritic steel. In the frame of COST 501-II, WP5 the component testing activities planned for the first year of the project were confined to exploratory internal pressure tests on tubes machined from the wall of a 2¼Cr1Mo steam pipe header. The tubes have been machined to the design recommended in a Code of Practice for Tube Testing, prepared independently by a working group chaired by a JRC-staff member. A total of four tests have been completed at 550°C and a single test at 600°C over a pressure range of 195-295 bar. As sufficient uniaxial creep data is not yet available for the cast concerned, it was not possible to apply creep models for the correlation of multiaxial with uniaxial data.

An important aspect concerning these tests relates to the failure mode. Whereas all tests exhibited creep ductilities greater than 5% three of the five tubes tested ended in catastrophic failure (Figure below). Very rapid extension of an axial crack must have occurred to allow complete opening of the tube and consequent damage to the rig. Metallographic examination has revealed large quantities of impurities in the form of stringers in this batch of material and it is concluded that the crack could easily run along such stringers in the thin wall tubes leading to the "brittle" failure observed. Until uniaxial data is provided, the influence of the embrittling phase on rupture life cannot be determined.

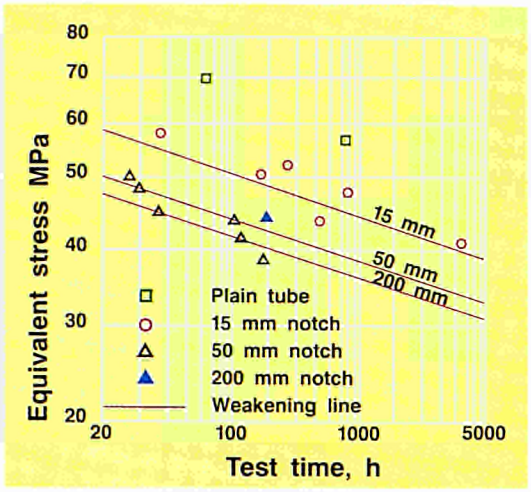


Above: *PD instrumented longitudinal notch (2¼Cr1Mo).*

Below: *Post test view of test cell*

Component crack growth testing. A novel approach has been made towards the determination of creep crack growth (C.C.G.) in tubular components. This technique is intended to bridge the gap between conventional C.C.G. specimen tests and full scale plant component performance. The ability to conduct controlled C.C.G. experiments on instrumented components under multiaxial stress condition is particularly important for structurally or geometrically sensitive materials. The growth of surface defects which may be either a spark eroded longitudinal notch (figure above) or a machined circumferential groove is monitored under multiaxial stress by a potential drop technique.

The exploitation of this methodology has been focussed on the Alloy 800H material although some preliminary experiments on 2¼Cr1Mo alloy were commenced at the end of the reporting period. For both alloys, longitudinal, spark eroded, part through wall notches have been studied under conditions of internal pressure only. In the case of Alloy 800H, cirumferential grooves could be tested using either axial load or internal pressure or a combination of both.



In order to understand the influence of (longitudinal) notch length and depth on creep lifetime, the basic internal pressure rupture data for the Alloy 800H has been transformed into predicted "weakening" lines using calculation methods available in the literature. Figure above shows, as an example, the influence of notch length for 1 mm deep notches in a 2.5 mm wall, and in particular, the marginal conservatism of the calculation methods used compared with some thirteen test results. This conservatism may also be considered as notch strengthening which is often reported for 2¼Cr1Mo material and is indeed supported from the preliminary testing of this alloy. Although not displayed here, similar rupture strength predictions have been made when comparing the circumferentially notched tubes tested in tension with tension tube data.

The use of the generated C.C.G. curves for comparison with data obtained from conventional compact tension tests has confirmed the potential of the methodology for both the longitudinal and cirumferential notches. Although good inter-correlation between individual component C.C.G. curves can be obtained using the stress intensity factor K_I in analysing the crack growth rate, the equivalence with conventionally obtained K_I data is poor. On the other hand C* which, in the absence of load line displacement measurements, has been calculated using a reference stress approach, correlates will with the conventional data.

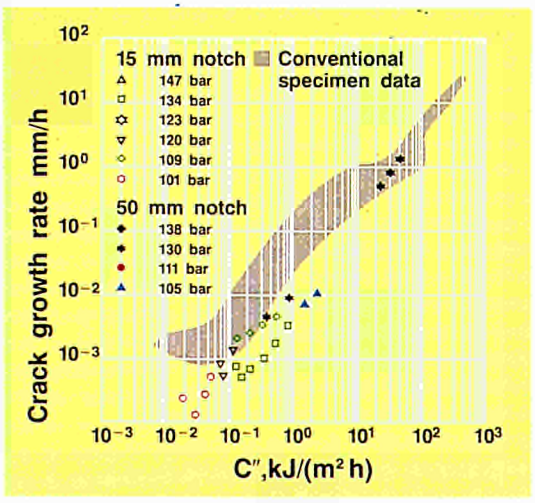
Figure below shows such a correlation for the cirfumferentially grooved tubes tested in tension, the conventional specimen data being obtained from the same cast of Alloy 800H studied in an EGF round robin testing programme.

An autoclave has been constructed to enable uniaxial creep tests to be conducted on solid samples in a high system pressure of an aggressive test gas. This rig which was developed for use in one third party contract and has since proved to be of interest for others, relies on capacitance transducers for continuous strain measurement on up to three specimens in a string. The same rig can be used to test tubular components with the external pressure greater than amospheric.

To enable experimental work to be conducted for the "Components and Thermal Fatigue" project a rig was designed for the thermal loading of tubular components. The tubular test-piece is heated externally by induction coils and internally cooled, in the first instance, by fast flowing water.
Suitable instrumentation is selected for the particular experimental objective.

Above: *Stress rupture behaviour for 1 mm deep longitudinal notches (Alloy 800H)*

Below: *CCG rate dependence on C* for longitudinally notched Alloy 800H*



d. Fatigue

The activities of this group encompass experimental and management work in the COST 501/2 programme, the development of a computer vision technique, and the building of the expert system ARTIC.

Experimental work in the COST 501/2 programme is geared towards generating a data base on the oxide dispersion strengthened alloy MA 760, in collaboration with outside laboratories participating in the Work Packages WP1 and WP5. The group will contribute with isothermal low cycle fatigue and with thermomechanical fatigue testing. The testing conditions and integrated testing matrices have been defined and the testing equipment is ready.
Because of the delays with the delivery of the MA 760 material referred to above, the actual testing will start in 1990.

Development of a state-of-the-art computer vision system started in 1988 in association with an outside company and continued in the reporting period. The system is intended for the in-situ monitoring of surface damage on specimens subjected to mechanical deformation. The surface of the sample is viewed with a long distance optical system. In a software controlled snapshot procedure images are grabbed, digitized and stored on hard disk at preselectable magnifications and time or cycle intervals. Magnifications of up to 500 x are possible, corresponding with a field of view of 0,2 x 0,2 mm^2. Image contrast and quality can be enhanced through filtering (Laplace, Sobel, Roberts and low pass filters).

The computer vision system also provides for post-test quantitative image analysis, allowing the in-situ monitoring of the initiation and of the growth of microcracks during, for example, fatigue testing. The current prototype is for single field-of-view monitoring imposing a compromise between resolution (magnification) and a statistically relevant field-of-view.

In order to alleviate this controversy a scanning facility software is being implemented which will allow the imaging of surface areas up to 16 x the single field-of-view area with the same resolution.

The building of a prototype of the Expert System ARTIC for the assessment and management of the residual lifetime of steam headers continued in 1989, in collaboration with ISE (JRC Ispra). The ARTIC project is executed in the framework of an interest club of electric utilities (ENEL, CEGB, Laborelec, MPA) and of the JRC. In the reporting period the elicitation of the domain experts continued with a series of interview sessions with experts of the electric utilities. The architecture of the expert system has been defined, the domain knowledge structured in rules and facts and a suitable control strategy was selected for implementation in the expert system.

The prototype of the ARTIC expert system was tested in November 1989. This prototype testing phase is planned to permit the developers and the potential users to verify the suitability of the approach as well as to define the final scope of the working version of the system. The testing phase also aims at the identification of those parts of ARTIC which require redesigning or expanding.

Engineering Ceramics

During the last decade, important progress has been made to improve ceramics processing technology and attempts are being made to design with new ceramics for thermo-mechanical applications.
In the framework, the project studies the corrosion and mechanical properties of advanced engineering ceramics at high temperatures (up to 1.400^{0}C). Special attention is devoted to the understanding of the degradation mechanisms by in depth microstructural investigations, for failure analysis and component life-time prediction purpose.
The engineering properties referring to ceramic/ceramic joining, ceramics machining and non-destructive evaluation form an integral part of this projects.

Highlights of the year have been:

- The development of test methodologies, a subject which is poorly developed in ceramics characterization and receives priority in our research strategy.
- Modern laboratories with unique test facilities have been installed and advanced measuring and test devices developed including in-situ equipment for crack nucleation and growth measurements.
- Methodologies for testing the gaseous and hot corrosion behaviour of ceramics have been established as well as method for static and dynamic fatigue testing especially in tensile mode at high temperatures.
- The microstructural analysis laboratories have been equipped with the most modern analytical facilities.
- A facility for the measurement of residual stresses in ceramics by on X-ray diffraction technique has been made operational. It is employed to determine stresses in machined ceramic surfaces and interphase stresses in ceramic matrix composites.

Efforts on ceramics processing R&D is directed to the development of high quality ceramic composites.
Here, a methodology has been established to fabricate dense SiC (silicon carbide) whisker reinforced Si_3N_4 (silicon nitride) based ceramic composites, by slip casting and low pressure sintering.

The research on mechanical properties focusses on the cyclic fatigue behaviour of Si_3N_4 and the subcritical crack growth of whisker reinforced ceramics. The prime objective during the reporting period being to generate cyclic fatigue life curves and to elucidate toughening mechanisms of the reinforcement respectively.

The gaseous and hot corrosion behaviour of a range of modern engineering ceramics is studied. The aim being to elucidate the fundamental mechanisms of high temperature oxidation and sulphidation in oxidizing and reducing environments on silicon nitride alloys.

A viable and efficient method of high precision machining of engineering ceramics has been developed. This technique is very cost effective and minimized damage in component surfaces which can act as sites for preferred crack propagation. The inter-relation between mechanical properties and machining is being studied.

The influence of metallic and non-metallic interlayers and of energetic beam treatments on the joining of silicon nitride to silicon nitride by diffusion bonding is studied. First results demonstrate the great potential of solid state bonding for meeting the industrial service requirements for ceramic joints operating in high temperature structural application. Results of mechanical behaviour are reported together with structural and chemical analysis correlation.

All activities are well integrated into European collaboration schemes either in the framework of CEC-programmes e.g. Science, or in direct collaboration with Universities/Research Centres and Industries. The available unique test facilities and the expertise formed the nucleus of:
- industrial contract research;
- support to the Commission Services;
- exploratory research activities;

during the reporting period; these new orientations have seen rapid and successful development.

The research and development work has spin-off's in multiple 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 Composite

Introduction

Ceramic whiskers are added to monolithic ceramics, primarily to improve resistance to cracking in the matrix and thereby to enhance toughness and reliability. The interaction between whiskers and matrix and therefore the effectiveness of the re-enforcement is dependent upon the matrix-whisker bond strength and the associated stress field in the surrounding matrix.
The project investigates the factors influencing the interphase stress state with the objective to engineer the whisker-matrix interface for optimal composite properties.
The first stage of this project has been to develop a methodology for fabricating the composite. Owing to the high temperature stability of the high purity, monocrystalline whiskers, these composites can advantageously be prepared by conventional processing methods.

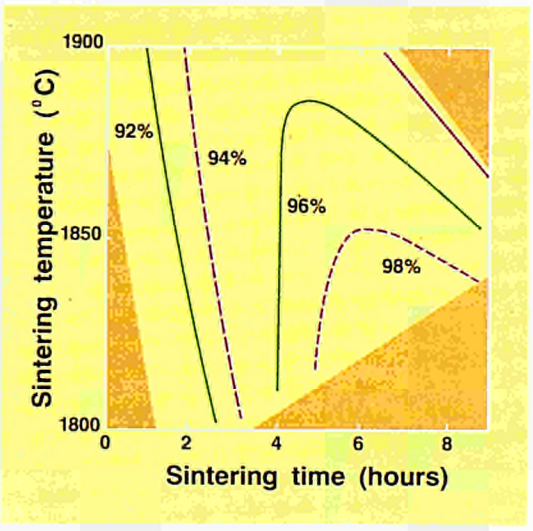
Densification is difficult. The open literature reveals that almost all studies to date have relied upon high cost hot pressing to achieve a dense ceramic. However earlier work (C. Olagnon, D. McGarry and E. Nagy) has shown that careful control of particle size and dispersion characteristics allows fabrication of monolithic Si_3N_4 by slip casting and low cost sintering. The present study extends this work to the Si_3N_4-SiC (w) composite with whisker content of 5, 10 and 20% respectively.

Results

Dispersion studies revealed that fully deflocculated slips of optimum rheological characteristics could be achieved with 65wt% solids content and whisker content in the range 0 to 20 wt % over the pH range 7.6 to 9 using proprietary dispersants. Segregation of heavier particles and whiskers during slip casting presents major problems in subsequent densification. Refinement of particle size range by sedimentation fractionating significantly improved slip stability. Green density (ca 61%) increases with whisker content in accordance with close packing theory confirming an absence of intrinsic agglomeration defects. Densification behaviour with variation of sinter time and temperature (figure below) showed that under optimum conditions of 1850^{0}C/6h, a 10% whisker composite can be sintered to 98% theoretical density, a value which compares well with hot pressed material.

Structural analysis revealed that whiskers are anisotropically oriented in the plane orthogonal to the slip casting direction. Early measurements show a modest increase in toughness to between 6.5 and 7 MPa.m$^{1/2}$, which are significantly higher in directions parallel with the whisker plane than perpendicular.

Below: *Plots of bulk density as a function of sintering time and sintering temperature of a 10vol% SiC whisker Si_3N_4 composite sintered under 1MPa N_2 pressure. (Shaded areas correspond to the zones where the model does not fit)*



Residual Stresses in Ceramic Surfaces

Introduction

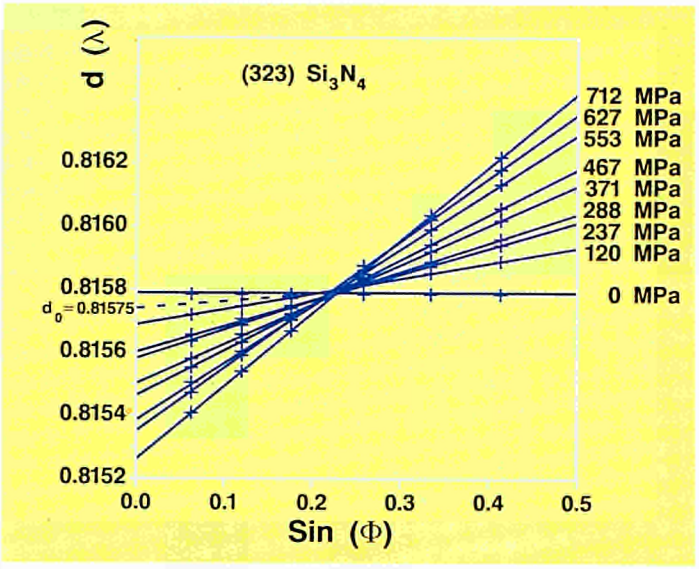
Residual stresses are produced during the manufacturing processes of a material. The entrapment of non-equilibrium structures within the crystal lattice gives rise to internal stresses which satisfy internally, force and moment equilibria.

These stresses influence material properties in the same way as an external stress. For ceramic materials which are highly sensitive to material conditions a full description of internal stress state will become an essential feature of material/component standardization.

The present activity has been initiated to select and create an operational facility for determining residual stresses in engineering ceramics based upon X-ray measurement of crystal lattice deformation using the well-known $\sin^2 \phi$ technique. The first phase has included, a theoretical evaluation of the methodology for residual stress measurement by diffraction techniques, an exhaustive literature review to establish the state of the art and future orientations, the acquisition and adaptation of a diffractometer and development of a working methodology. The methodology has been evaluated in a research project to determine residual stress in machined ceramic surfaces, and has been extended to measure interphase stresses in ceramic composites.

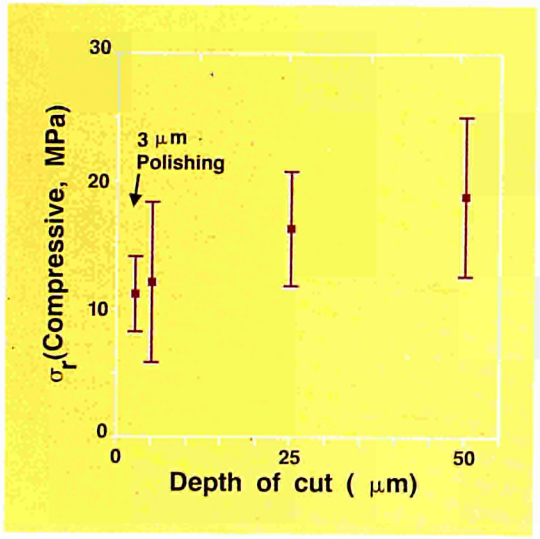
Results

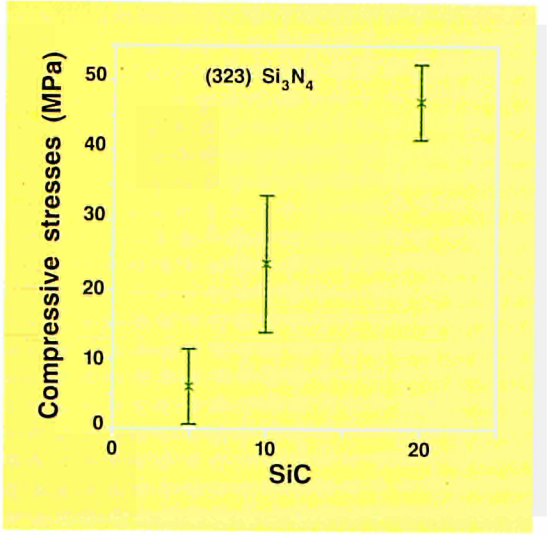
The stress-lattice strain relationships for the selected characteristic lattice plans were calibrated by determining the variation of d-spacing with mechanical stress applied in a straining stage mounted inside the diffractometer. Figure above, shows the Lattice spacing/$\sin^2 \phi$ relationship (ϕ = tilt angle) for the (323) diffraction peak of silicon nitride under several applied stresses. The elastic constants for the diffraction planes; $\frac{1+\nu}{E}$ and $\frac{\nu}{E}$ are obtained from the slopes and cutting points ($d\phi = 0$) respectively (ν = Poission Ratio). From this calibration actual residual stresses were determined from the appropriate $\Delta d = \sin^2 \phi$ plots, using CuKα radiation on surfaces machined under a variety of conditions.



Above: *Lattice Spacing vs. $\sin^2 \phi$ as a function of applied stress*

Early results show that residual stresses at the mean CuKα penetration depth (ca 60 µm) are low and generally compressive, but higher in orientations perpendicular rather than parallels to the grinding direction. Scatter in measured values are of the same order as the difference in value, as shown in a comparison of machining downfeed conditions figure on page 28 left. Future work will use Cr Kα radiation with a much lower penetration depth to measure stresses nearer the surface.





The determination of interphase stresses in ceramic composites in contrast show significant differences of residual stress values. For a silicon nitride-silicon carbide (whisker) composite, stresses generated by thermal expansion mismatch are compressive in the matrix and tensile in the whiskers. Variation of matrix stress with whisker content shows a significant increase (figure right).

Conclusion

An operational methodology for measuring residual stresses in engineering ceramics has been established and has been evaluated for machining induced surface stresses and interphase stresses in composites.

Left: *Variation of residual stress in creep-feed ground surfaces with depth of cut. The residual stresses of a sample polished with 3 μm diamond paste are also shown*

Right: *Variation of compressive stress in Si_3N_4 with increase in SiC content*

b. Corrosion Properties

The aims of this sub-activity are twofold:

1) To compare the high temperature corrosion behaviour of a range of modern engineering ceramics in simple environments simulating those of industrial importance for the application of these ceramics.
2) To seek an understanding and explanation of the corrosion mechanisms underlying their behaviour in terms of thermodynamic and kinetic criteria.

As in previous years, project activities have concentrated on silicon nitrides, mainly of commercial origin, fabricated by a range of techniques in which metal oxides such as yttria, magnesia and alumina are used to assist densification.
As an agent of high temperature corrosion, sulphur is increasingly recognised to be of major importance and consequently work has been focussed on bioxidant environments containing sulphur as well as oxygen, at both high and low activity.

The main achievements in 1989 are summarised below, encompassing three simple environments of importance towards an understanding of the corrosion mechanisms:

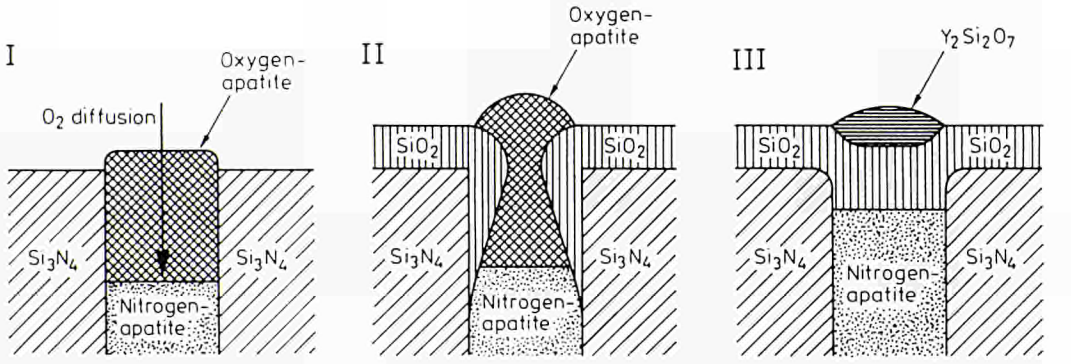
- oxidation in air,
- corrosion in SO_2-air (high pO_2),
- corrosion in H_2S-H_2O-H_2 (low pO_2).

Oxidation in Air

Yttria is used to advantage as a sintering aid in the densification of silicon nitrides largely because it readily forms highly refractory secondary phases yielding good high temperature behaviour. However, these crystalline intergranular phases oxidise readily at low temperature (800°C) with an accompanying volume change which creates surface stresses ultimately leading to cracking of the substrate.

In a doctoral study completed in 1989, this behaviour has been investigated for a hot-pressed silicon nitride containing 9wt% yttria in which the predominant secondary phase is an Y-N-silicate with an apatite structure, $Y_{10}(SiO_4)_6N_2$. From the results obtained the process was found to conform to the model shown in figures below. In stage I, at low temperature, the oxidation of silicon nitride is negligible and the Y-N-apatite oxidises slowly, by oxygen diffusion through the larger grain boundaries, to O-apatite, $Y_{4.67}(SiO_4)_3O$. At higher temperatures in stage II, significant formation of silica begins and the O-apatite is extruded onto the surface of the material. At still higher temperatures, stage III, only the final oxidation products, $Y_2Si_2O_7$ and SiO_2, are evident, the grain boundaries are 'closed' and internal oxidation ceases to be significant. Silica growth is possible only on the external surface of the material.

Below: *Oxidation model for a HPSN(Y) ceramic*

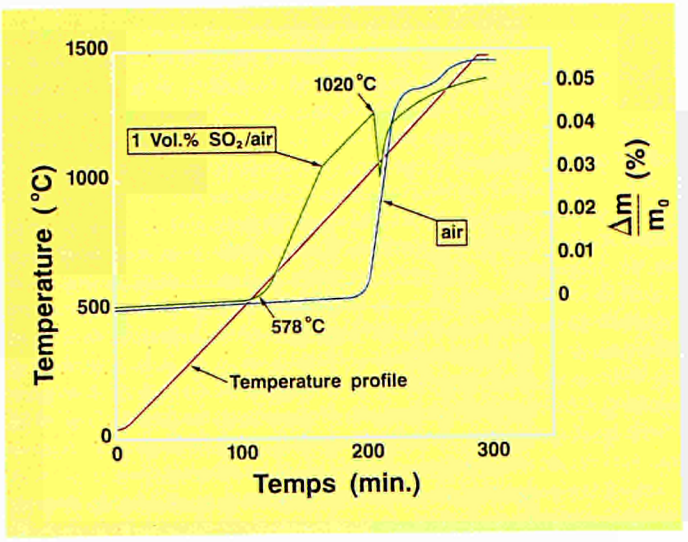


Low temperature ~800°C
no SiO_2 formation
nitrogen-apatite oxidation

Intermediate temperature
$900 < T < 1000$°C
SiO_2 formation $\Rightarrow$
oxygen-apatite extrusion

High temperature
$T > 1000$°C
silicon nitride reaction dominant
$Y_2Si_2O_7$ silicate formation

In a separate project, this oxidation process was observed 'in situ' by hot-stage microscopy. In addition to the simple behaviour described above, at temperatures around 950°C, the surface of the material was seen to 'boil': a liquid phase was evident in which the rapid evolution of (presumably) nitrogen bubbles was clearly observed. Surface analysis after cooling revealed significant amounts of impurity cations, such as iron, calcium and sodium, unevenly distributed over the surface. Clearly the model is a simplified version of reality and the presence of a surface liquid during oxidation is significant. Inward diffusion of oxygen is accelerated and the explosive evolution of nitrogen is believed to be the origin of the destructive pitting corrosion often observed with this type of ceramic.



Above: *Thermogravimetric comparison of the behaviour of a HPSN(Y) ceramic in air and 1 vol% SO_2-air*

Corrosion in SO_2-Air

The combustion of hydrocarbon fuels containing sulphur leads to an oxidising environment in which sulphur dioxide and oxygen are the important corrodants for structural materials in contact with the hot gas.

Experiments with silicon nitrides in SO_2-air mixtures have shown similar processes of corrosion to those in air alone. Oxidation of the surface to silica and silicates predominates with some enhancement of the corrosion at low temperatures which can be attributed to the formation of sulphates of metal cations originating as additives or impurities.

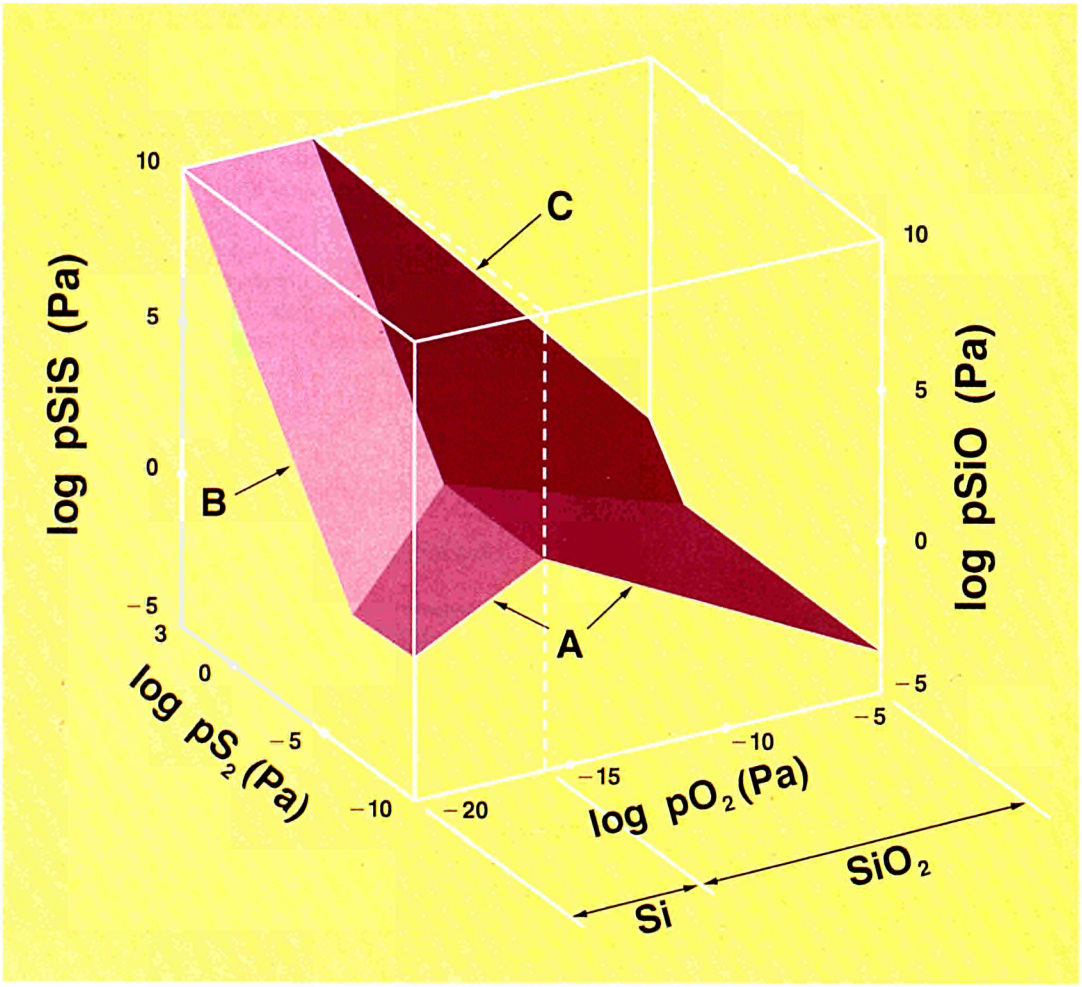
Figure above illustrates, for the same material described in the previous section, the additional mass gain at lower temperatures in SO_2-air than in air alone, primarily due to the formation of yttrium sulphate. Yttrium sulphate begins to decompose at temperatures above 880°C and is completely dissociated at 1140°C in this environment. The figure also illustrates how the oxidation in SO_2-air closely parallels oxidation in air alone at temperatures where the sulphate is no longer stable. At these and higher temperatures, the influence of SO_2 is marginal.

Corrosion in H_2S-H_2O-H_2

The situation is quite different in environments and industrial processes with high sulphur and low oxygen activities where so-called 'active oxi-sulphidation' may occur. Little or no protective scale may form and the primary corrosion products will be volatile SiO and SiS. A competition will exist between these two for the status of 'rate controlling species' depending on the temperature and the precise conditions.

A range of silicon nitrides containing both yttria and alumina in various ratios have been studied in these environments at high temperature. A newly constructed volatility map shows when the surface of the material should corrode as volatile SiO or SiS. In figure opposite above, for 1300°C, three regions are identified:

a, where pSiO > pSiS and active oxidation predominates;
b, where pSiS > pSiO and SiO_2 formation is thermodynamically forbidden and active sulphidation predominates;
c, where pSiS > pSiO but SiO_2 formation is possible. SiO_2, which is quasi-stable, forms at the nitride/scale interface and decomposes to SiO at the scale/gas interface. SiS formation is inhibited by the presence of the scale from which it appears not to form directly.



This thermodynamic model has been confirmed by the experimental data which show that the catastrophic corrosion rates observed in region B (mass losses up to 850µg $cm^{-2}h^{-1}$) may be reduced by two orders of magnitude by small additions of water vapour to the environment, thus shifting the thermodynamic activities into region C.

Above: *Map of pSiO and pSiS as a function of pO_2 and pS, at 1300°C in the region of the Si/SiO_2 stability boundary.*

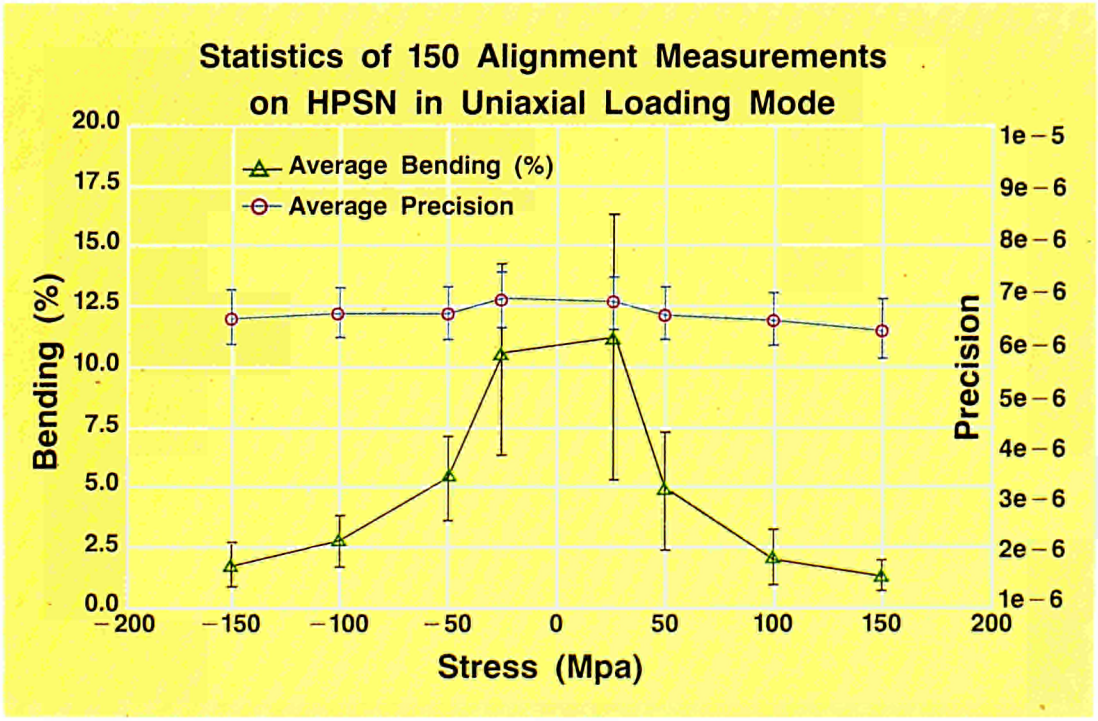
c. Mechanical Properties

In the research is focussed on various topics related to the high temperature mechanical properties of Si_3N_4 and on the design, building and commissioning of special test rigs.

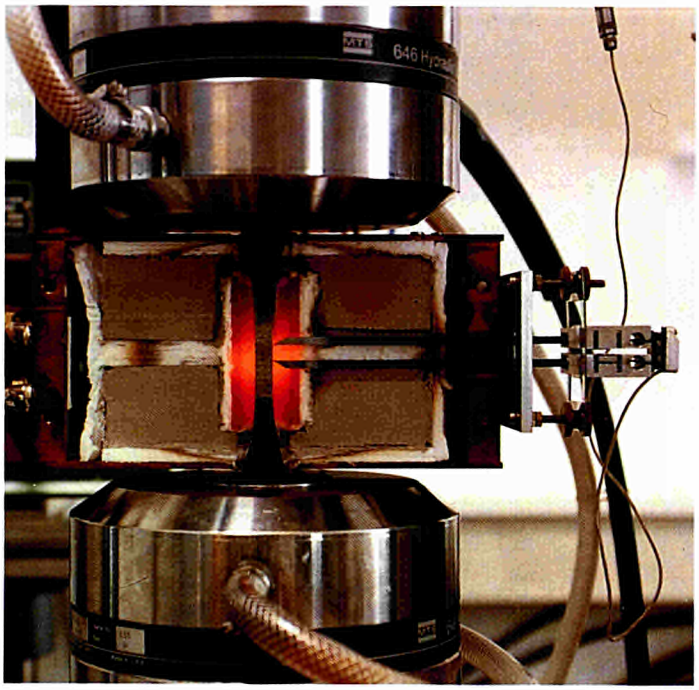
The cyclic fatigue behaviour of Si_3N_4 in the uniaxial testing mode is the subject of ongoing research in the group. The uniaxial mode of testing is selected to circumvent the restrictions of bend testing with regard to the size effect and with regard to the unknown stress distribution away from the planes of maximum stress under time dependent conditions. The major problem associated with the uniaxial testing of ceramics is to obtain excellent alignment in order to reduce the bending component of the average strain to an acceptable level. This has been achieved by means of a loading train consisting of carefully aligned hydraulic grips in a specially aligned testing rig. The quality of the alignment has been tested by strain-gauging a number of rectangular samples in three cross-section planes in order to measure the bending.

The statistics of 150 alignment measurements on Si_3N_4 are shown in figure below in terms of the percentage of bending and of the precision of alignment as a function of the stress applied to the sample. The measurements show that a precision of alignment of $6\text{-}7.10^{-6}$ is achieved reproducibly. This corresponds to an average percentage of bending of 1.25% at the stress level of + 150 MPa. The failure location of fatigue tested samples suggests that this is an acceptable level of bending, although a further decrease is advisable in order to reduce the fraction of invalid tests.

Below: *Percentage of bending and precision of alignment statistics of the cyclic, uniaxial fatigue testing rig*

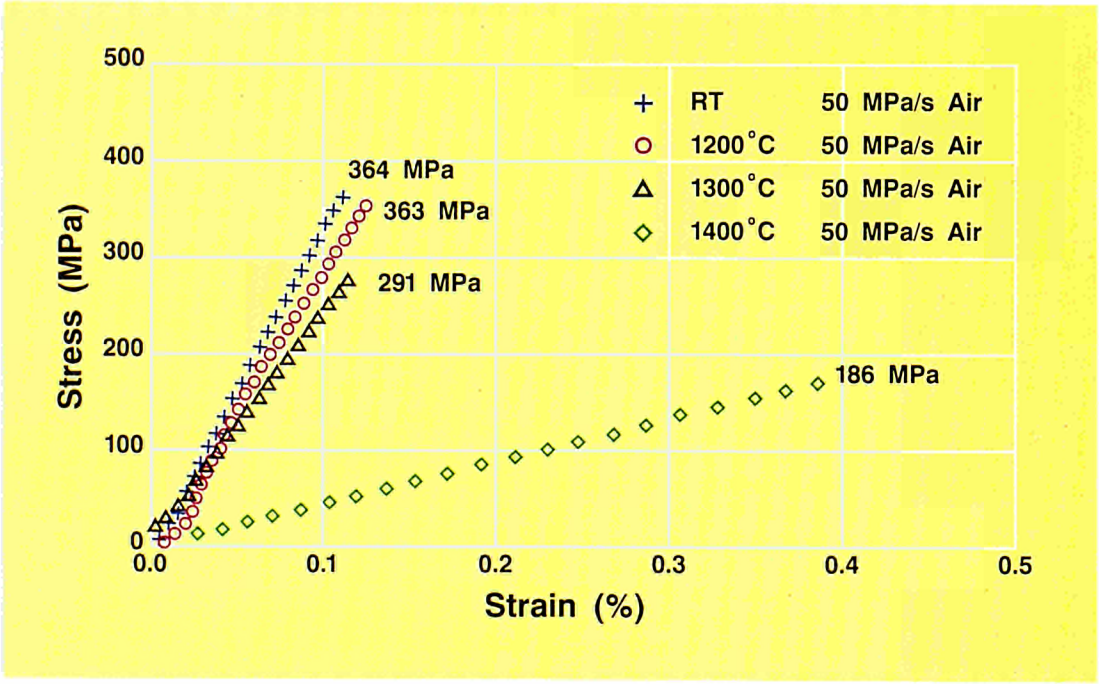


The sample is heated by means of radiation from a susceptor which in turn is heated by high-frequency induction. The figure above is a view on the in-house designed split furnace showing the sample, the susceptor, the isolation and the water-cooled housing of one half of the furnace system. The extensometer rods made of SiC are also visible. Sample temperatures of up to 1400°C are achievable with a temperature gradient over the 10 mm gauge length of less than 0.5% of the temperature for temperatures over the range 1000°C-1400°C. The performance of the testing system is illustrated in the figure below, showing uniaxial tensile data of Si_3N_4 at various test temperatures.



Above: *View on half of the split furnance used in cyclic fatigue testing of ceramics*

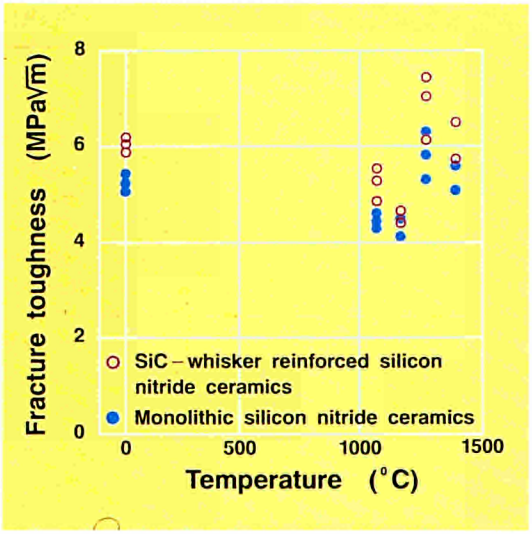
Below: *Uniaxial tensile data of Si_3N_4 at various temperatures*



Subcritical crack growth of both monolithic and SiC-whisker reinforced Si_3N_4 at temperatures up to 1300°C in air is studied through different test methods. The aim is to determine the effect of the reinforcing whiskers and to elucidate the dominating toughening mechanisms as a function of temperature. For this purpose, mechanical tests are combined with in-depth microstructural investigations.

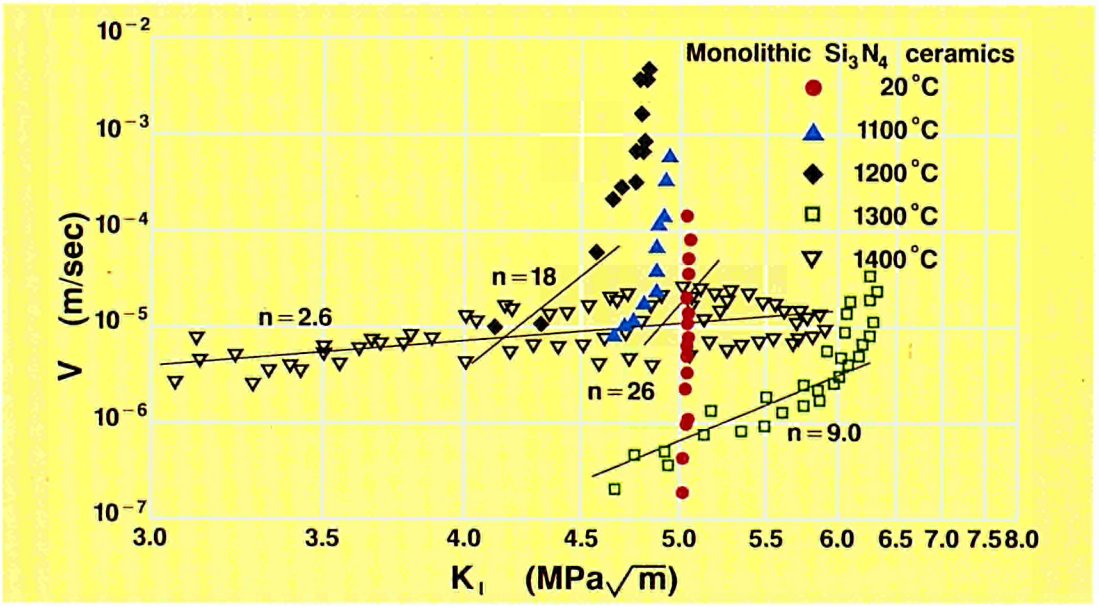
In order to determine the load level for the crack growth experiments, fracture toughness tests on chevron notch beam specimens in four point bending have been carried out in the temperature range RT - 1400°C.
The results for the monolithic and the composite materials are shown in the figure above. As can be observed, the toughness peaks for both materials at 1300°C.

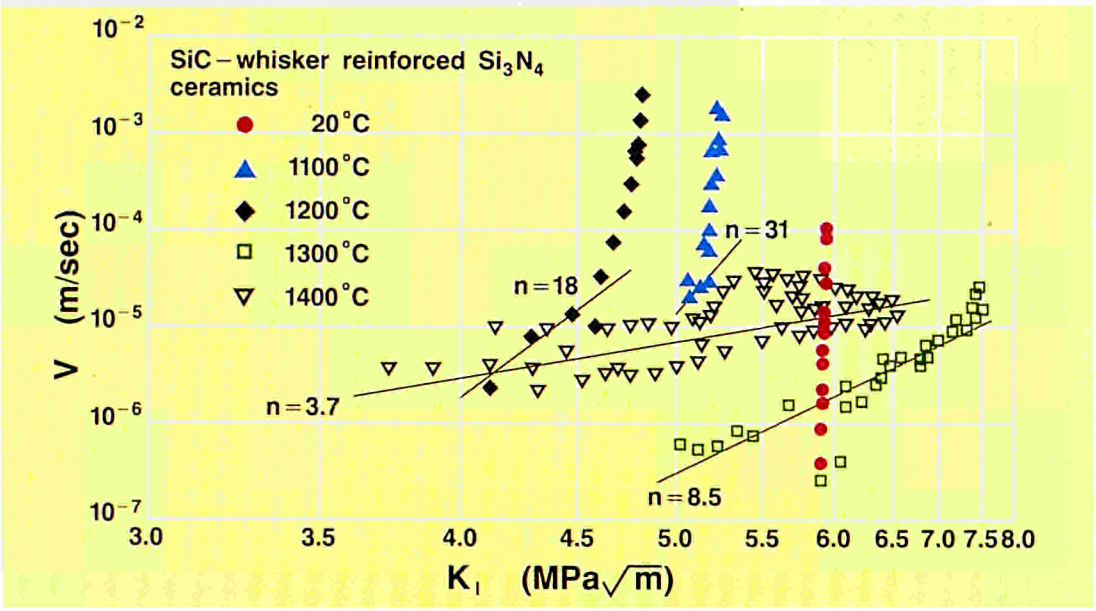
The subcritical crack growth behaviour is studied through chevron notched beam (CNB) tests as well as by the indentation induced flaw method. In the first set-up, the crack growth is followed indirectly through changes in the compliance of the specimen. Stable crack growth can be initiated rather easily and a large part of the (K_I,v) diagram can be covered with a single specimen. The results of the tests on the monolithic and the whisker-reinforced material are shown in figures below and on page 35 above respectively.



Above: *Variation of fracture toughness with temperature*

Below: *Crack growth rates VS. the stress intensity factor at various temperatures for monolithic si_3N_4*





Above: *Crack growth rates vs. the stress intensity factor at various temperatures for SiC-whisker reinforced Si_3N_4*

For both materials, a steady increase in crack growth rate v is observed with temperature up to 1200°C. A sudden drop occurs at 1300°C. Currently, microstructural investigations are underway to study the cause of this behaviour. On comparing figures on page 34 below and page 35 above, it can be observed that the subcritical crack growth rates are rather high (> 10^{-6}m/s). To explore the lower crack growth rate regime (> 10^{-10}m/s), tests are currently performed by the indentation induced flaw (IIF)-method, both under static and cyclic four point bend conditions. Drawbacks of this method are that crack lengths have to be measured and that only one point in the (K_I,v)-diagram is obtained per specimen.
Both types of crack growth experiments describe the growth of an artificially induced crack. The crack growth behaviour of natural flaws is studied by dynamic fatigue experiments in four point bending over a range of temperatures. The results of these tests will be compared to those obtained on artificial flaws.

Several special purpose testing rigs were designed, installed and commissioned in the reporting period. Three pneumatically controlled rigs for four point bend testing on ceramic materials under static or dynamic conditions up to 1000°C in air were designed and installed. The load capacity of the rigs of 100 N maximum is adapted for testing of hollow specimens. A closed-loop pneumatic system is used to load the sample at a constant stress rate to fracture. The stress rate is controlled by a converted Eurotherm 822 programmable temperature controller.

At fracture the system automatically switches off and unloads the specimen, thus preventing the sample from being crushed. The loading train consists of a RDP-miniature load cell with a maximum capacity of 100 N connected to a rolling diaphragm pneumatic piston. The accuracy of the loading system is ± 0.15 N (i.e. under static conditions the load can be held at ± 0.15 N). The maximum controllable loading rate is 180 N/min. Under these conditions the stepping rate is 1.5 N. The fracture stress is recorded using a Keithley digital multimeter with a high and low data memory.
Heating of the sample is by a single zone resistance furnace with a constant temperature zone of 50 mm length. The temperature is controlled within narrow limits by means of a Eurotherm 822 programmable controller. The rigs are designed with the possibility of adding an SLVC displacement transducer for measuring the bending displacement of the specimen.

For loading rates in excess of 180 N/min. and for temperatures exceeding 1000°C a commercially available, high-performance closed loop testing machine was commissioned. The rig is equipped with a $MoSi_2$ furnace with a maximum allowable temperature of 1500°C.

To accommodate four-point bend testing in corrosive environments, four testing rigs with load and temperature capabilities of 2 kN and 1400^{0}C respectively, were redesigned and relocated to the ETL-N laboratory.

Finally the group provides a service activity to other groups in the area of four-point bend tests on two old screw-driven Instron testing machines which were modified to allow for constant load rate tests.

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 and without simultaneous irradiation damage. The results of the study bear relevance to industrial applications where thermal fatigue is the life limiting factor, whereas the addition of an irradiation damage component is aimed at yielding results in support of the design of the first wall of NET.
The progress achieved in 1989 relates in particular to the design of the out-of-pile tube testing rig which will be used to experimentally validate the results of the numerical modelling. The design of the rig is completed, all its constituent parts have been ordered and are delivered.

Assembly of the rig has started although at a lower pace than anticipated because a substantial fraction of the manpower allocated to the project was not made available.
Preliminary thermal and thermomechanical calculations were performed to establish the operating parameters of the out-of-pile rig and of the size of the tubular samples required to achieve the temperature gradient typical of NET over the tube wall thickness. Elastic stress distributions were calculated using the finite element code ABAQUS, assuming free expansion of the tube ends.
Various approaches to the prediction of crack growth life under cyclic thermal gradient fields are under consideration.
In a first step the problem will be tackled by applying an LEFM approach. A corresponding scenario describing the experimental and analytical numerical steps has been put together. In this context an ad-hoc code ('CRKPRO') for thermal fatigue problems in flat plates was developed and a proposal to verify and to optimise this code was presented in the context of this project. For the temperature calculation a finite element formulation is chosen in order to take temperature dependent material data into account.

The Bauschinger effect is modelled by a so-called parallel overlay model. Since under normal conditions the bulk material remains elastic, the crack propagation is expressed in terms of cyclic stress intensity factors.
The stress intensity factors are evaluated in an approximate way by an analytical-numerical integration procedure. It is felt, however, that the importance of this code lies in the application of a line spring model for the characterisation of the stress intensity factors of a semi-elliptical part through surface crack.

Insofar the fatigue data base which is required as input into the numerical model is not available from the literature it wil have to be generated as part of the research project.
For this purpose a closed loop testing machine has been adapted and testing on laboratory type fatigue samples of 316 L has started.

A SUN 386 workstation was installed as a support tool for the analytical numerical work. The software, previously running on a VAX, was implemented on the SUN work station. This required rewriting of the graphic facility parts. In order to take as much advantage as possible of the SUN operating system, the routines concerning the screen lay out and the graphics are being coded in the C language and linked to the Fortran main code.

Operational Defects in Materials and Lifetime Prediction

To improve the lifetime predictions for components operating at high temperatures, it is important to identify and to quantify the microstructural defect state which finally leads to failure. This defect state will be referred to as "damage".
The residual life of a structure can only be reliably predicted if both the actual damage and its evolution in time for a given load-spectrum can be quantitatively described. In this study "creep damage" is considered which denotes the degradation of the material under stress at temperatures above about 0.4 T_m where T_m is the melting temperature in K.
The micromechanisms of this type of damage have been reviewed e.g. in [1,2,3]. It is shown that for many metals and alloys the nucleation and subsequent growth of cavities on grain boundaries is the life determining damage process.

There exist different methods for the assessment of the actual creep damage in a component. Most are destructive.
However, in-service inspection requires non-destructive testing techniques. One method is the measurement of ultrasonic wave velocities [4,5,6] which is showing promising results, but which needs further investigation to take full advantage of its capabilities to detect small microstructural changes.
The JRC activity outlined below aims at obtaining a relationship between the ultrasonic (US)-measurements and creep damage measured by other techniques.
An important part of the programme is to correlate the US-velocity changes with damage parameters used in material models.
Such models describe the macroscopic inelastic stress-strain behaviour including the evolution of creep damage. By this means a more sound basis for residual lifetime prediction can be obtained.

1) Ultrasonic Measurements on Creep Specimens

Up to the present time the major part of the experiments have been performed on a Mn-Cr austenitic steel (trade name AMCR 0033) which is considered as a candidate structural material for fusion reactors [7]. Using flat specimens to facilitate the US-velocity measurements, uniaxial creep tests were performed at T = 923 K.
Some tests were continued until rupture and others interrupted at different stages of the creep test, mostly in the tertiary stage.
US-velocity measurements [8] were performed in the specimens heads and at different places within the gauge length before and after the creep test. Both longitudinal and orthogonally polarized transverse waves with propagation direction perpendicular to the stress axis were applied.

In all the creep specimens the greatest reduction of the sound velocity is obtained when the incident shear amplitude lies in the plane perpendicular to the stress axis (V_S), the smallest if it lies in a plane which contains the stress axis (V_S). This agrees with the result expected for the microstructural damage mechanism (microcracks perpendicular to the principal stress axis) explained in the next section. The reduction for the longitudinal waves lies between V_{S_I} and $V_{S_{II}}$.
A measure for the anisotropy is given by the relative difference

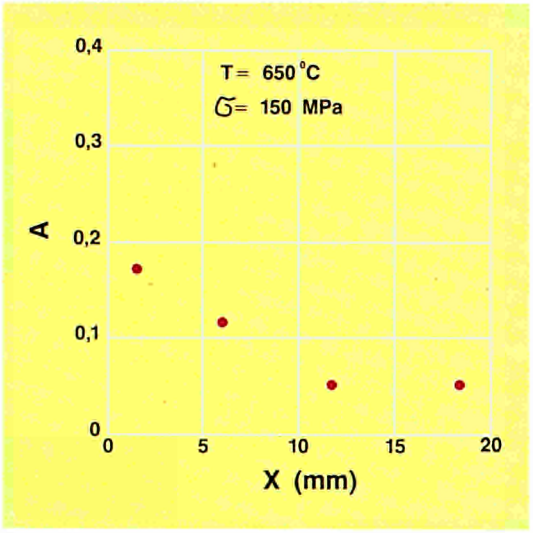
$$q = \frac{V_{S_I} - V_{S_{II}}}{V_{SO}} \qquad (1)$$

where V_{SO} is the reference shear wave velocity in the undamaged material. Knowing the anisotropy in the virgin material q can also serve as a measure for damage. A typical result for q is shown in the figure opposite below for a specimen loaded to 150 MPa which ruptured after 167 h. This figure also contains the values for q measured in an unbroken specimen from a creep test at the same load interrupted after 146 h. Here, and in all other cases an inhomogeneity within the gauge length is observed indicating an inhomogeneous damage distribution. This is probably due to geometrical inhomogeneities of the virigin specimens and needs further investigation.

2) Other Methods for the Determination of Damage

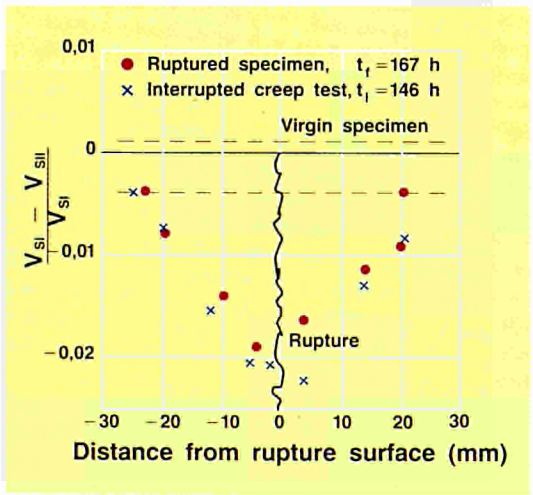
Two other methods to determine the damage have been applied. The first consists of a metallographic evaluation of the microstructural damage. As discussed in [9,10], the origin of creep damage in this steel is given by the cracking of grain boundary facets perpendicular to the stress axis. This is due to a grain-boundary embrittlement because of intergranular carbide ($M_{23}C_6$) precipitations. Following [11,12] the damage is quantified by determining metallographically the number fraction A (referred to as "A"-Parameter) of cracked grain boundary facets which intersect a line parallel to the direction of the applied load.

In [11,12] the A-Parameter was introduced to measure damage due to constrained cavity growth [13]. In this case a cavitated facet acts mechanically as a microcrack. Therefore this concept can be immediately applied to the microstructural damage considered here. The figure above right shows the typical dependence of A on the distance from the rupture surface.



Above: *A-parameter determined in the ruptured specimen t_f = 167 h*

Below: *Values for q within the gauge length*
• ruptured specimen, t_f = 167 h
x interrupted creep test, t_i = 146 h



The shape of the curve corresponds to the US-measurements shown in the figure below for the same specimen.

The second method for damage determination is based on continuum damage mechanics [14]. Damage is quantified by a parameter D which is determined by inversion of the flow law

$$\dot{\varepsilon}_i = f(\sigma, D) \qquad (2)$$

describing the inelastic strain rate $\dot{\varepsilon}_i$ in dependence of the stress σ and D. For $\dot{\varepsilon}_i$ experimental values obtained from the creep curve must be inserted. The material used here can be approximately described by the model of Hutchinson [15] which was extended by Riedel [3], leading to the flow law

$$\dot{\varepsilon} = \frac{B\sigma^n}{1-D} \qquad (3)$$

B and n being material constants. Figure above shows the dependence of D determined from (3) on the time t normalized by the lifetime t_f of the specimen. Assuming $D\alpha\varepsilon^{\gamma}$ (γ : material parameter) the theoretical result is represented by the dashed line independently from the applied stress in the creep test. The experimental results exhibit some scatter but are in rather good agreement with the theoretical curve.

3) Comparison of the Different methods for Damage Determination

The Hutchinson-Riedel model is based on the assumption that the microstructural damage can be described by a distribution of microcracks perpendicular to the principal stress axis. Therefore, according to [16] the relation

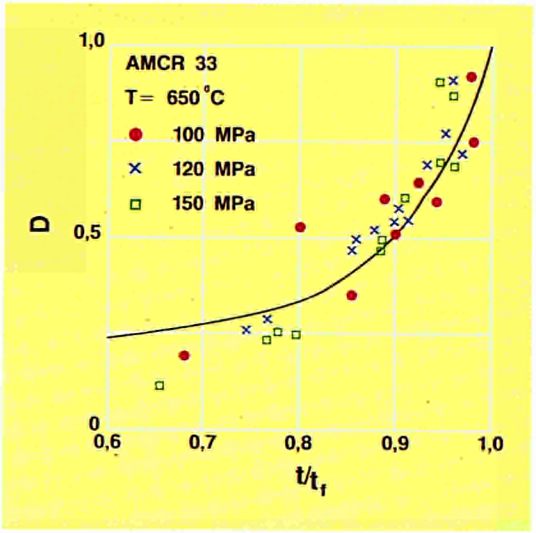
$$D = 0.6 \frac{n+1}{\sqrt{1+3/n}} A \qquad (4)$$

can be derived, which is an approximation for a statistically isotopic grain structure. With n = 8 in the present case A = 0.22 D, and assuming a critical value $D_c = 1$ for rupture, the values presented in figure on page 39 above fit rather well.
A solution for the wave velocities in a material with a dilute concentration of parallel cracks has been given in [17]. Using the same approximations as in (4), the relationship

$$A = -2.51\, q \left[3 - 2\left(\frac{V_{so}}{V_{lo}}\right)^2\right] \qquad (5)$$

between the A-parameter and the relative wave velocity difference q as defined in (1) can be derived. V_{lo} is the velocity of longitudinal waves in the undamaged material. As shown in the figure on page 39 below a value of q = -0.02 was measured putting the center of the US transducer with a crystal diameter of 6.35 mm at a distance of 6 mm from the rupture surface.

With (5) and $V_{so}/V_{lo} = 0.57$ this leads to a value A = 0.1 which coincides reasonably well with the average result in this zone as shown in the figure on page 39 above.



Above: *Damge-parameter D determined from the creep curve. Dashed line calculated with the Hutchinson-Riedel model*

4) Summary

Results of different methods for creep damage measurement in an austenitic steel are presented. It has been briefly outlined how they can be compared and how they can be used in a material model. Of course, creep experiments show considerable scatter and further investigations have to be performed on a larger data base. In addition, other materials will be studied.

References

[1] A.C.F. Cocks, M.F. Ashby, Progress in Materials Science 27 (1982) 189-244.
[2] M.F. Ashby, B.F. Dyson, "Creep Damage Mechanics and Micro-mechanisms", Math. Phys. Lab. U.K. Rep. DMA(A)77, March 1984.
[3] H. Riedel, "Fracture at High Temperatures", Springer Verlag Berlin, Heidelberg, New York 1987.
[4] U. Stigh, Eng. Fract. Mech. 28 (1987) 1-12.
[5] H.M. Ledbetter, R.J. Fields, S.K. Datta, Acta Metall. 35 (1987) 2393-2398.
[6] H. Willems, in: Proceedings of the Int. Conf. "Life Assessment and Extension", June 13-15, 1988, The Hague/The Netherlands.
[7] G.Piatti, P. Schiller, J. Nucl. Mat. 141-143 (1986) 417-426.
[8] F. Lakestani, P. Rimoldi, Proc. of the 3rd Int. Symposium of the Non-Destructive Characterization of Materials, October 3-6, 1988, Saarbrücken (FRG).
[9] R. Matera, Thesis, Politecnico Milano (1988).
[10] E. Ruedl, R. Matera, G. Valdré, J. Nucl. Mater. 151 (1988) 238-244.
[11] M.S. Shammas, in: Conference on Life Extension and Refurbishment of Steam Plant, Institution of Mechanical Engineers, London, 1987, pp. 289-299.
[12] M.S. Shammas, in: International Conference "Life Assessment and Extension" VGB, KEMA, EPRI, Essen, Arnheim, Palo Alto, 1988, pp. 238-244.
[13] B.F. Dyson, Met. Sci. 10 (1976) 349-353.
[14] J. Lemaitre, J.L. Chaboche, "Mécanique des Matériaux Solides", Dunod (1988).
[15] J.W. Hutchinson, Acta Metall. 31 (1983) 1079-1088.
[16] H. Riedel, Lecture given at: DGM-Symposium Festigkeit und Verformung bei hoher Temperatur, Bad Nauheim, April 1989.
[17] M. Piau, Int. J. Engng. Sci. 18 (1980) 549-568.

Wear and Corrosion Resistant Coatings

Introduction and Objectives

Lifetime and load capacity of tools and other construction elements depend both on how the particular material reacts to wear as well as on the working conditions the part is subjected to. For this reasons wear and corrosion reduction is of great economic importance. The development of protective coatings has become in recent years an important branch of advanced material technology. 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 operation in oxidising and high temperature environments.

The activity 'Wear and Corrosion Resistant Coatings' has the objective to contribute to an improvement of both existing coatings and to a 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 of the thin films laboratory of the IAM Ispra. Surface modification techniques as e.g. ion implantation, ion beam mixing and laser treatment might successfully be used to modify hardness, adhesion to the substrate and structure of the film.

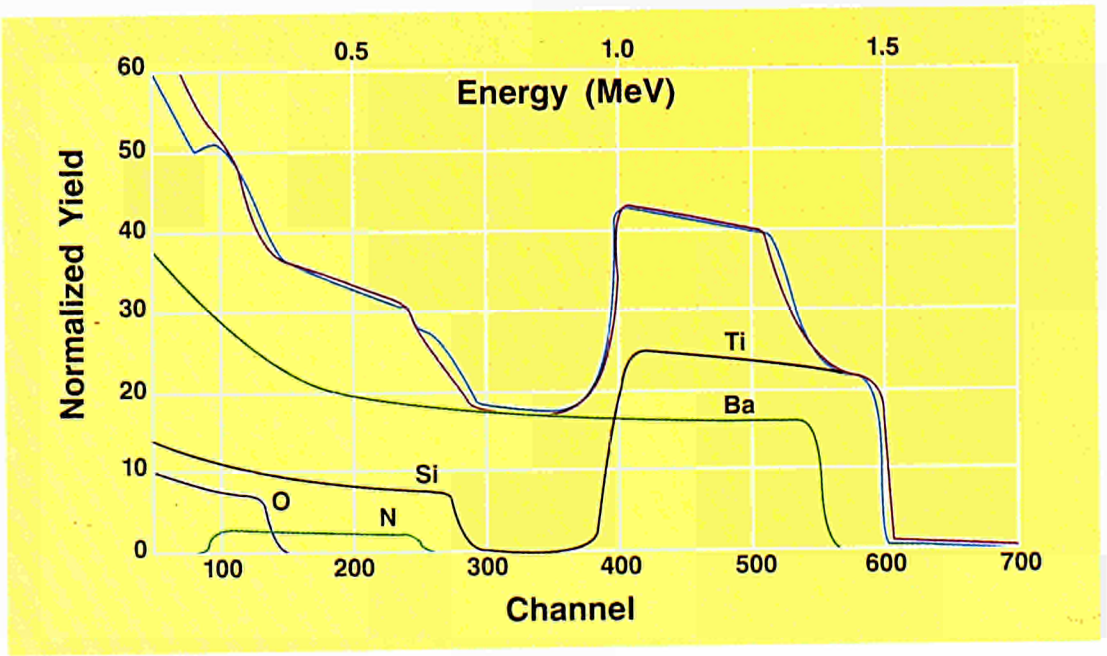
Results

1. Low Temperature Deposition of Titanium Nitride Films

Objective of the investigation was i) to become familiar with the technique of metal nitride deposition, ii) to investigate the deposition at low substrate temperatures and the resulting film properties, iii) to prepare samples for a later surface modification by the ion implanter. The work was performed in close collaboration with K. Reichelt of the KFA Jülich.

TiN_x films of different composition x were prepared by reactive ion beam sputtering at substrate temperatures of 77 and 300 K.
The films were characterized with respect to microstructure, electrical resistivity, residual stress, hardness and Young's modulus in dependence of x.
Figure below shows a RBS spectrum of a TiN film sputtered on a glass substrate.

Below: *Experimentally determinde RBS spectrum of a TiN film on a glass substrate containing the element barium*



The chemical composition of the film (and substrate) has been obtained by modelling a spectrum from single element contributions.
Glancing angle X-ray diffractometry indicates a mixed phase of Ti, Ti_2N, TiN, TiN at $x < 0.05$ and of cubic TiN at higher values of x. Considerably high stress values up to 12 GPa and hardness values up to 25 GPa were found for films deposited at 77 K.

Figure above shows the microhardness as a function of the flow ratio of Ar and N_2 at which the films was prepared.
These films consisted of very small grains of 150 Angstroem in the average. The electrical resistivity showed the usually observed dependence with a minimum of 54 microohm-cm at nearly stoichiometric composition.

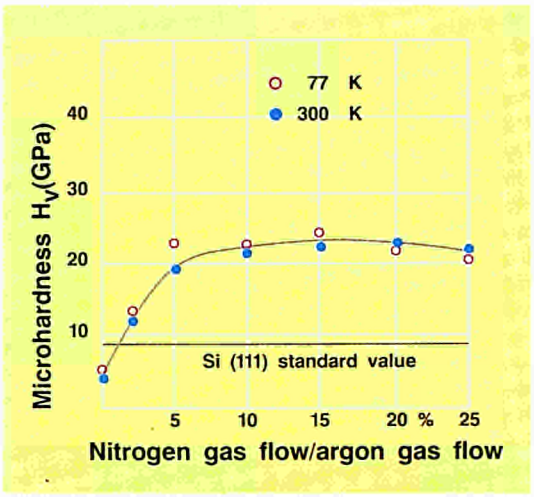
2. Plasma Sputter Deposition of Boron Nitride Films

The stable phase of BN has a h.c.p. structure, but there are also metastable phases among which a cubic phase (c-BN) with a diamond-like structure and also similar mechanical properties (high hardness and high Young's modulus).
Due to these properties but also due to a much higher temperature resistance this phase is of particular interest.

Following several procedures described in the literature we tried to obtain c-BN on steel substrates by a reactive sputter deposition process applying high bias voltages (up to 200 V).
The films were analyzed by glancing angle X-ray diffractometry and IR spectroscopy (for these investigation deposition was made on polished Si wafers) and SEM.

Both methods gave no indication for the presence of a cubic phase; however, the diffraction patterns which were obtained did also not display the stable and the non-cubic metastable phases, which have been observed so far.

The diffraction lines are extraordinarily broad and suggest the existence of a nanometric grain size. Presently we are trying to obtain a picture of the microstructure by comparing the experimentally observed diffraction pattern with calculated ones based on models of various grain size, shape and orientation.



Above: *Microhardness of TiN films, which have been sputter deposited at different gas flow ratios of nitrogen and argon, respectively*

3. High Temperature Corrosion Resistant Coatings

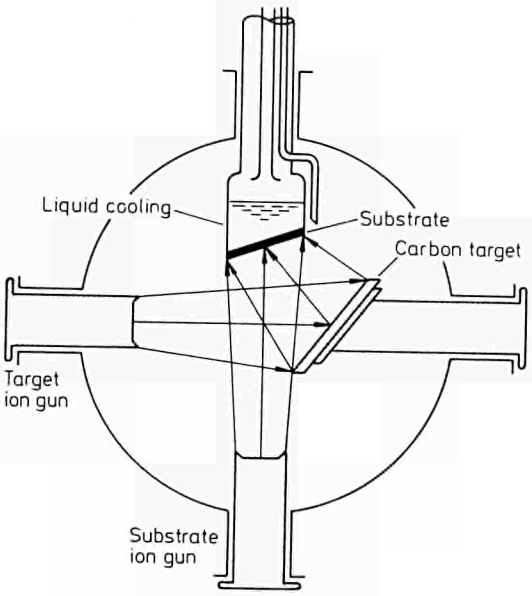
The beneficial effect of active elements (Yttrium, Hafnium, rare earth metals etc.) on the oxidation behaviour of high temperature resistant coatings is well known.
Usually the active element is added directly in the melt of the base material. In this study, however, we try to incorporate the active element only into the outermost surface layer of the base material by using the techniques of ion implantation and ion beam mixing.

This might offer the possibility to improve the performance of the coatings, because ion beam techniques allow to shape and vary the height of the concentration profile within certain limits in contrast to conventional methods. Furthermore such a techniques might be also economically advantageous because the expensive active elements is only restricted to a thin surface region.

First ion beam mixing experiments were performed on a FeCrAl type base material and Y was used as active element.

By plasma sputter deposition from an Y target (99,9%) a layer of 3000 Angstroem was deposited onto sheets of the base alloy. Then in a second step the layer was mixed into the base material by an Ar^+ ion beam of 200 keV at various doses ranging from 10^{15}- 10^{17} ions/cm^2.
Direct implantation of Y atoms will be performed in a later time as soon as the Y ion source of the implanter will be available. High temperature corrosion experiments are in course with the mixed samples.

Below: *Dual ion beam deposition facility*



4. Hard Carbon Coatings

Carbon coatings offer the unique combination of extreme hardness, low friction, chemical inertness and optical transparency.
However, these properties depend sensitively on the method and parameters of deposition. So far much work has been done using CVD, rf and dc plasma sputtering techniques, but only few studies on the preparation by ion beam techniques are known. This study has the objective to investigate this method in more detail.

For this purpose a dual ion beam deposition facility was constructed (Figure below). It consists of two ion guns allowing two modes of operation namely i) direct deposition of carbon ions from gun 1 using e.g., CH_4 as working gas or ii) sputter deposition from a graphite target using gun 1 to generate a sputter beam with e.g. Ar as working gas. In both cases gun 2 can be used to assist the deposition process by a simultaneous bombardment of the deposited layer by low energy Ar ions. First deposition experiments have shown the feasibility of the facility.

For the characterization of the films three different methods are presently under investigation (in collaboration with other JRC Institutes): IR absorption, XPS and Raman spectrometry. These methods should reveal the ratio of sp_3 to sp_2 bonds contained in the produced films. Difficulties arose in IR absorption due to lack of suitable substrates, in the case of XPS due to lack of standard reference material and in the case of Raman spectrometry from instrumentational limitations, which are being resolved.

Properties, Performance, Characteristics and Innovation of FUNTIONAL MATERIALS

Characterization of High Tc Superconductors

Introduction and Objectives

High Tc ceramic superconductors have become one of the most intriguing and fascinating subjects in solid state physics. Their technological relevance, especially in the magnetic and electronic industry (SQUID, fast computers) is shown by the considerable effort developed in all countries in order to develop suitable ways of preparation and utilization of this class of compounds. However, it is felt more and more that only a basic understanding of their superconduction phenomena can allow a more rational development after the initial rush in the past two years. Effort has been concentrated on the copper based perovskite structured superconductors, especially YBaCuO and BISCO (Bismuth-Strontium-Calcium-Oxygen compound), and in particular on the following questions:

- is there a thermodynamic, possibly model-free theory of superconducting Fermion pairs in the reciprocal space which would give a coherent and compact explanation of the most peculiar features of this class of compound (e.g. their high critical temperatures, their short coherence length, observed features of non-monotonic behaviour in Tc's vs carrier concentration, short coherent lenghts, anisotropy)?
- can the transport properties of a simple copper oxide system (e.g. CuO_{2+x} lead to the understanding of the assumed role of Cu^{+3} (or Cu^{2+x}) in high Tc superconduction?
- in the bismuth containing compound, can a relationship be found between the crystal structural data and electronic properties as they may be determined by electron spectroscopy?

In the present report, some preliminary answers and progress on each of these points will be discussed.

A Generalized BCS theory for High Tc Superconductors

An important tool for the study of critical phenomena in interacting systems is interaction thermodynamics, especially when it can be applied in a model-free way. We have therefore attempted to employ a statistical method, Spacing Statistics, which has been used in the past for different interacting systems (1,2). In this case, the statistics was applied in the appropriate reciprocal space for Fermion-Fermion interaction.

Our statistical analysis of the interaction between Fermions uses a general appropriate attractive Boson mediated interaction potential between charged Fermions as employed in BCS theory for metals. However, we have avoided the usual approximation of a constant interaction employed in standard treatments of BCS. No special hypothesis has been made either on the nature of the interacting Fermions (whether electrons, or polarons or any other form of particle coupled with a lattice polarization) or on the nature of the mediating Boson (whether a phonon magnon or other). In these terms, the thermodynamic treatment is model-free.

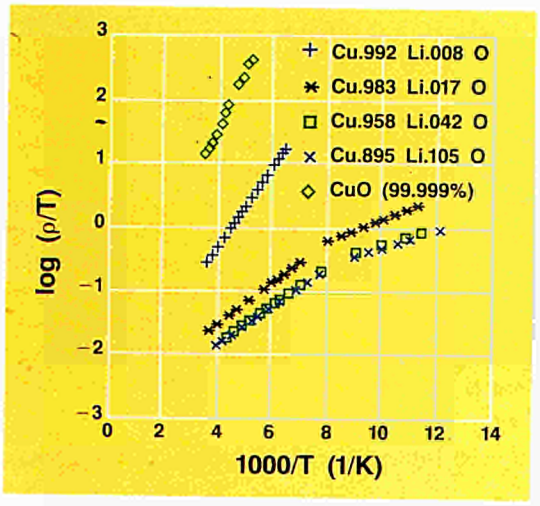
Starting from the interaction potentials, the treatment defines appropriate interaction envelopes (or "spacings") in the reciprocal space for the Fermion-pair interaction, and calculates the probability of the existence of Fermion pairs with Boson couplings with specific momentum exchanges (therefore rejecting the "constant interaction" approximation).

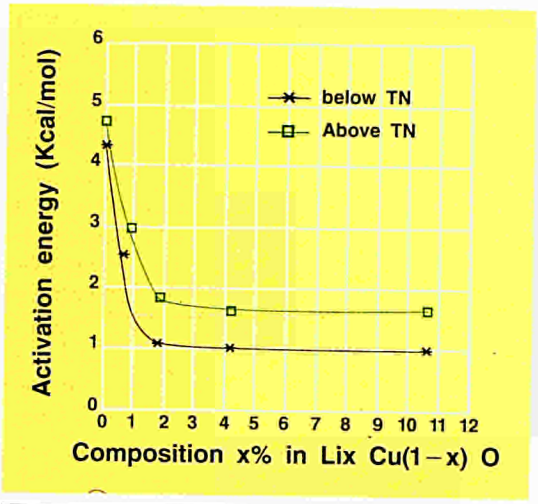
By making use of the instability condition which is in-built in Spacing Statistics, the BCS relation between Tc and the Boson and Fermion quantities is obtained. The Tc expression reproduces the well known BCS expression for "classical superconductors" when hypotheses are made for the Fermion and Boson quantities which are appropriate for electrons in metallic systems. However, a more general expression is obtained for Tc, i.e.

$$kTc = (M^2\,\sigma)\,\exp(-(1/N_F U))$$

where N_F and U are as, in classical BCS, the density of states and the value of the interaction at the Fermi level, whereas M^2 is a Boson-Fermion coupling matrix element and σ is proportional to the damping ($\sigma = X\Gamma$; where X is the ratio between the coherence length and the lattice constant and Γ a damping factor).

It is seen that, given a value for M^2, the T_c may be increased by a lowering of both the coherence length and the damping of the oscillating pair. This suggests immediately that high critical temperature may be obtained whenever the oscillation of the Fermi pair responsible for superconduction is scarcely damped. This may occur whenever the coupling is "strongly localized" in the lattice so





Above left: *The depence of log (ρ/T) vs. 1000/T (K^{-1}) for five compositions x% in $Li_xCu_{(1-x)}O$.*

Above right: *The depence of the activation energy (Kcal/mol), above and below the resistivity change of slope, on the dopant concentration.*

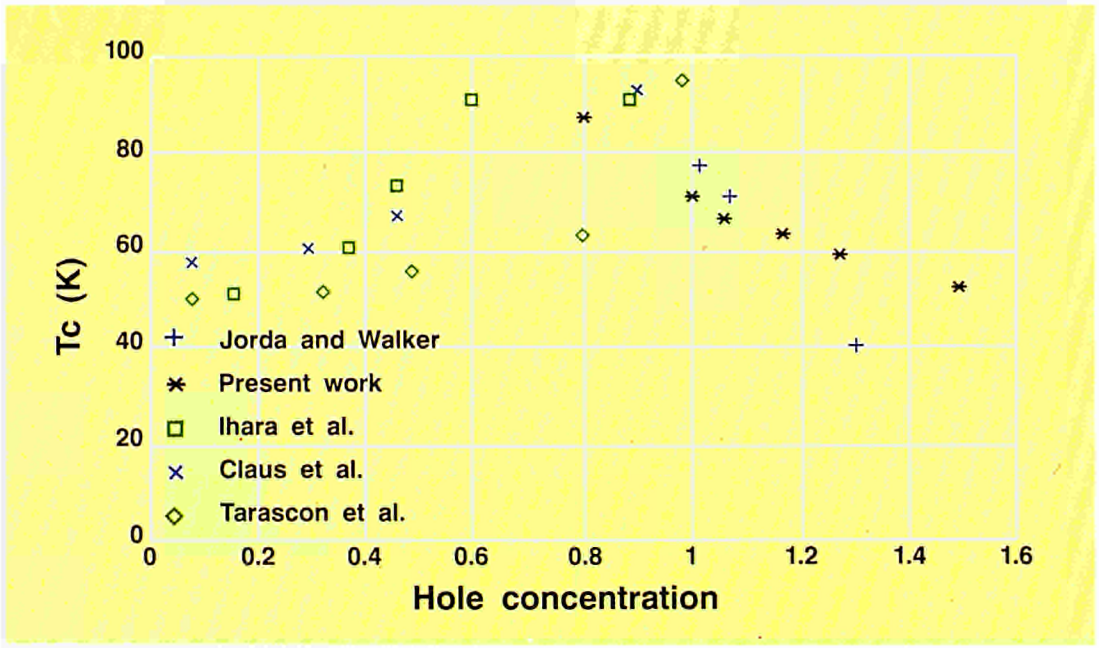
that only Bosons of similarly "very localized" nature (e.g. optical phonons) may be responsible for damping or breaking up the oscillation (long range collective Bosons, e.g. acoustic phonons, will not affect the damping process).

Since both M^2 and σ depend on the density of states function N, which, in turn, depends on the density n of the carriers which are responsible for the superconductive phenomenon, the equation above is not necessarily a monotonic curve when the density of carriers n is varied. In fact, in ceramic high Tc superconductors in which the density of carriers n is varied by suitable doping, the presence of a maximum of Tc vs n has been observed (refs. 2 to 10) figure above left, in which data on Li-doped YBaCuO by F. Lanza and R. Feduzi (11) are also reported).

A further manipulation of the above equation, together with the simplest assumption for the dependence on n of the density of state function N, leads to a useful linearization, which can be compared with experimental data (Figure above right, in which data on Li-doped YBaCuO by F. Lanza and R. Feduzi (11) are also reported). In this figure, the 1/U quantity in the above equation has been extracted for Tc measurements from different Authors (Ref. 2 to 10) in different high Tc ceramic superconductors and plotted vs $n^{1/3}$. The coherence length parameter has been used as a variable in order to obtain the best linearization. The striking feature is that all the data fall on a fairly good straight line for a coherent length Xa = 10A, which compares extremely well with the short coherence lengths observed in these superconductors by transport and magnetic methods. This shows that this thermodynamic treatment, assuming a general Boson mediated Fermion attraction, is able to describe a very large part of the high Tc superconductors in a simple, unified way.

A further result of the above equation and discussion is that the theory predicts a depression of the isotopic effect in superconduction for the coherence lengths usually encountered.

The depression of the isotopic effect, which is found experimentally, is one of the most intriguing problem of this class of superconductors.



Above: *T_c dependence on hole concentration of 123 compounds. The data from Tarascon et al. appear to deviate significantly from the data of Ihara et al. and Claus et al. which show a plateau around 50 k.*

Effects of Lithium Oxide on the electrical properties of CuO at low temperature

In all copper based superconductors, the superconduction behaviour is attributed either to the electronic structure of Cu^{+3} (or at least Cu^{+2+x}) or to changes in the oxygen band caused by the $Cu^{+2} \rightarrow Cu^{+2+x}+xe$ reaction (the latter explanation, which leads to a hole conduction mechanism, is perferred). It is therefore of interest to study the electrical transport in CuO, a simpler copper oxide, when the concentration of Cu^{+2+x} ions is varied by a suitable monovalent dopant. We have chosen Li^{+1} as a dopant in view of its ionic radius which makes it a valuable substitutional ion for copper, and its high ionization energy which assures that it is a monovalent ion.
The influence of four different lithium concentrations on the electrical properties of CuO have been therefore investigated. X-ray measurements have revealed a single phase up to 4.2% of Li, whereas a second phase Li_2CuO_2 appears at higher Li concentrations. The log (ρ/T) vs. 1/T plots (see figure above), are better represented by two straight lines than by one. It is recalled that the ρ vs T dependence employed is characteristic of a hopping mechanism. An abrupt decrease of the conduction activation energies is already observed for Li concentrations less than 2% at.
For higher concentrations the decrease is much lower (Figure page 50 below).

The change in slope observed at T = 230 K in the resistivity measurements as a function of temperature at all Li-concentrations can be associated with an antiferromagnetic order transition, which already exists in CuO. Calorimetric measurements reveal a specific heat anomaly which is also evidence of this transition. However, specific heat anomalies of this kind are also found in high Tc superconductors in this temperature range, (e.g. YBaCuO at the same temperature).
Further investigation and an attempt to correlate these results with the properties of copper based superconductors are under way.

Structural determinations and electron spectroscopy on Bismuth - containing Perovskite type ceramic compounds

An interesting observation, now in course of further elaboration and interpretration, has been performed in BISCO.
A BISCO batch, prepared by J.C. Spirlet in the JRC-Transuranium Institute of Karlsruhe, which displayed superconducting properties immediately after preparation, lost its superconducting properties during the transport to Ispra. This is not unusual, because the Bismuth-containing superconductor is known to be extremely unstable.

However, when half of the material was heat treated at 870°C in air the superconduction properties were restored with a Tc = 80 K. The two half parts of the compounds (which will be termed hereafter superconductor and non-superconductor) have the same cationic composition, and show the same monophasic diffraction pattern from BRAGG - BRENTANO X-ray analysis.

The two parts were subsequently compared by performing the following experiments:
a) X-ray Absorption Near Edge Structures (XANES) (at the Frascati Synchrotron Radiation Facility);
b) X-ray Photoemission Spectroscopy (XPS).
Both methods are sensitive to the electron charge density in the bonds of the relevant constituting ions.

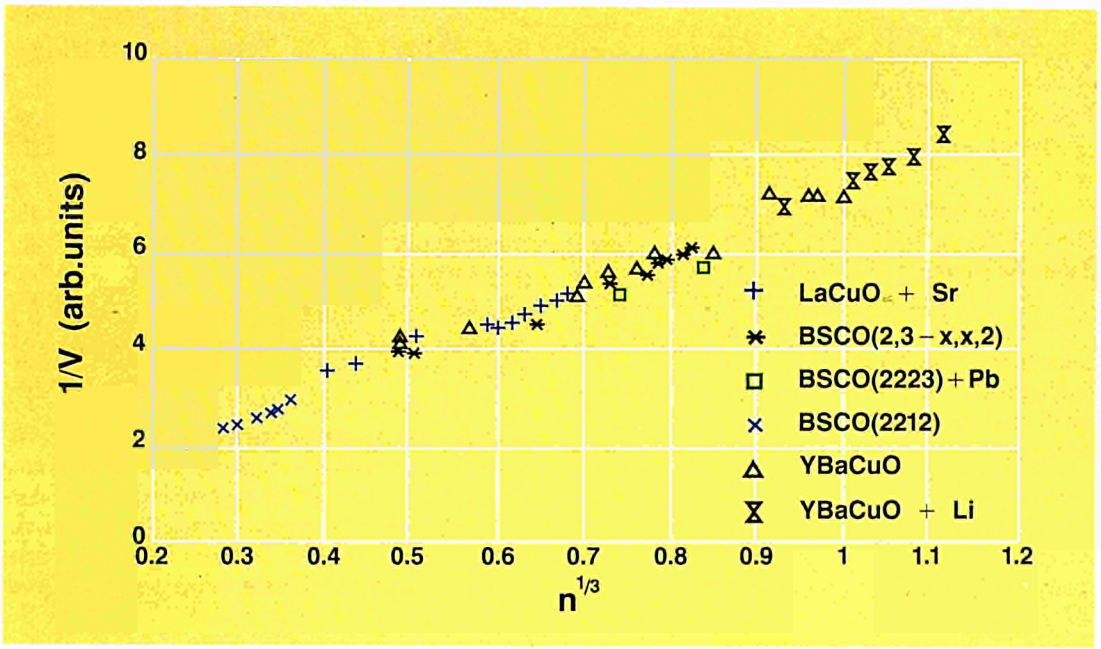
The following observations were made:

1 from both XANES and XPS analysis, the Calcium ions in the superconductor appear to occupy two non-equivalent positions. If this is not due to a second phase undetected by the Laue method, the observation would suggest a distortion in the Perovskite lattice, which may cause a distortion in the overall electron charge density. Superconduction, in this case, is correlated to this distortion;

2 the Bismuth core levels, as measured by XPS, indicate the presence of Bi^{+3-x} in the superconductor, but not in the non superconductor; this will be discussed later.

3 the valence band spectrum of the superconductor, sensitive to the external binding electrons, would indicate a stronger hybridization (or covalent bonding part) in the Cu-O bond in the superconductor than in the non-superconductor (see also 12).

Below: *1/V (arbitrary units) plotted vs. hole concentration as explained in the text. All of the data linearize fairly well on a single curve. + Fisk et al., Ref. 3, * Urland and Tiez, Ref. 4, Wang et al., Ref. 5, Mamgung et al., Ref. 6, Tarascon et al., Ref. 5, Ihara et al., Ref. 7, Claus et al., Ref. 8, Present work, Ref. 11.*



Observations 2 and 3 are of particular interest for the understanding of BISCO (as well as of other Copper - containing Perovskite superconductors). In a physico-chemical model that is being developed, the effect of the Bismuth ion on the Copper ions of the Perovskite basal planes would cause a charge transfer for electrons from Cu to Bi, thus creating a number of Cu^{+2+x} ions and a more covalent Cu-O bond. Current ideas on this class of compounds tend to attribute the conditions for p-type superconduction to the presence of Cu^{+2+x} and to covalency.

An important factor that is not well known and is frequently disregarded, is the influence of possible non-stoichiometric deviations in BISCO due to absorption or desorption of oxygen in the open Perovskite structure.

References

1. L. Manes, "Fundamentals of spacing statistics and its application to the defect structures of high temperature nonstoichiometric phases", High-Temperature High Pressures (20), 239-246 (1988)
2. L. Manes, and V. Dallacasa ICMAS-89, Modern Aspects of Superconductivity, Proceedings, Paris, France (1989)
3. Z. Fisk, S.W. Cheong and D.C. Johnston, MRS Bulletin, January 1989
4. W. Urland and F. Tiez, Solid State Commun. 69, 995 (1989)
5. W.N. Wang et al., Supercond. Sci. Technol. 2, 55 (1989)
6. C. Namgung et al., Supercond. Sci. Technol. 2, 181 (1989)
7. J.M. Tarascon et al. Phys. Rev. B 36, 226 (1987)
8. H. Ihara et al., Physica C 153-155, 948 (1988)
9. H. Claus, G.W. Crabtree, W.K. Kwok and B.V. Veal, Physica C 153 155, 846 (1988)
10. J.L. Jorda and E. Walker, European Workshop on: High Tc Superconductors and Potential Applications, Proceedings, Genova, Italy (1987)
11. L. Manes, F. Lanza. V. Dallacasa and R. Feduzi, "Interpretation of Tc depence on carrier density of high temperature superconductors by a generalized BCS theory", sent to Phys. Rev. Letters.
12. F.U. Hillebrecht et al., Phys. Rev. B 39, 236 (1989).

Low Activation Steels

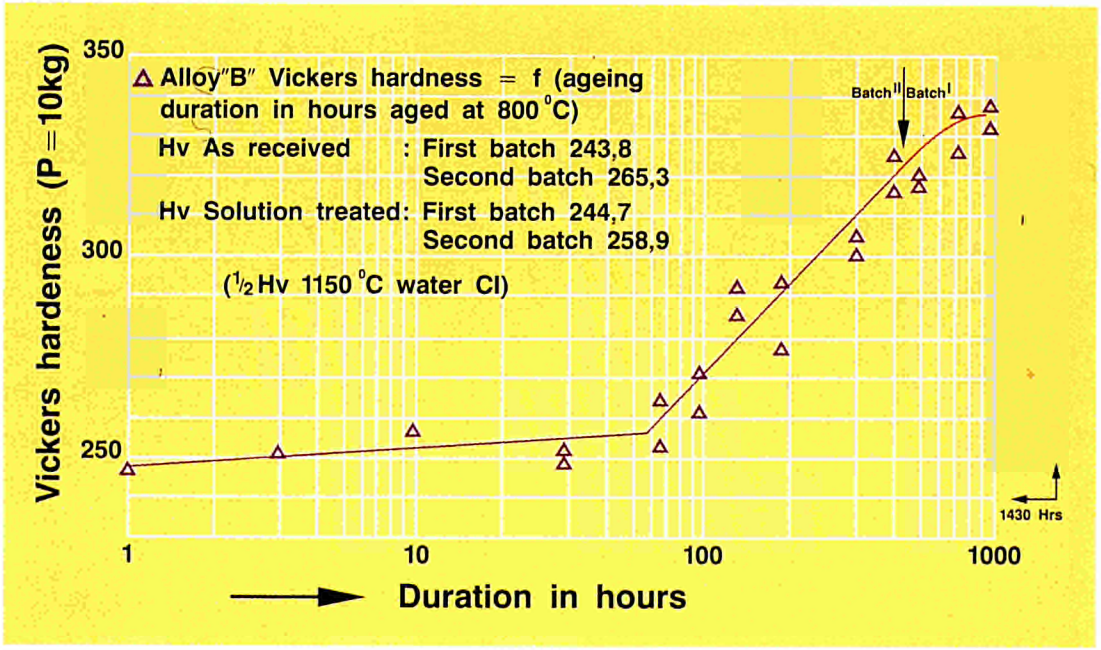
Optimisation of Chronium - Manganese Steels

The properties of the steels IF-B and IF-D (Table I-2 page 88) have been optimised by a heat treatment which induces precipitation of a strengthening dispersion of V and W carbides. A first series of treatments at 400,700 and 900°C has been carried out.
At 400 and 700°C the precipitation process is extremely slow. After 1000 hours the optimum hardness was still not reached, while at 900°C the process was too fast for a reproducible treatment. A metallographic study evidenced the fact that not only annealing temperature and time were important, but also that temperature and time of the preceding solution treatment had a strong influence on the hardening process.

A measurement of the ferrite content after solution annealing by means of a Fischer Ferritescope showed a ferrite content always between 0.1 and 0.4%, and rather independent from the treatment.

The procedure which was finally chosen was a solution treatment of 30 min at 1150°C and water quench, with an additional heat treatment at 800°C and a slow air cooling. For alloy IF-B, an increase of the Vickers hardness of 35% can be obtained (figure below).
The alloy IF-D can also be hardened by a precipitation of carbides. The increase after a slightly modified procedure was similar to the alloy B. Tensile tests on both alloys in the optimum annealed condition are under progress.

Below: *Vicker hardness as a function of ageing duration at 800°C for Alloy "B".*



Composite Materials Properties Improvements

A) Al-SiC metal-matrix composites

Metal matrix composites (MMCs) have been the subject of intense development in the last decades due to their interesting features such as high specific strength and stiffness at room and high temperature as well as good wear resistance and thermal conductivity.

Recently, ceramic particulate reinforced metal matrix composites have been developed with promising results by several laboratories and industries using molten metal mixing processes.
To achieve the goal of commercial applications, further development of microstructural and mechanical properties (above all ductility) and fabrication processes and their reproducibility is necessary. The gradual deterioration of these materials due to the nucleation and growth of microcracks is the most important damage mechanism. To develop a better understanding, the following studies on Al-SiC metal matrix composite have been started:

i) microstructural observations, optical metallography, scanning electron microscopy, energy-dispersive X-ray elemental analysis and X-ray diffraction analysis of the composite in the as-received conditions and after remelting by conventional foundry techniques (sand cast and permanent mold cast);

ii) tensile tests at room temperature on specimens taken from the composite in the as-received conditions and from an unreinforced aluminium alloy (same nominal composition as the matrix);

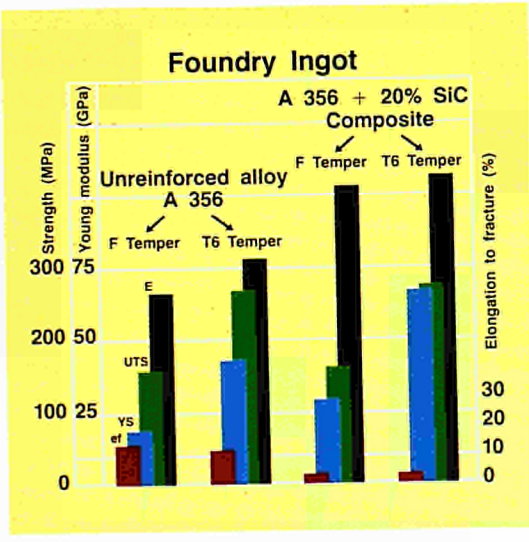
iii) microstructural (quantitative metallography) analysis of selected deformed composite specimens.

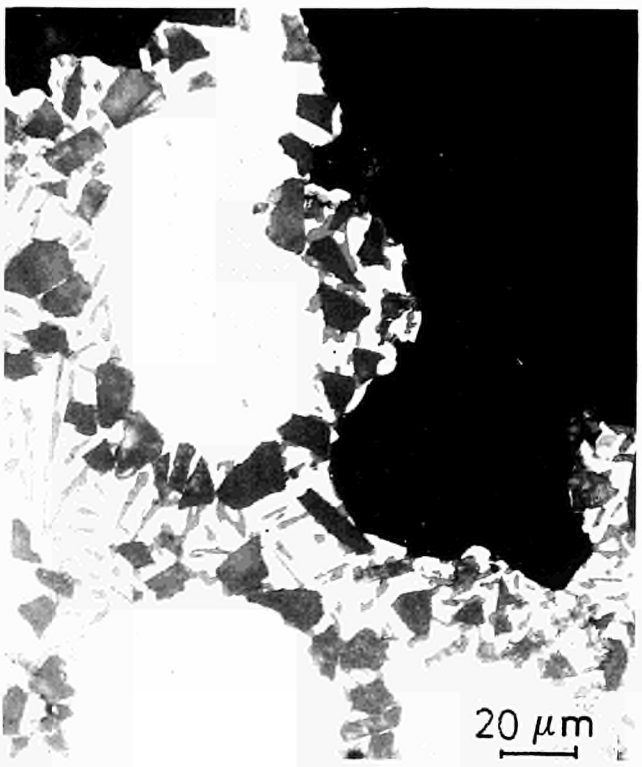
The material studied was supplied by Agusta-FOMB (Benevento Italy) in the form of foundry ingots. It is a particulate SiC-reinforced Al composite characterized by a dispersion of a nominal particle volume fraction of 20% in a matrix constituted of an Al-Si-Mg alloy, A356. The SiC reinforcement is added to the matrix after a surface treatment which improves the mixing process with the molten metal. For comparison purposes, an Al-Si-Mg alloy, A356 ASTM in the form of a foundry ingot without reinforcement, has been considered. According to ASTM standards the nominal chemical composition of the Type A356 alloy is (wt%): Si 7.00, Mg 0.35, Fe 0.07, Ti 0.061, Al balance. This composition is suitable to optimise composite characteristics because the high Si content improves the castability, and lowers the thermal expansion coefficient and solidification shrinkage. The Mg addition allows heat treatments to be carried out and possibly behaves as melting agent.

The properties of both materials were studied in as-received (F temper) and heat-treated (T6 temper) conditions. The heat treatment consisted of 8 hours at 535°C plus 10 hours at 540°C, water quenching and artificial ageing at 160°C for a period of 8 hours. The composite has also been remelted by conventional foundry techniques (sand and permanent mold cast) and successively heat-treated (T6 temper) in order to study the influence of the cooling rate on the microstructure.

The results of the study can be summarized as follows:
The cast metal matrix composite (A356 + 20% SiC) investigated by tensile tests at room temperature shows a higher yield strength, a comparable ultimate tensile strength, a lower elongation to fracture and a more elevated Young modulus as compared to the matrix metal (A356 Al alloy) (figure below).

Below: *Tensile characteristics (ef: stipples, YS: stippled; UTS: white, E: black) at room temperature of the unreinforced A356 alloy and A356 + 20% SiC composite in foundry ingot fabrication conditions and two tempers (F and T6)*

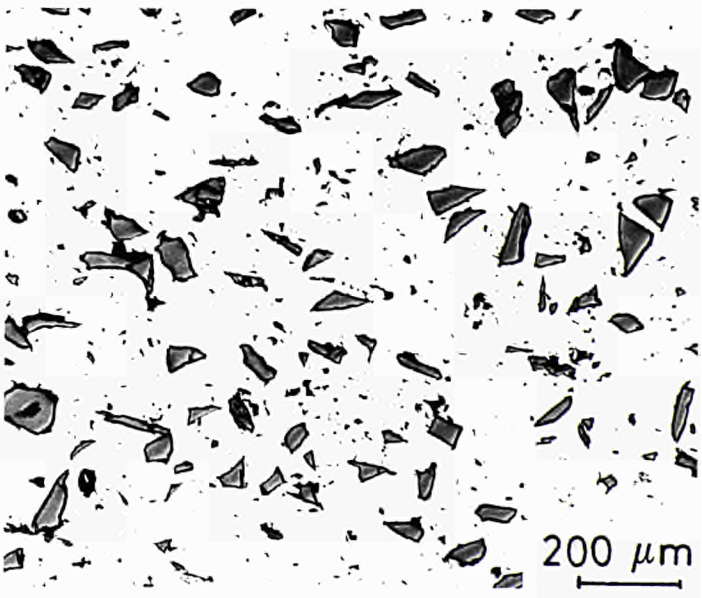




Above: *Fracture surface profile of the A356 + 20% SiC composite (F temper) broken at room temperature ($\varepsilon = 1x10^{-4}s^{-1}$).*

Below: *Microstructure of the Al + 20% SiC composite (as-deposited by plasma-spray)*

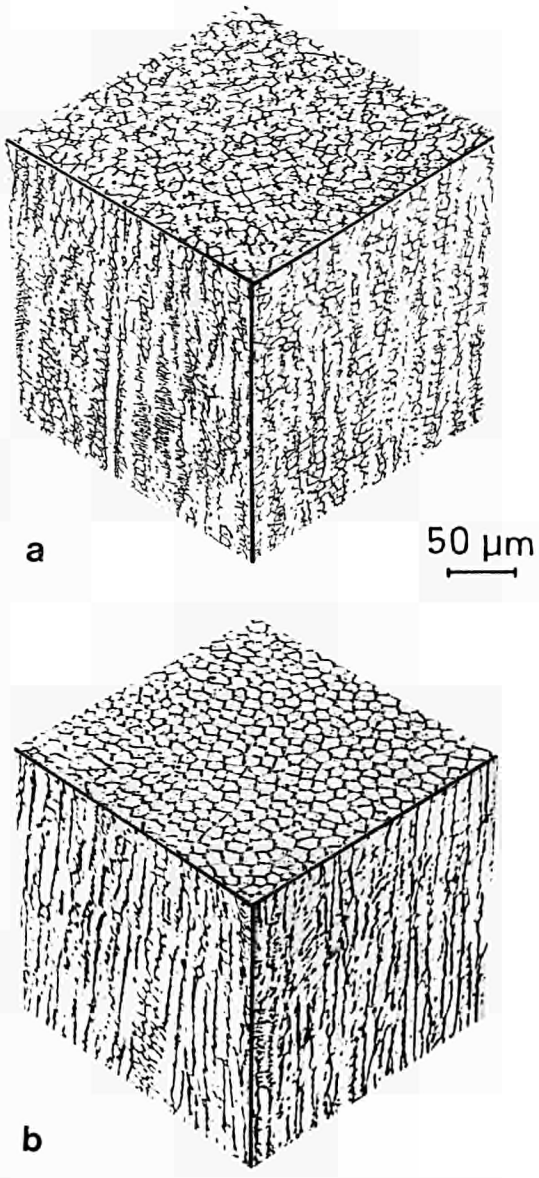
The fracture is strongly affected by a damage process which occurs during the plastic deformation by debonding of SiC particles-Al matrix interface with the formation of cracks which propagate by a linkage process along the interdendritic regions where the SiC are clustered (figure above).
The particulate composite morphology of the foundry ingot is not lost after remelting by conventional foundry operations (sand cast or permanent mold cast).
Lastly, preliminary experience with a plasma-spray device in order to produce an "Ispra" material led to a microstructure (figure below) characterized by a low segration index. The evaluation of this material is in progress.



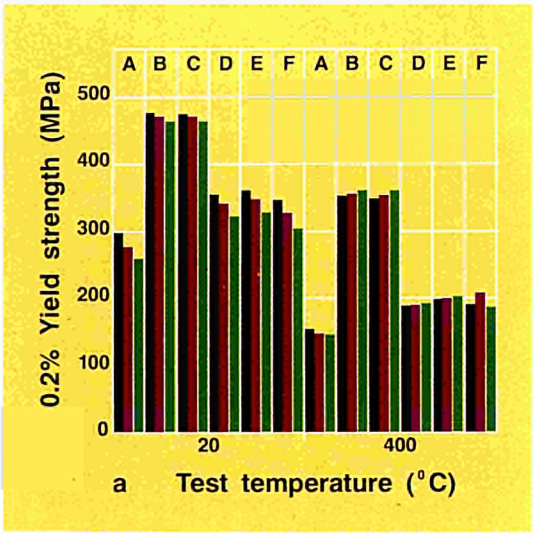
B) Metallurgical and mechanical characterization of a submerged arc welded heavy section joint in a 316 Type stainless steel

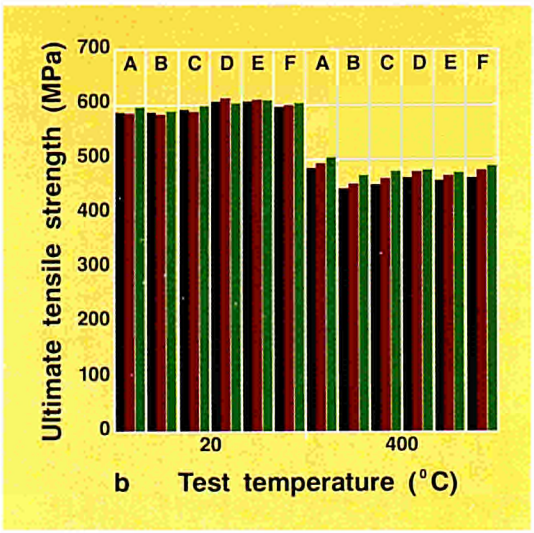
Type 316 austenitic stainless steel appears to be the likely candidate material for systems such as fast breeder nuclear power plants and thermonuclear fusion reactors.
The use of this alloy in a number of nuclear components requires that it must be joined by various welding procedures.
Generally, when large thick sections (>25 mm) are concerned, the submerged arc (SA) welding process is used because of its high deposition rate, high thermal efficiency and metal recovery.
However, attention has to be paid to avoid damaging the material properties (reduction in toughness and resistance to corrosion) because of the high heat input which characterizes this process.
In particular, when the number of passes is large, significant variations occur from the surface to the centre of the weld because of the local differences in the complex thermal and mechanical histories occurring during the welding process.
The effect of the above variability has been assessed on tensile and creep behaviour of the weld-deposited metal itself and also of the weld as a whole and has been revealed to be significant.
However, the number of experiments on the mechanical properties of thick section welded joints, and their evaluation, are quite limited.
It was useful for design and safety stress considerations of Type 316 nuclear components, to acquire more basic data on the material constituting the welds.

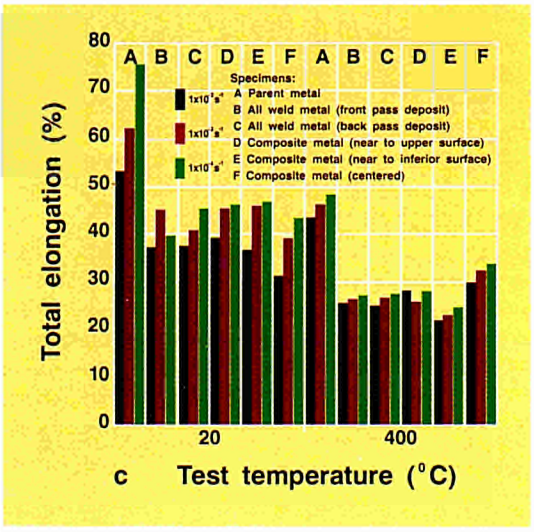
Accordingly, the authors have investigated a thick (50 mm) Type 316 SA welded joint prepared with a multipass procedure and using two different types of 316L filler metals.
An important material variability from the surface to the centre of the weld-deposited metal has been found, constituted by differences in dislocation density, columnar grain size, ferrite content, distribution and morphology (Figures above) as well as chemistry. This situation leads to a tensile properties variability through the thickness of the weld metal with the centre of the weld characterized by higher strength and lower ductility than the weld metal near to the surface.



Above: *Three dimensional composite micrographs showing different ferrite morphology in Type 316 SA weld-deposit: (a) vermicular δ-ferrite (dark etching) located at the cell-axes (ferrite-austenitic solidification mode); (b) vermicular δ-ferrite (dark etching) distributed at the cell-boundaries (austenitic-ferritic solidification mode).*
The δ-ferrite alignment is the columnar growth direction which is also the heat direction







More exactly, the results summarized in the figures left show:

(1) the weld-deposited metal shows a higher YS but a comparable UTS with respect to the parent metal;

(2) the transverse to weld composite metal, including the parent metal, HAZ and weld deposited metal, has an YS slightly higher than the parent metal but significantly lower than the weld-deposited metal, while the UTS is comparable with respect to the parent metal and the weld-deposited metal;

(3) the material variability in the weld-deposited metal, although significant, is not too large to influence in a negative manner the tensile properties of the welded joint as a whole;

(4) the tensile behaviour of the transverse to weld composite specimens is characterized by a supporting restraint effect from the weld-deposited metal to the HAZ and parent metal because of the different deformation resistance between the fusion zone (higher YS) and the parent metal (lower YS).

Left: *Comparative tensile properties: (a) YS; (b) UTS and (c) Ef related to various location specimens Type 316 SA weldment as a function of test temperatures (20 and 400°C)*

Chemical Sensors

Introduction and Objectives

The development of solid state chemical sensors is of great importance for their application in industrial processes as well as for environmental control. Solid state sensors, which rely on surface variation of properties of solids (chemical potential surface conductivity etc.) when exposed to gaseous atmospheres, have the advantages of relatively low cost, small size, long life continuous and fast response (easily processed and converted to components movements, thus allowing automatic control of valves or other mechanical systems). A well known disadvantage of these sensors results from their lack of selectivity, which limits their application in complex gaseous atmospheres, where the contemporary presence of molecules presenting similar surface reactivity may introduce uncertainty in their response.

The aims of the present project are the following:

i) to improve and calibrate existing chemical sensors, also in the presence of complex atmospheres,
ii) to develop new types of solid state sensors for gaseous molecules (e.g. NO_x) of industrial or environmental relevance, the detection of which is at present impossible or possible only with less accurate or costlier methods.

In both aims, close cooperation with other Laboratories as well as with industry (sensors' fabricants and sensors' users) is of great importance. The project foresees the formation of an industrial project club around associated laboratories, each providing special expertise to the fulfillment of these aims.

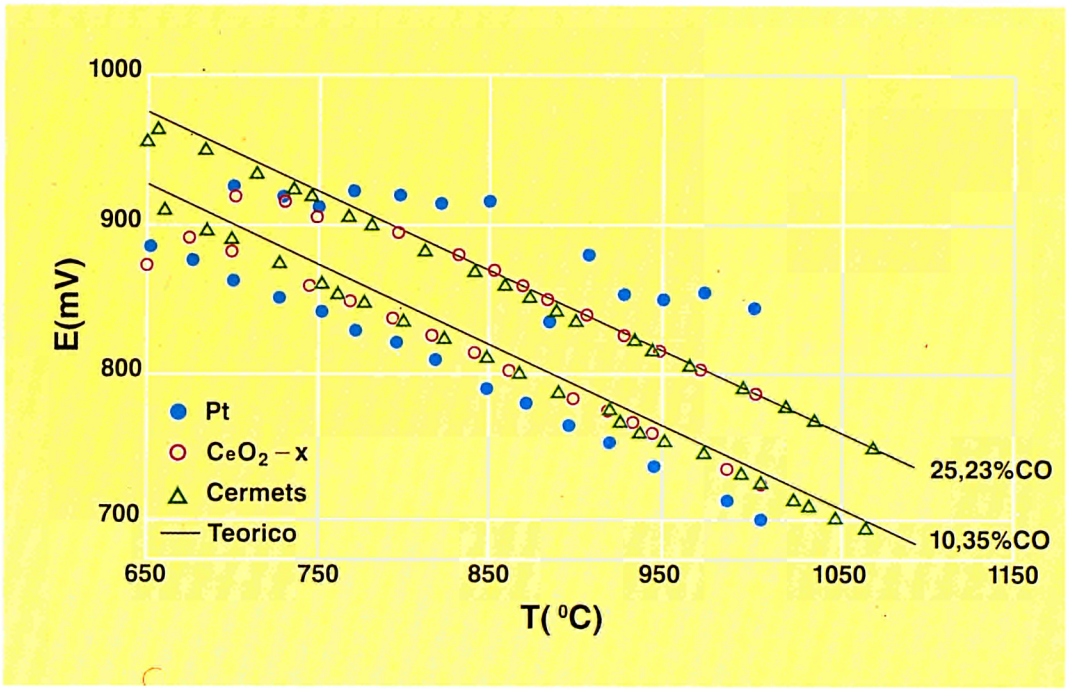
The "Cermet" Oxygen sensor

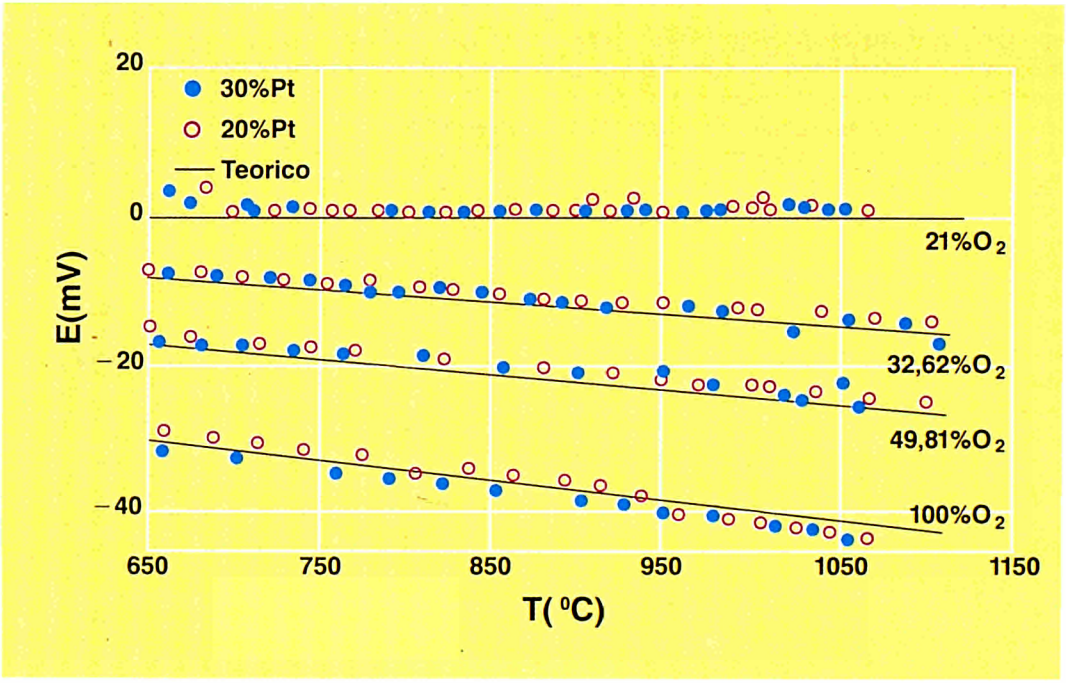
A first result in the chemical sensors' project is the development of an electrochemical oxygen sensor, employing a "CeO_{2-x}/Pt Cermet" electrode as the sensing unit. This sensor is able to measure the oxygen partial pressure in a wide range of oxygen compositions in highly reducing as well as highly oxidizing complex atmospheres (pO_2 10^{-12} - 10^5Pa). The sensor developed competes with the well known porous Pt sensor, but has a much longer life in severe conditions of utilization. The response of the porous Pt electrode, for instance when submitted to very high concentrations of CO (highly reducing atmospheres), is considerably changed and becomes unreliable, and is frequently destroyed.

In Figure on page 58 above the emf response of CeO_{2-x} , CeO_{2-x}/Pt cermet, and Pt electrodes are compared for high CO content CO/CO_2 atmospheres and N_2/O_2 atmospheres respectively, and also with the theoretical straight line calculated by the Nernst equation. It can be observed that the CeO_{2-x} and CeO_{2-x}/Pt cermet electrodes' experimental points are very close to the theoretical plot for highly reducing atmospheres, whereas (for 25% CO) the Pt electrode data are clearly unstable. For oxidizing atmospheres (Figure on page 58 below) the experimental points of the three electrodes compare well with the theoretical plot.

Page 58 above: *Electromotive force (e.m.f.= E) measurements vs. T when measuring very low oxygen partial pressures in CO/CO_2 mixtures.*
The results show that the best fit with the theoretically calculated values from the CO/CO_2 thermodynamic equilibrium data (straight line in the figure) are obtained when using a Pt-containing Cermet electrode (Δ) in comparison with: a conventional porous Pt electrode (•) and a CeO_{2-x} electrode (o).
Futhermore, no effect appears of Pt-poisoning as shown for the porous Pt results due to the high dispersion of Pt in the cermet.

Page 58 below: *Electromotive force (e.m.f.= E) measurements vs. T when measuring high oxygen partial pressures in N_2/O_2 mixtures with a Cermet electrode having different Pt compositions (30%: •; 20%: o; straight line: theoretical values). The figure shows good agreement with the thermodynamic value even when varying the Pt dispersion in the cermet.*


1000
900
800
700
E(mV)
650
750
850
950
1050
1150
T(°C)
Pt
$CeO_2 - x$
Cermets
Teorico
25,23%CO
10,35%CO


20
0
−20
−40
E(mV)
650
750
850
950
1050
1150
T(°C)
30%Pt
20%Pt
Teorico
21%O_2
32,62%O_2
49,81%O_2
100%O_2

An earlier oxygen sensor for reducing atmospheres using a CeO_{2-x} sensing electrode has been developed and licensed by us (and is now marketed). The lifetime of this electrode has been tested in realistic industrial conditions (i.e., in a furnace for steel carburization) for more than one year. No appreciable deviation of the response has been so far detected. In the CeO_{2-x}/Pt cermet electrode, the Pt content is very low and dispersed and a similar behaviour can thus be expected with a long life.

Its stability and ability to work in a very wide range of oxygen partial pressures makes the CeO_{2-x} cermet electrode a good sensor for the case of abrupt changes of oxygen pressure (due to accidents, maintenance periods etc.).

The cermet oxygen sensor electrode concept has been submitted for licensing by the European Communities, and already licensed in Luxembourg.

The NO_x sensor

For the NO_x sensor two research lines have been identified and initiated.
1) The potentiometric sensor comprises a solid electrolytic galvanic cell whose electromotive force can be readily related to the logarithm of the nitrogen oxide partial pressure.
It is necessary that the solid electrolyte has a transfer number one for silver ions, in order to avoid polarization effects at the electrode/electrolyte interfaces. Silver nitrate has been selected because of its electrical conductivity and its stability.

The conductometric sensor is a layer of metal oxide deposite on to an inert ceramic support and its response to NO_x is seen in terms of electrical resistivity variations. Preliminary studies are under way for this type of sensor.

Cooperations with industry

With the intention of establishing a pool of necessary competences for the improvement of existing sensors and for the development of new Sensors, some important associations were established in 1989.

The first association is MILANO RICERCHE which is directed towards industrial technological needs. The considerable expertise already available at the State University of Milan will be fully exploited.

A second association is with the Centro Superior de Investigaciones Cientificas, Spain, where internationally recognized competence in the field also exists.

The Institute for Advanced Materials of the Joint Research Centre will contribute essentially in the field of accurate characterization of the surface analysis techniques.
A first meeting with sensors' fabricants and users is envisaged in the first half of 1990 for the promotion of an industrial project club, aiming firstly, at the accurate calibration and characterization of existing sensors for use under complex and severe atmospheres.

Modulation of SURFACE PROPERTIES

Surface Treatments for Improved Performance

Surface Modification Centre

Substantial effort was devoted to the final installation of the different parts of the equipment.
The 5 KW CO_2 Laser has been installed and tested. The test results were satisfactory. It is now possible to apply the power of the laser in beams of different geometries on rather large surfaces by the motion of a x-y table. The computer connected to the table allows coverage of the specimen surfaces in very different ways. Experiments have been made for surface melting on bare metals and on plasma coated metals. Problems such as the surface tension of the melts and wetting between melt and substrate have been recognised as important.

The electron beam gun, derived from an industrial welding machine is not designed for E.B. scanning. However, a simple scanning programme was ordered and applied to our apparatus without changing the existing hardware. Three scanning programmes have been provided (a sawtooth, triangular and spiral). Preliminary experiments have shown that surface remelting and heat treating can be obtained on small surfaces (3 cm x 3 cm) with "low speed scanning" : 1 m/sec. This corresponds to frequencies of about 16 Hertz. Several remelting trials were performed on stainless steel specimens coated with triballoy.
Metallographic examinations after remelting showed clearly that wetting problems of triballoy on stainless steel were the main problem in this procedure.

The ion implanter has been tested with gaseous ions and satisfies the specifications. The first experiments with the implantation of metal ions have shown that the procedure via the decomposition of chloride is very cumbersom and requires extended cleaning of the ion source and the first section of the accelerator.

It has therefore been decided to purchase a sputter ion source which facilitates the production of metallic ions in a much cleaner way.
A number of implantations on various materials offered the possibility to calibrate the temperature measuring devices and to develop first procedures for controlling the surface temperature of the implanted specimens.

The following implantations have been performed:
a) He and D in stainless steel AISI 316,
b) Argon with different densities on sputter coated specimens,
c) Nitrogen ions in ceramics in order to test the heating of the surface,
d) nitrogen in austenitic steel samples,
e) nitrogen in aluminium samples,
f) nitrogen in titanium alloys.

The Auger scanning microscope has been installed and tested. While its performance as scanning microscope satisfies the specifications, the performance for depth profiling of surface impurities has still to be improved. In addition, software and image recorder need improvements. The present software does not allow the data treatment in real time, transfer to another computer is necessary. Colour images cannot be recorded. A new software will be supplied by Riber. The recorder system will be purchased in the next months.

The definition and preparation of the connected testing laboratories is proceeding and it is expected that in 1990 all laboratories will be working after the delivery of the remaining equipment.

High Temperature Corrosion Laboratory

The increasing need for materials with good performance in aggressive high temperature gaseous atmospheres may be partly fulfilled by surface coating of selected alloys.
A laboratory facility has been set up in Ispra in order to test the corrosion resistance of coated samples. The experimental apparatus consists schematically of a closed loop with three furnaces in parallel which may operate at three different temperatures.
The composition of the gas circulating in the loop is fixed at the beginning of each experiment. It is possible to add up to six corrosive species to a carrier gas. A mass spectrometer controls the mixing of the different gases in order to obtain the predetermined composition.
During the experiment, the mass spectrometer controls the opening of solenoid and piezoelectric valves for the addition of the corrosive species which are consumed by the corrosion reaction.
In this way it is possible to maintain a constant level of corrosive impurities in the loop.

The maximum operating temperature is 1500°C for one furnace and 900°C for the others.

The mass spectrometer was delivered at the end of 1989; in the meantime all the accessories have been ordered and the installation of the loop has been planned. It is not expected that the installation will be operating before April '90.

The research programme has already been established in consultation with ENEL (Italy). The work will be focussed on materials problems in high temperature gas turbines. The corrosion tests will be carried out mainly on coatings which should resist oxidation and sulphidation at temperatures between 800 and 1100°C. The coatings will be prepared in Ispra, initially using air and vacuum plasma spray (APS and VPS).

Low Temperature Corrosion Laboratory

1. *Corrosion in high temperature water and aqueous solutions.*

For corrosion testing in high temperature water and aqueous solutions, five autoclaves of 4000 ml volume, for pressures up to 50 bar and temperatures of 200°C were ordered. Two are intended for use with reducing solutions and are therefore equipped with PTFE lining on all internal surfaces. The remaining three are made from alloyed steel DIN 1.4571 (AISI 316 Ti), which is suitable for a variety of oxidizing fluids. Delivery is foreseen between January and April 1990 and the procedure for the commissioning by ISPESL (Italian authority for safety control of pressure vessels) has been established.

2. *Electrochemical corrosion testing.*

Equipment for electrochemical corrosion tests using DC current and potential measurement techniques (ASTM G 5 and G 15, Tafel plots, etc.) has been ordered. For a modest additional cost accessories can be provided which also facilitate AC impedance measurements.
This is particularly useful for the study of passive films and protective coatings. In addition to services for the surface laboratory, it is intended to investigate the corrosion behaviour of amorphous metal surfaces which will be prepared in the surface laboratory.

3. *Upgrading of equipment for testing corrosion in atmospheres containing sulphur dioxide.*

As a contribution to the Solar Energy programme experience was gained with accelerated corrosion tests in condensating water atmospheres containing sulphur dioxide (DIN 50 018). This rather severe test procedure was applied because of a general lack of less agressive standardized test procedures, more appropriate to atmospheric conditions. Conclusions drawn from recent experiments show that the standard test procedures currently in use, cannot easily be modified by decreasing the prescribed sulphur dioxide quantities.
In an attempt to resolve the difficulty a system has been designed for upgrading the test chambers by means of an automatic control of the sulphur dioxide concentrations using a gas analyser and an electron regulator. The system can be applied on a spray test such as salt mist corrosion (DIN 50 021 and others) or to the conden sating water atmosphere test (DIN 50 018).

Microstructural Studies on Al-Cu Surface Alloy

A microstructural investigation of a single pass electron beam alloying of plasma sprayed Al coatings on a Cu base was carried out by scanning electron microscopy and energy dispersive X-ray microanalysis. Backscattered electron imaging (atomic number contrast) and X-ray elemental distribution mapping were used to assess the degree of mixing achieved and to determine the phases present. The major phases identified were a copper rich Al Cu f.c.c. solid solution and the intermetallic -phase (Al_4Cu_9). Studies of vacuum deposited thin films of titanium nitride and boron nitride on various substrates were also initiated. The resolution of conventional scanning electron microscopes, using tungsten or lanthanum hexaboride filaments, proves inadequate for this purpose.

A high resolution microscope with a field emission electron source is required. Both titanium nitride and boron nitride films have been successfully examined with this type of instrument, observations being made in the direction normal to the surface and in cross-section. Titanium nitride films exhibited a columnar grain structure,of lateral grain diameter 30-40 nm, while the structure of boron nitride films appeared to be completely amorphous.

Permeation Barriers for Deuterium and Tritium

It has been reported in literature that by implanting helium into nickel targets at energy levels in the region of 25 KeV and up to 10^{17} He^+/cm^2, diffusion traps for deuterium would be obtained. These traps consist of defects, clusters and bubbles of helium and are reported to decrease significantly the diffusion of deuterium in nickel.

In order to find out the efficiency of a helium barrier in stainless steel (first wall and blanket material), the following implantations are required:

i) different doses of He^+, at different energies (KeV) and low temperature,
ii) the above targets should be implanted with different doses of D^+ at low energies and elevated temperatures to allow for deuterium diffusion,
iii) in the same way as described in ii) targets are implanted, which contain no helium.

The samples were ground, lapped, and polished with diamond paste to obtain a surface roughness of about 0.1 µm. They were also subjected to ultrasonic cleaning and were finally degreased and dried in air. Under these condtions no oxide layer on the treated surface could be detected by scanning electron microscopy. On the other hand, the Auger Electron Spectrometry revealed an oxygen concentration profile which corresponds to an oxygen layer of about 100 Å.

The implantation experiments have been carried out with an implanter of the GSI (Gesellschaft für Schwerionenforschung, Darmstadt FRG, courtesy by Dr. B. Wolf). Due to technical difficulties in cooling the target, the He^+-ions have been implanted at a
temperature of about 200°C and the D^+-ions (40 KeV only) at about 100°C for 20 minutes. The results of the helium concentration profiles in targets 1 and 2 are not yet available, because the SIMS installation at the Physics Department, Padua University, not currently available.

The following implantations have been performed:

a) targets 1 and 2
2.10^{17} He^+/cm^2, 200 KeV, about 200°C. Then an additional amount of 2.10^{17} He^+/cm^2 at 100 KeV was implanted. Assuming a reflection coefficient of 0.5, the net quantity implanted would be about 10^{17} He^+/cm^2 for each of the two energy levels. This corresponds approximately to the saturation concentration
$Ns = UR_m/S$, where U is the density of the target material in atoms/cm^3 and S the sputtering coefficient.

b) target 3 and 4
The helium implantation was the same as in a) above at about 200°C.
Additionally 6.10^{16} D^+/cm^2 with 40 KeV and at about 100°C were implanted during a period of 20 minutes to favour the diffusion of deuterium into the bulk.

c) target 5 and 6
These were implanted with 6.10^{16} D^+/cm^2 at the same conditions (about 100°C) as described in b).

It was expected that the deuterium concentration profiles would be different in shape and extension, because the diffusion barrier in the targets 3 and 4 would prevent the deuterium transport to the bulk, whereas in the targets 5 and 6 the deuterium could freely diffuse.

After the implantation,the ERD-spectra (Elastic Recoil Determination) have been determined in the laboratory of the department of physics, Padua University. The conclusions drawn from the shape of the spectra were completely opposite to those expected.

The deuterium concentration distribution in the samples 3 and 4 (which contain He and D_2) is of the same shape and intensity, which means that the implantation procedure was reproducible. However, the deuterium is evenly distributed between the surface and a depth of about 1000 Å, instead of showing the "typical" implantation profile (a negative concentration gradient between 1000 Å and surface). Possibly back diffusion to the surface or slow desorption have "smoothed" the initial part of the profile.

The deuterium distribution in the samples 5 and 6 (containing only deuterium) is again very similar in both samples, but in place of a deuterium concentration profile extending deep into the bulk, as was

expected, the ERD-spectra show a deuterium peak near the surface, as if there were a segregation of deuterium. A comparison of the spectra of the targets 3 (He, D_2, 200^{0}C) and 5 (D2, 100^{0}C) shows that the deuterium peak of the sample 5 is about ten times higher than that of sample 3.
Some explanation of this behaviour may be revealed when the helium distribution in samples 1 and 2 is analysed.

Ion Beam Mixing and Characterisation of a Copper/Silver Three Layer System

As a preparatory study for the foundry project an ion beam mixing experiment on a Cu-Ag sandwich was performed. The aim was to gain familiarity with the problems arising in ion implantation technique and to test several characterization methods such as SEM, RBS, AES and X-ray spectroscopy. It could be shown that the result of the mixing process is a metastable solid Ag-Cu solution, whereas in thermodynamic equilibrium the Ag-Cu system shows a large miscibility gap. The results obtained are in agreement with earlier experiments.

Characterisation of Sulphidised Steel Surfaces using Synchrotron Radiation

Glancing angle X-ray diffraction and soft X-ray absorption spectroscopy have been combined to characterise stainless steel surfaces corroded at 973 K in sulphidising environments. The data reveals the surface to consist of large multi-phase crystallites which are a macroscopic mix of three sulphide structures; Spinel (M_3S_4), Triolite (MS) and Pentlandite (M_9S_8) where M = Cr, Fe or Ni. The local environment around S is highly asymmetric and the structure appears to have high content of cation vacancies.

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 in 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 1989 a major effort has been devoted to the improvement of the quality of the databank functions up to the level set by modern commercial products.

The most remarkable innovation was brought about by the implementation of the user-friendly PC-based query interface. 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.

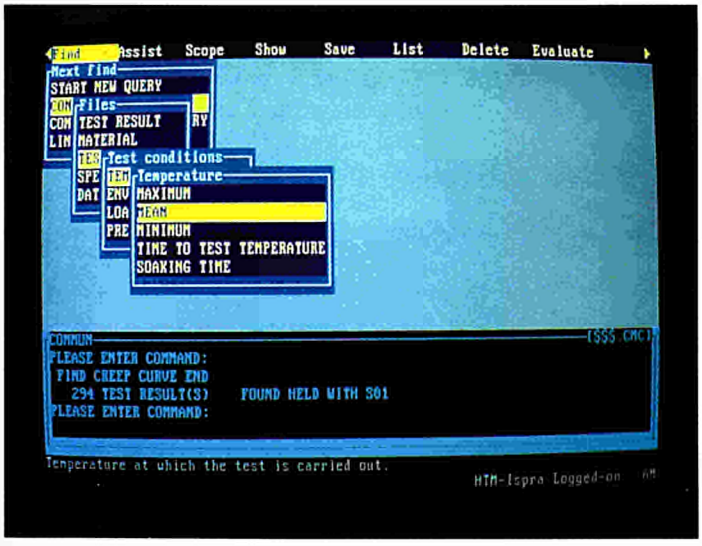
Among the modules to have been completely revised, figures the data input PC-program which received extra functionality like pulldown menus, thesauri and some facilities for validation.

New routines have also been added to the Evaluation Program Library to which datasets retrieved from the HTM-DB can be submitted for further elaboration. This library now contains more than 70 Fortran programs which can be selected from a menu system.

Some of the routines are simple spline or linear regression programs, whereas other modules of the Evaluation Program Library allow the calculation of constitutive equations with user guidance through the different program steps. These calculations allow the user to calculate actual material parameters for design purposes.
Figure on page 70 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.

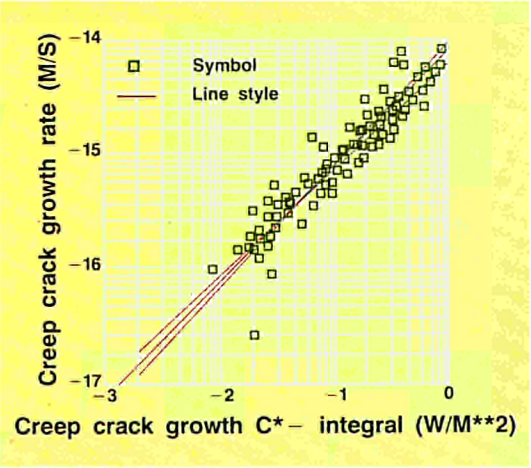
The extensive testing of the Evaluation Library has lead to a de facto validation of the data through the systematic correction of errors whenever they occurred. With respect to new data, major input activities have been concentrated on the support of joint projects like VAMAS, COST 501 WP1 and WP5A,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

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



The described modifications 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. 1989 was indeed characterised by a continued effort to promote the use of the HTM-DB and to exploit the acquired expertise. A two day training course on the online use of the HTM-DB was attended by 18 participants, mostly COST members. The staff furthermore contributed to six demonstration workshops of the Demonstrator Programme organised by DG XIII and to its Concluding Workshop in Petten on 6-8th December.
Investigating the possibilities for a transfer of technology, the HTM-DB finally promoted an enquiry on the needs for a databank on properties of Ceramic Materials. This enquiry arose considerable interest and its results are intended to be evaluated by a workshop in the course of 1990.

In conclusion it can be stated that the High Temperature Materials Databank system has now reached a commercially acceptable level which merits the major public relations and data input effort planned for the coming years.



Above: *Creep crack growth rate VS. C*-integral, alloy 800H at 550 ^{0}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 1989, efforts have been focussed on the following activities:
European Colloquium on the High Temperature Corrosion of Technical Ceramics.
The meeting was held in Petten in June.

The objectives of this colloquium were:
- To create an international forum for the review, presentation and discussion or recent progress in understanding the high temperature corrosion properties of technical ceramics;
- To bring together expert scientists and engineers with interests in the topic and to encourage and promote mutual exchange of knowledge and experience;
- To aid the identification of research topics of vital importance to industry for improving the market penetration of these new materials.

The colloquium was attended by 79 scientists and engineers from 14 different countries, representing a cross-section of universities, research institutes and industries, both producers and users of technical ceramics. In addition to 8 invites lectures given by recognised experts in fields as wide as the fundamental science of corrosion of ceramics to the applications of ceramics to high technologies, there were 13 contributed papers or posters.
Discussions after presentations created a crucible for the exchange of ideas at the crossroads of fundamental science, engineering and practical applications. Proceedings will be published.
In parallel to the scientific sessions, a technical exhibition was held with presentations from 6 organizations. The colloquium was co-sponsored by 15 industrial companies.

The study: "Research and Development of High Temperature Materials for Industry" was completed during the reporting period.
The Study reviews materials requirements in high temperature technologies from aspects of:
- Potential and performance limits of new and conventional high temperature materials;
- Operational constraints imposed by high temperature technologies and identification of materials shortfalls.

The individual materials and technology topics covered by the study are represented by original contributions from specialist authors who have been selected on the basis of their association with the particular field of materials or engineering.

The contents of the study are divided into three main groups:
- Materials Potential; Materials Production and Processing-to provide a developers outlook.
- Materials Constraints in the High Temperature Industrial Technologies: Optimization of components-giving the viewpoint of materials users, and of plant and component designers.
- Research Trends in Industrial Development; Constraints-to focus upon future orientations and research needs.

The study identifies the priorities for research and development in the short term future (approximately ten years) of structural materials operating in major high temperature technologies.

"The Economic Effect of Fracture in Europe" is the subject of a study which was contracted to the European Group of Fracture (EGF). An assessment of the European situation could provide criteria for demonstrable economic benefits resulting from efforts into the development and improvement of codes of practice, specification and standards and fracture related research. A similar study was undertaken by NBS and Battelle Columbus in 1978 in the U.S.A. and showed that:
- large costs are incurred by fracture, i.e. 4 % of the gross national product;
- a savings potential through fracture related research of the order of 1 % of the gross national product could be expected.

The present study aims to assess the total costs of fracture in Europe, to identify the future potential for reduction in costs of fracture, achievable by conducting R & D.
Priorities for R & D to advance the understanding of materials fracture and fracture control technology through codes of practice, specifications and standards will be identified.

COST Secretariat

In addition to its direct involvement in the investigations, the Institute also provides the Secretariat for two of the larger worksharing programmes which are organised under the auspices of COST.

These programmes are COST 501; Advanced Materials for Power Engineering and COST 505, Materials for Steam Turbines. In addition to conventional Secretariat duties there is a significant involvement at a technical level by interacting with coordinators, acting as an advisory centre etc.

In COST 501, 13 European countries plus the Institute for Advanced Materials of the JRC are contributing more than 500 my effort which has been valued at about 50 Mecu over a three year period for Round II. Most participants receive funding from their national sources, but 15% of them take part with only their own resources. Almost two thirds of the participants are in the power engineering industry, the remainder representing research institutes and universities. The industrialists have played a strong role in setting the targets to be met in each of the 10 integrated work packages and in managing them. The experimental activities in these work packages started in 1989 and approval was given by the COST Committee of Senior Officials for COST 501 to run for a further three year period, until December 1992. During 1989 the Secretariat also handled considerable activity associated with the completion of Round I activities and the production of evaluation Study reports for that period. Achievements of Round I which are already being tried in industry include materials for gas turbine discs, cost effective diesel engine valves, and gas turbine vanes.

The year 1989 saw the completion of COST 505 in which 9 European countries plus the Institute for Advanced Materials of the JRC had participated.

About 100 my effort was contributed to the programme at a value of about 10 Mecu, over a five year period. To enable intergroup discussions to take place, the Secretariat organised a 2 day Status Seminar at Petten in March 1989 which was attended by 80 expert participants. Since then a number of Study Reports have been processed by the Secretariat in order to evaluate technically the results of various coordination groups. Future investigative work in this sphere has been channelled into COST 501-Round II.

2. Contribution to the Specific Programme:

REACTOR SAFETY

Project for the Inspection of Steel Components (PISC)

Introduction

The safety of nuclear power plants depends on the mechanical integrity of the materials, particularly the welds, used in their structure and on the ability to detect any degradation at an early stage or prevent it entirely. Non-destructive methods have been developed for finding surface and internal flaws that might develop and for determining their size. Techniques using sound waves of ultrasonic frequency are among the most widely applied, particularly for the important vessels and piping of the reactor plant. These components present very challenging inspection problems because of their size, complex shapes, combination of materials and difficulties of access after construction.

Various inspection procedures using ultrasonic techniques have evolved to deal with particular circumstances but, in some cases, uncertainties have remained about their capability and reliability. Such problems were first addressed in several national programmes of round robin inspection trials. It was recognized that such trials could be more effective if they involved the much larger number of teams and additional procedures available if a number of countries participated.

Beginning in 1974, a series of major co-operative programmes known collectively as PISC was carried out: PISC I (1974-1980), PISC II (1981-1987), PISC III (1988-1991).

Programme Results

PISC I results showed shortcomings of some usual industrial In-Service Inspection (ISI) procedures; PISC I was in fact analyzing the existing ASME 1974/1977 procedure (1).

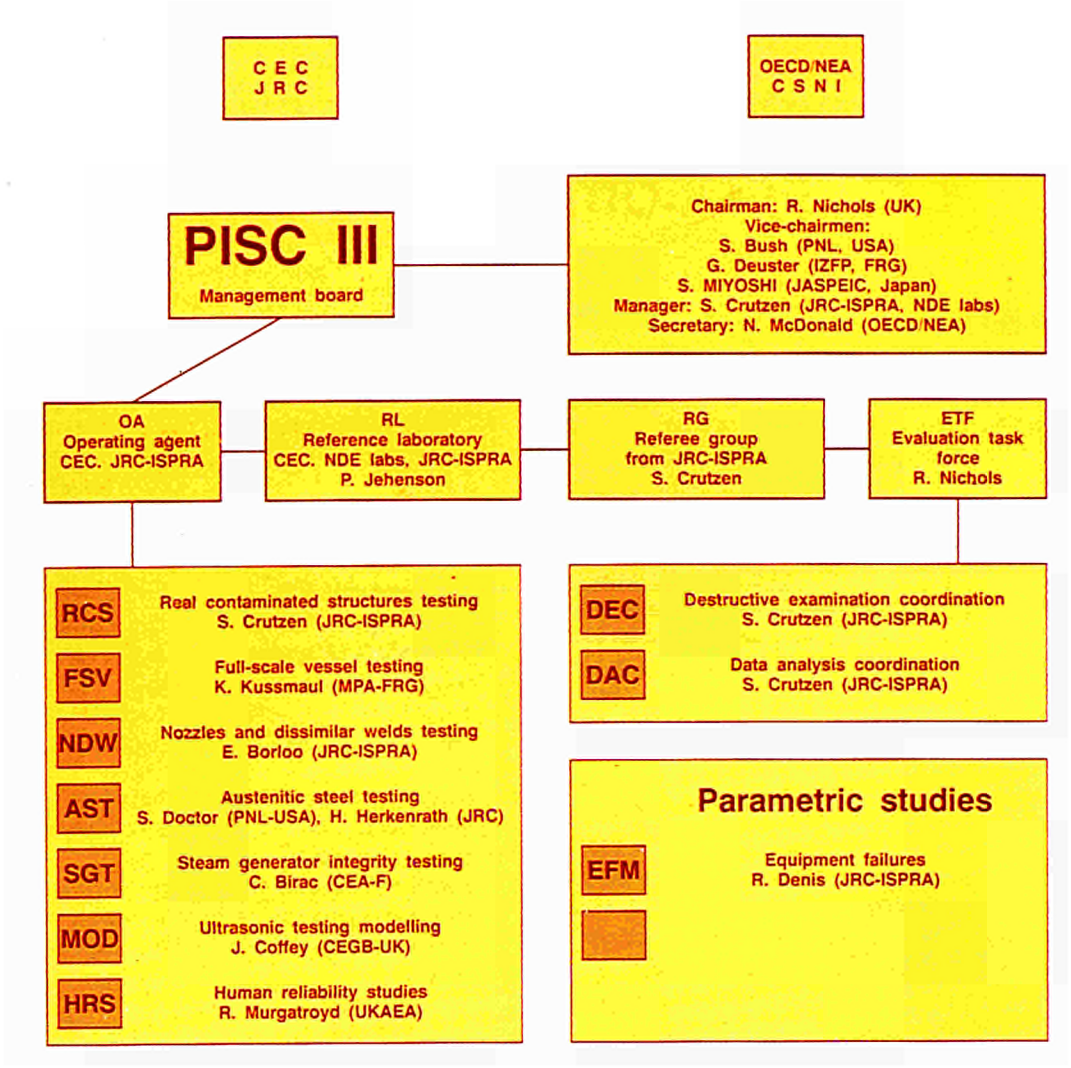
PISC II RRT results (2) (as well as some trials of PISC I) showed that:

a) improvement of the usual industrial NDE attitude was possible; consequentely, some changes of the ASME procedure were proposed:
   - o 20% DAC instead of 50% DAC:
   - o use of techniques adapted to the defects to be detected: e.g. 70 deg SEL probes;

b) several ISI procedures exist that meet the requirements (such as the ones established by the UK LWR Safety Group).
   For such procedures, no change was obviously required; optimization from an economic/industrial/reliability point of view was however necessary.

PISC II parametric studies on the effect of defect characteristics (3) showed the importance of defect parameters like the type of defects (planar or volumetric Figure below), the crack tip aspect, the position in depth, the angular position, the surface roughness.

Below: *Circumferential fatigue in a BWR safe-end as examined in the JRC hot laboratories, after removal from the reactor*



These results were in fact the exact quantification of the trends shown by the PISC II RRT on welded assemblies. As a conclusion, it was understood that the blocks realized for the PISC parametric studies and containing cheap "realistic" artificial defects were good and even conservative for the performance evaluation of NDE techniques.
In 1986, starting from the PISC results, the ASME Code for pressure vessels and boilers, Section XI, has been discussed in the USA and modified. Presently, the ASME Section XI Committee is going beyond the modifications proposed in 1986 and the new appendix VIII on performance demonstration clearly starts from the PISC results (4).

PISC III Programme Status

PISC III, the third phase of the PISC series insists on the capability demonstration with assemblies containing realistic defects (Tab. above and right) (4).

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
Duration	1991
Secretariat	NEA/OECD
Management	CEC Joint Research Centre, Ispra, Italy
Members	Belgium, Denmark, Italy, Finland, France, Germany (Fed. Rep.), Japan, Netherlands, Norway, Spain, Switzerland, United Kingdom and United States

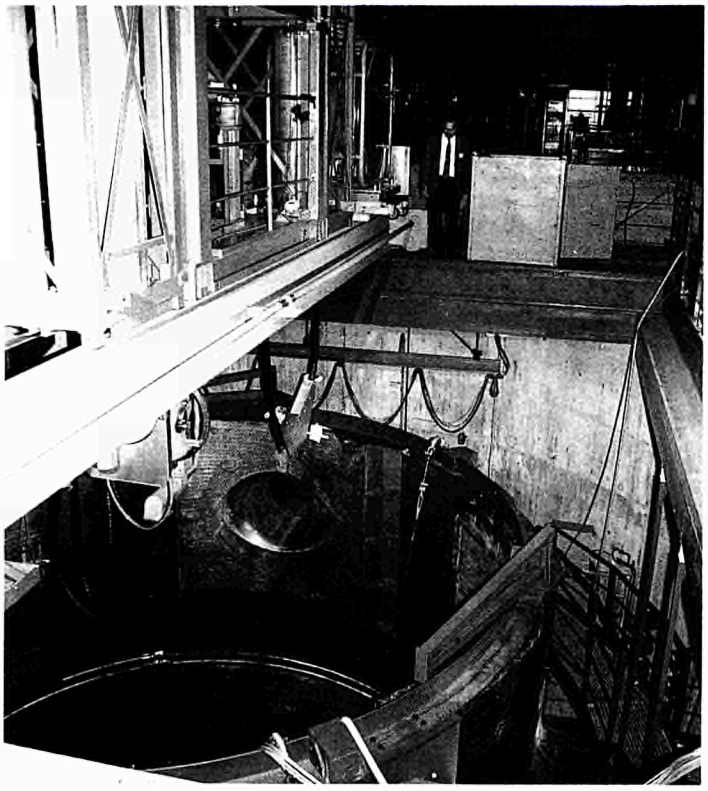
The PISC methodology is also extended to all major parts of the primary circuit of the LWR reactors. More-over, the work done on the austenitic steel testing is of real value for the inspection of LMFBR components.

Testing activities have begun in several of the PISC III actions. Substantial progress has been made in defining the final work to be undertaken in all the seven Actions and in acquiring the necessary test samples and other resources. The uncertainties that prevailed during 1986/87 concerning the level of budgetary support have mainly been resolved. Adequate resources from the CEC and the organisations of participating member countries are now assured to carry out the essential elements of all the planned actions to participate in the various round robin and other test activities.

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

Action No. 1 (Real Contaminated Structures)
seeks to collect results from specific investigations and limited round robin tests (RRT) on real service induced defects in materials and structures of the primary circuit of light water reactors (LWRs). The hot cell facilities at JRC-Ispra are fully equipped for non destructive and destructive work on a collaborative basis. Cracked austenitic steel primary circuit pipes from Switzerland have been inspected to propose a full demonstration of the whole resources available. Further samples are available from Spain, Sweden, USA and Finland (Figure on page 75).

Action No. 2 (Full Scale Vessel Tests)
validates results obtained by procedures in the PISC II exercise realistic inspection conditions (Figure above). As a start, the installation offered by Staatlich Materialprüfungsanstalt (MPA), Universität Stuttgart, Federal Republic of Germany is being used. It is made of a BWR full scale vessel plus modular full scale PWR components. Seven teams participated in 1988 and 1989 to phase 1 concerning the sizing of selected defects in order to establish the capability of sizing techniques; sizing results from the German National Programme are also included.
Eight organisations have registered their interest in the phase 2: validation of ASME type procedures

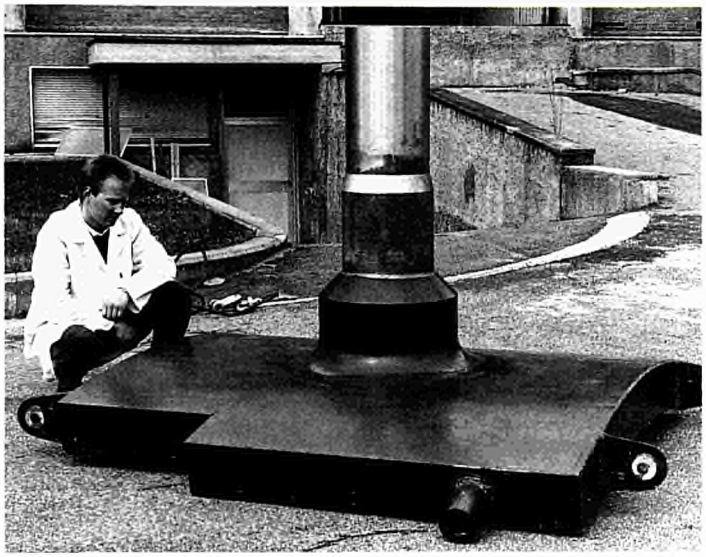


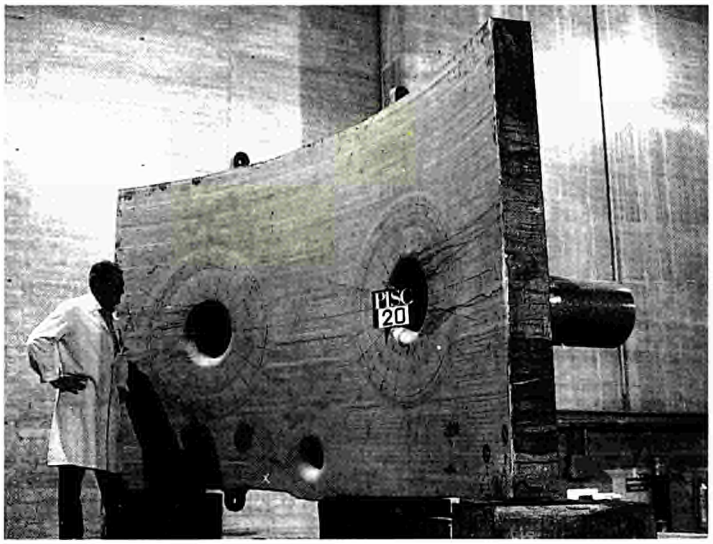
by an international JRC-Ispra team using an ISI robot offered by RWE and MAN to PISC for the period of the exercise (September 1989 to February 1990).
It is foreseen, in a third phase, that individual rams will apply their ISI procedure.

Action No. 3 (Nozzled and Dissimilar Metal Welds)
has undertaken round robin tests of safe-ends and geometries representing some of the most difficult technical aspects of in-service-inspection. A Japanese-Italian BWR assembly of nozzle plus safe end is circulating since March 1988 to twenty teams in ten countries (Figure below).

Above: *Full scale components at MPA, University of Stuttgart, used in Action 2 of PISC III: 3 PWR nozzles in a support ring on the top of a full scale BWR vessel*

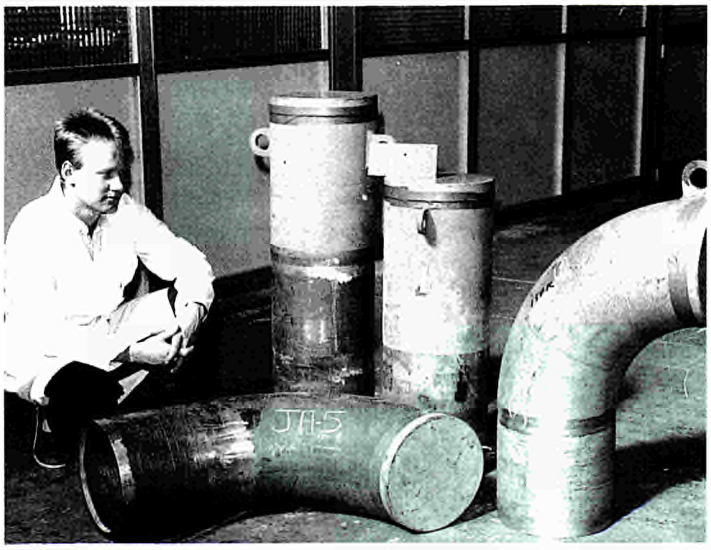
Below: *PISC III Action 3 (NDW). BWR Assembly with nozzle, safe-end and piping*





Above: *PISC III Action 3 (NDW). Assembly with three nozzles and safe-ends of the BWR type. This assembly is also a key element for any evaluation of new inspection possibilities of old plants considered for life-extension*

Below: *PISC III Action 4: Austenitic Steel Testing (AST). Wrought austenitic steel assemblies containing IGSCC's, TGSCC's, fatigue defects in the welds and sensitized material areas*



An American BWR assembly with two nozzles and safe ends and a Spanish PWR nozzle and safe end began their circulation mid 1989 (Figures above). These round robin tests are planned to continue until January 1991.
An important aspect of the RRT organization is the "Certification" of the defects in the assemblies. Such a certification, general rule in PISC, is conducted by the JRC-IAM Reference Laboratory and ensures that all assemblies proposed for testing are of value and suitable to meet the aims of the actions.

Action No. 4 (Austenitic Steel Testing)
applies the PISC II methodology to the primary circuit piping of LWRs.
Round robin tests for the capability assessment and parametric studies are considered as well as reliability evaluation of the testing procedures. Wrought pipe samples are available in the USA and from Japan; moreover, large cast samples have been ordered by JRC.
Twenty five teams have registered their intent to participate in one or more phases that should start end 1989 and will extend up to 1991 (Figure below).

Action No. 5 (Steam Generator Integrity Testing)
undertakes round robin tests both of individual tubes and tubes in realistic uncontaminated and contaminated mock-ups of steam generators containing real and artificial defects.
Capability tests and reliability tests are included. The PISC Management Board has carried out a reassessment of the technical details to be considered taking into account the results coming from the Surry steam generator studies in the USA. Preparatory work is now under way to acquire tubes and introduce and validate defects; tubes with defects have been offered from France, Japan and the United States. France is preparing the hardware necessary for circulating the tubes and the mock-ups.
The Management Board has received advice from CSNI - Principal Working Group on Primary Circuit Integrity- on the defect types and characteristics of most importance with respect to safe operation.
Twenty four teams from ten countries have registered an interest to participate in the RRT planned for 1990-1991.

Action No. 6 (Mathematical Modelling on NDE)
has the objective to validate mathematical models and perform parametric studies in order to assess the importance of defect characteristics. Sixteen organisations in eight countries have registered their intent to participate in studies to assess mathematical models of ultrasonic inspection by validating the physics of the models, verifying it with experiment and assessing the utility of the models in terms of limits of valid application, satisfactory and efficient computer performance and accuracy.
Fifteen models have been offered for investigations. An important objective is to promote the practical application of models as an aid to more effective and efficient inspection procedures and interpretation of results. The Modelling Group of PISC III has selected (April 1989) three models to be studied by the Reference Lab. for validation in 1989 (2 UK and 1 German models) as well as models to be studied in 1990 (1 UK and 2 French ones). Parametric studies are an essential source of data for verifying models; the studies commenced in PISC II have been extended in PISC III; four reports on the effect of defect characteristics on the ultrasonic signal response, one report on the effects of the cladding and two reports on the effects of the equipment characteristics are completed. This work was carried out in the United Kingdom, France, Belgium, Italy and at the JRC-Ispra.

Action No. 7 (Human Reliability Studies)
seeks to evaluate the influence of human interpretation of inspection results, equipment malfunctions and human interaction on the overall inspection procedure. Part of this work was performed in the UK and at the JRC-Ispra utilizing the PISC II data and supplementary questionnaires.
Other aspects will be undertaken within some of the round robin exercises, firstly full scale vessel inspections.

Action No. 8 Proposal (PISC III RRT's extensions for both Nuclear and Non Nuclear Plants components):
At the request of the Management Board, studies of damaged components coming from non nuclear plants are examined. The defects contained and inspection results would be of great interest to complement presently planned RRT's on nuclear components. Transfer of PISC II and PISC III results to non nuclear plants structural integrity aspects could be effective in cases such as:
- heavy section piping and pressure vessel testing (wall thickness from 50 to 100 mm);
- heat exchanger tubes in general;
- large turbine components.

Examples were contributed from Italy (ENEL and ENEA) and from CEC (COST 505 programme).
Further assessment of possible work will be made in December 1989.

Action No. 9 Proposal (Support to Code and Standard Organization).
The Management Board has agreed, subject to acceptance by OECD-NEA and CEC-JRC, that it should directly encourage and support the development and improvement of NDE codes and standards by national and international bodies (for example CEN, ISO, IIW).
This can be done in three ways:
- the provision of information through presentations and reports on PISC results and PISC related programmes;
- the critical review by PISC members of code and standard proposals developed by the technical groups of codes and standards bodies;
- the preparation of technical reports to assist to technical groups of codes and standards bodies elaborating and discussing.

The execution of Action 9, involving the establishment of an expert group within PISC, supported by the Operating Agent, will be discussed in December 1989, with a view to its finalization.

Conclusions

PISC III is a large and comprehensive international exercise.
The strong support from national authorities and NDE organizations attests a wide-spread recognition that room remains for progress in understanding and improving inspection techniques applied to nuclear plant components, particularly for their optimal use in field conditions.
The support also highlights an acceptance of the important role of collaborative efforts to achieve this goal.

References

[2.1] PISC (Plate Inspection Steering Committee)
Full PISC I reports
CEC, EUR 6371en Volumes 1 to 6 (1979-1980)
OECD, CSNI Report No. 48.

[2.2] S. Crutzen, P. Jehenson, R. Nichols, N. McDonald
The Major Results of the PISC II RRT
Nuclear Eng. and Design, Vol. 115 (1989), 7.21.

[2.3] S. Crutzen, M. Certo, Ph. Dombret, R. Murgatroyd
Summary of the PISC Parametric Studies on the Effect of Defect Characteristics (EDC)
PISC III Report No. 8, OECD-CEC, 1989.

[2.4] PISC (Programme for the Inspection of Steel Components)
The PISC III Programme
PISC III Report No. 1 - OECD-CEC, 1989.

3. Contribution to the Specific Programme:

RADIO-ACTIVE WASTE MANAGEMENT

Safety of Final Storage in Geological Formation: Materials Research Aspects

During the year 1989 investigations have been performed on the effect of thermal gradient on leaching of borosilicate glasses and on corrosion of mild steel.

A large number of tests have been performed in isothermal conditions, which correspond to a time of life of the waste package of more than 500 years. For the first period of storage of the radioactive waste package, the radioactive decay generates a significant amount of heat, which creates a thermal gradient in the media surrounding the waste package.
An attack under a thermal gradient corresponds to normal repository conditions as far as the mild steel corrosion is concerned but to accidental conditions for the glass leaching. An apparatus producing a constant thermal gradient on a column of a wet porous media has been designed.

It is composed of a thick wall brass cylinder, which contains a tight fitting stainless steel sleeve.
A column of the porous media, 20 cm long, is held in the stainless steel sleeve. The sample is positioned in the upper part of the column. In order to obtain the desired thermal gradient, the two heads of the thick cylinder are kept at two different temperatures using two independent heating circuits. A series of five thermocouples, situated every 5 cm along the stainless steel sleeve checks that the thermal gradient is constant and of the desired amplitude.
The thermal gradient could affect the attack phenomena in two ways. Firstly, the diffusion could be influenced by the thermal gradient (Soret coefficient).

Secondly, the thermal gradient could alter the chemical equilibria of the minerals composing the porous media.
Two series, consisting of ten tests each, has been carried out using firstly one carbonaceous sea sediment as a porous media and then a paste made by mixing montmorillonite clay with distilled water in order to simulate the backfilling material which is usually put around the waste package. Both series of tests have been performed leaching a glass simulating the high level radioactive glass enriched in cesium (5%).

Tests were performed up to a maximum exposure time of 6 months using a thermal gradient of 1^0C/cm. At the end of each test the sample was retrieved and examined. The column was sectioned and sent for the cesium analysis, following a neutron activation technique.

While the glass samples leached in montmorillonite showed a normal appearance, those leached in carbonaceous sea sediments showed a surface layer of calcium carbonate which increased with the increase of the exposure time. The analysis of the cesium distributed in the porous media was used to calculate the diffusion coefficients. In the case of the montmorillonite, the diffusion coefficient show a slow decrease with time while for the carbonaceous sediment the decrease is more marked, indicating that the calcium carbonate deposit also acts as a diffusion barrier. The final report is in preparation. Another series of tests is being performed to analyse the influence of a calcium carbonate backfilling on a fissure formed in the mild steel container.

4. Contribution to the Specific Programme:

FUSION TECHNOLOGY and SAFETY

Materials Integrity

The Institute for Advanced Materials participates in the European Programme for Fusion Technology. It concentrates its effort mainly on the study of the behaviour of austenitic stainless steels in a fusion environment. By these studies it contributes within the EC programme to the projects NET technology, test blanket development and long term activities.

In the absence of a fusion typical neutron source, radiation damage is simulated by light ions from the Ispra Cyclotron or by neutrons from the HFR.

In addition to contributions to the data base on AISI 316L for the NET design, a substantial part of the effort is dedicated to the development and characterisation of low activation materials.

Low activation materials are alloys and materials where the neutron induced activation decays rapidly and to low values.
This can be achieved by:

- elemental substitution of critical elements in known compositions;
- isotopical tayloring of known materials,
- development of completely new materials composed only from elements which form rapidly decaying activation products.

Up to the present time the route of elemental substitution in stainless steels has been followed. In Tables below and on page 88 lists are given of alloys which have been derived from AISI 316 by substituting nickel by manganese. The composition of these alloys has been optimised with respect to their mechanical properties. An in depth characterisation of these alloys is underway. Studies on the mechanical behaviour and the microstructure before, after and during irradiation are reported. The oxidation under accident conditions and the interaction with liquid breeders are investigated.

For the other projects of the EC programme, the work on the interaction with liquid breeders is enlarged to include other structural materials. The consequences of disruptions on metallic surfaces are determined. Studies on the behaviour of stainless steel components under thermal fatigue conditions are underway.

Table below: *Chemical composition (wt%) of the Cr-Mn steels investigated and of the European Reference 316L type steel.*

	AMCR 0033	AMCR 0034	AMCR 0035	316L
Cr	10.1	10.1	14.1	17.44
Mn	17.5	17.7	19.9	1.82
Ni	<0.10	0.15	0.26	12.33
Mo	<0.06	1.52	<0.06	2.3
C	0.10	0.10	0.029	0.024
N	0.19	0.16	0.048	0.06
Si	0.55	0.64	0.63	0.46
S	0.008	0.008	0.006	0.001
P	0.016	0.025	0.018	0.027
Cu	<0.06	<0.05	<0.05	0.2
Al	<0.05	<0.05	<0.04	-
V	<0.02	-	-	-
Ta	-	-	-	0.01
Pb	0.0001	0.0001	0.0001	-
B	0.0025	0.0036	0.0028	0.0008
Co	-	-	-	0.17
Fe	balance	balance	balance	balance

The development of coatings which enhance the optical absorptivity of stainless steel is requested by NET. Finally, the application of fibre reinforcement to the development of a divertor plate is being considered.

Table below: *Chemical composition of optimized Cr-Mn stainless steels.*

	IF-A	IF-B	IF-C	IF-D	IF-E
			(wt%)		
Cr	13.57	12.37	13.14	10.24	17.86
Mn	11.34	10.62	18.00	16.92	11.00
Ni	2.04	0.23	2.14	0.13	2.08
Mo	0.031	0.023	0.037	0.026	0.041
C	0.10	0.31	0.10	0.26	0.08
N	0.047	0.036	0.042	0.080	0.054
Si	0.20	0.17	0.20	0.50	0.30
V	0.63	0.64	0.021	0.032	0.74
W	1.42	1.38	1.92	2.04	2.02
			(ppm)		
S	70	70	50	30	70
P	130	140	130	80	140
Cu	370	290	360	240	370
Al	30	30	30	45	40
Nb	50	50	50	50	50
Ta	50	50	50	50	50
Pb	2	1	2	1	1.5
Co	220	200	210	200	220
B	3	3	3	3	3
Bi	1	1	1	0.5	1
Ag	1	1	1	1	1
Ti	10	10	10	20	10
Fe	balance	balance	balance	balance	balance

Deformation and Fracture Mechanisms in Cr-Mn Steels

Transmission and scanning electron microscopy studies of deformation substructures and fracture mechanisms in chromium manganese austenitic steel have now been completed. The development of dislocation structures in AMCR 0033 has been investigated in the temperature range 20-800°C. Strain induced martensitic gamma to epsilon transformation occurs during low temperature deformation. A parabolic rather than sigmoidal strain dependence of martensite volume fraction was found. At temperatures from 200°C to 600°C equiaxed dislocation cell structures were present, while above 600°C dynamic recovery lead to subgrain formation. The variation of dislocation cell diameter has been determined as a function of temperature. Scanning electron microscopy has shown that martensitic transformation causes a brittle, transgranular fracture node at low temperature.

A related activity is concerned with the effect of thermomechanical treatments on the dislocation substructure and the resulting influence on mechanical properties. Small amounts of pre-strain inhibit martensitic transformation and can produce significant improvement in low temperature ductility. Three different thermomechanical processes are considered: (i) deformation at 200°C; (ii) deformation at 400°C; (iii) room temperature deformation followed by annealing at 400°C.

Behaviour of Mechanical Properties under Irradiation

HFR Irradiations

The experiment series FRUST (Fusion Reactor Utilisation of Stainless Steel) provides irradiation data of stainless steel tensile samples irradiated in a central position of HFR. The irradiations were first performed in a TRIO type capsule and are pursued in a new irradiation capsule named SIENA (Steel Irradiation in Enhanced Neutron Arrangement).

The experimental conditions of the SIENA irradiation device are:

- irradiation temperature 180-500°C for 316 L stainless steel;
- neutron fluence: corresponding to 30 or 35 dpa, which implies an irradiation duration of about five years in the best HFR position, with the possibility to unload part of the samples, after accumulation of a lower dose;
- helium/dpa ratio as close as possible to the value of Cr-Ni austenitic steel in a fusion reactor first wall, which is obtained by spectrum tailoring.

During this reporting period the irradiation of 316 steel welds at 180°C up to 1.2 dpa have been completed. The post irradiation analysis of the specimens have also been carried out.

In the same period irradiations up to 10 dpa of tensile specimens of Cr-Mn steels (AMCR type) weldments irradiated at 250 and 450°C and of Cu-Cr-Zr alloy, foreseen for divertor applications, irradiated at 150 and 250°C have been completed.

Starting this year, new optimized Cr-Mn steels, IF type, will be selectively irradiated in SIENA, based on the knowledge acquired from the previous irradiations, at 250 and 450°C up to 10 and 25 dpa.

Cyclotron Irradiations

Fatigue Crack Growth under Light Ion Irradiation

The anticipated pulsed operation of Fusion Reactors will impose cyclic thermo-mechanical loads in structural materials. The resulting creep and fatigue associated with radiation damage can limit the life time of the first wall. Fatigue crack propagation under irradiation is of critical importance for the design of Fusion Reactors first wall structures.
A stress controlled in-beam fatigue crack growth experiment has been operation at the Ispra cyclotron for several years.

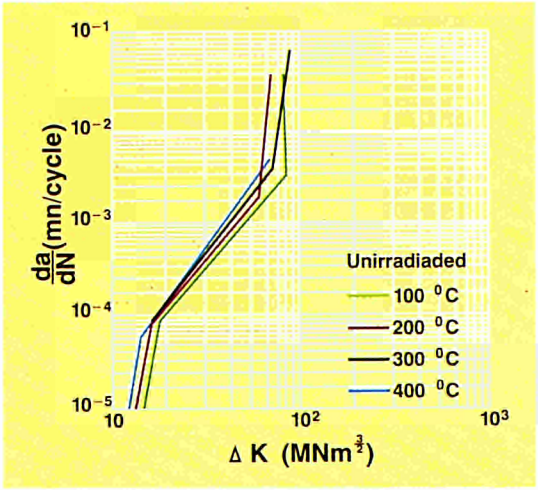
Following the NET requirements, a series of in-beam fatigue crack growth experiments on AISI type 316 stainless steel at low temperature have been continued. Following the irradiation planning, tests will be conducted at 300, 200 and 100°C with 18 MeV protons producing damage at the rate of the order of 10^{-7} dpa s^{-1}. The series of tests at 300°C and 200°C have been completed.

The average crack growth per cycle, da/dN, has been plotted against the stress intensity factor range ΔK.

The results are similar to those obtained at 500°C, where at increasing growth rates a gradual deviation from the data without irradiation has been observed. These results indicate a slightly lower fatigue crack growth at 300°C and 200°C in AISI type 316 steel.

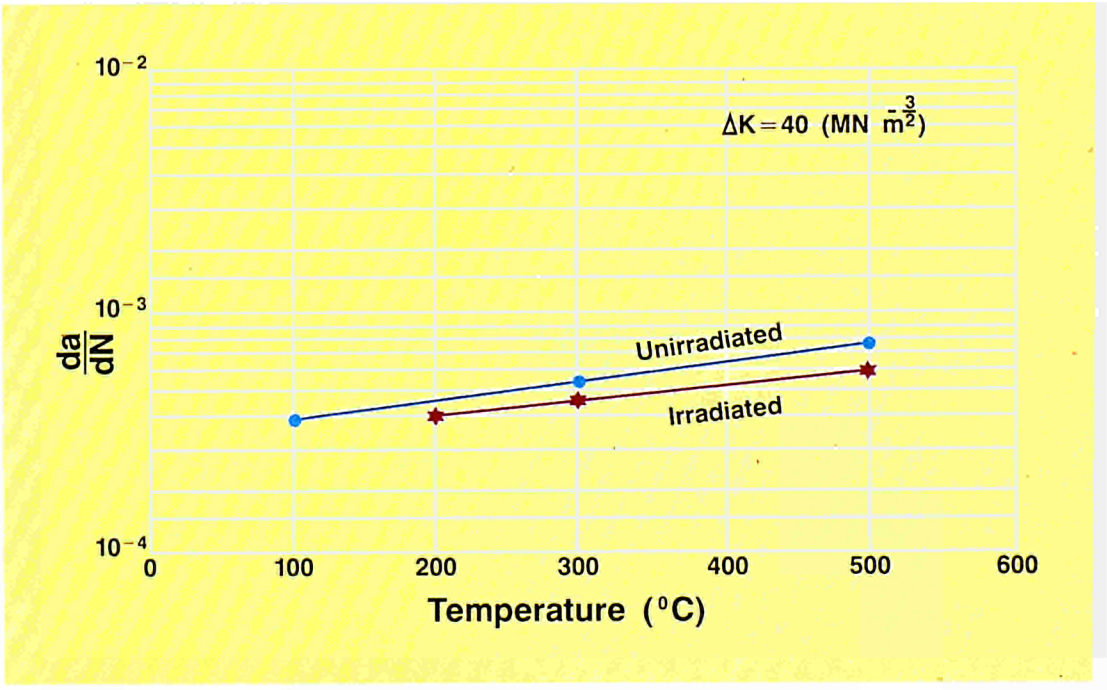
Tests for comparison on unirradiated material at 300, 200 and 100°C, have been completed. A small environmental chamber is used, which allows testing in an inert helium atmosphere. Testing is performed using a Schenck-Trebel mechanical testing machine operated under closed loop conditions in a load controlled tension-tension mode. Results are presented in figure above.

Figure below shows the average crack growth per cycle da/dN in the secondary stage against the testing temperature, for a fixed stress intensity factor ΔK, a clear relationship between crack growth and temperature is observed in the unirradiated and irradiated material.
The results indicate also a slightly shorter fatigue life at lower temperatures in AISI Type 316 steel.



Above: *Crack growth per cycle versus stress intensity factor for unirradiated AISI type 316 steel*

Below: *Average crack growth per cycle versus temperature for unirradiated and irradiated material*



Effect of Low Irradiation Fluence on the Mechanical Properties of Type 316 Stainless Steel

Materials studies for Fusion Reactors necessitate investigations of the effect on mechanical properties of irradiation with 14 MeV neutrons. Due to the unavailability of a fusion test facility two basic types of experiment are carried out: irradiation with fission neutrons or with light ions to simulate the effect of displacement damage of high energy neutrons.

The planned experiment is aimed at the study of the effect of low proton fluence on the mechanical properties of AISI 316 type stainless steel at low temperature. This study has two objectives. The first is to investigate the mechanical behaviour of 316 steel under various operating conditions, as expected for the physical phase of NET. To attain this objective the following variables are considered:

Proton fluence	: 10^{16} to 10^{19} pcm^{-2}
Damage level	: <1 dpa
Damage rate	: ~5.10^{-7} dpa s^{-1}
Irradiation temperature	: RT-300^{0}C

The second objective of this study is to compare the results obtained using proton-beam experiments with those obtained using different irradiation sources (HFR, HFIR, RTNS etc) for the same type of material. Such a comparative study is necessary in order to correlate the neutron effects with proton effects and it will contribute to the understanding of the validity of the different simulation techniques used for radiation damage studies.

During this period irradiations at different fluences up to 0.3 dpa at 300^{0}C and at room temperature have been performed.
Post irradiation tensile testing, micro-hardness measurement and SEM analysis are in progress.

Mechanical testing, micro-hardness tests and microstructure analysis of solution annealed and heat treated (300^{0}C) unirradiated material have been completed.

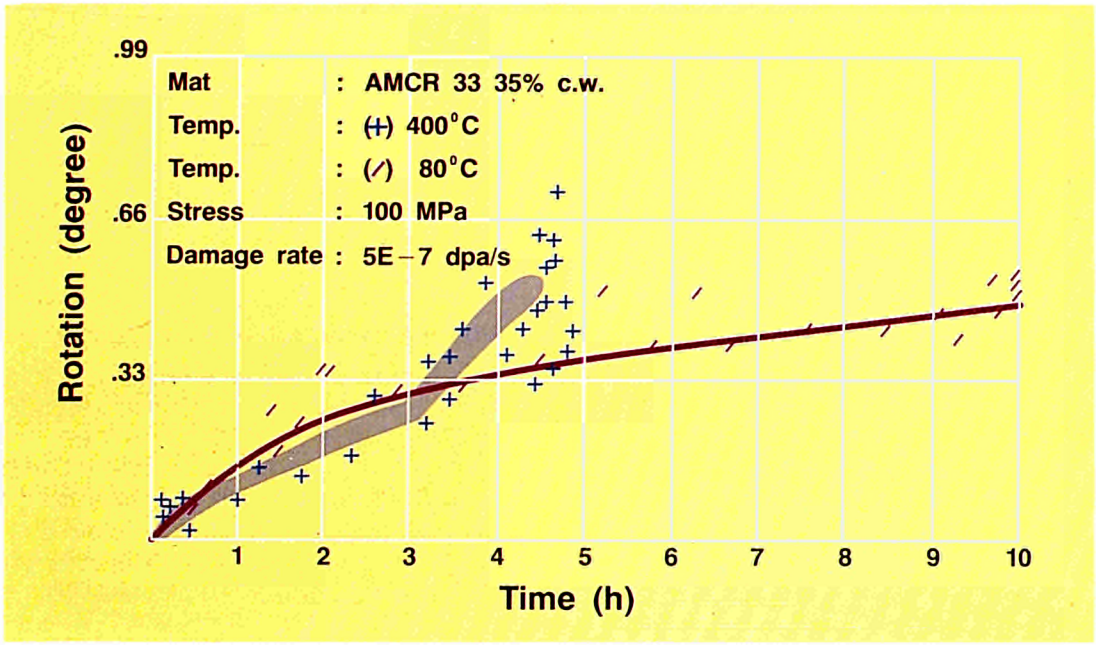
High Temperature Implantation Chamber

A high temperature irradiation chamber for implanting α-particles up to 800^{0}C in specimens of different geometries is under construction. The development of the irradiation chamber is carried out in collaboration with ENEA-Casaccia and the Engineering Department of the University of Ancona. This device will also be used to implant protons in 316 stainless steel tensile specimens, in order to study the effect of implanted H_2 on the mechanical properties. A preliminary study concerning the definition of the Al beam degrader and the geometry of the specimen to be irradiated is at present in progress. The thickness and the number of Al layers to be used to produce uniform p or He implantation in the whole specimen have been calculated using the computer code TRIM-88.
The geometry of the specimens resulted to have 0.5 mm thickness for protons and 0.24 mm for α-particles. These values were obtained taking into account the ion beam energy, the irradiation time, the heat transfer and the possibility to perform mechanical tests after implantation. Our results also showed that 40 Al foils are necessary for 28 MeV protons and 100 foils for 38 MeV α-particles in order to produce a uniform ion implantation.

Deuteron Irradiation Creep of 20% Cold-Worked Type 316 L Stainless Steel at "low" Temperatures

Previous irradiation creep tests on two types of stainless steel, 20% cold-worked 316L and 35% cold-worked AMCR 33 showed that for both materials:

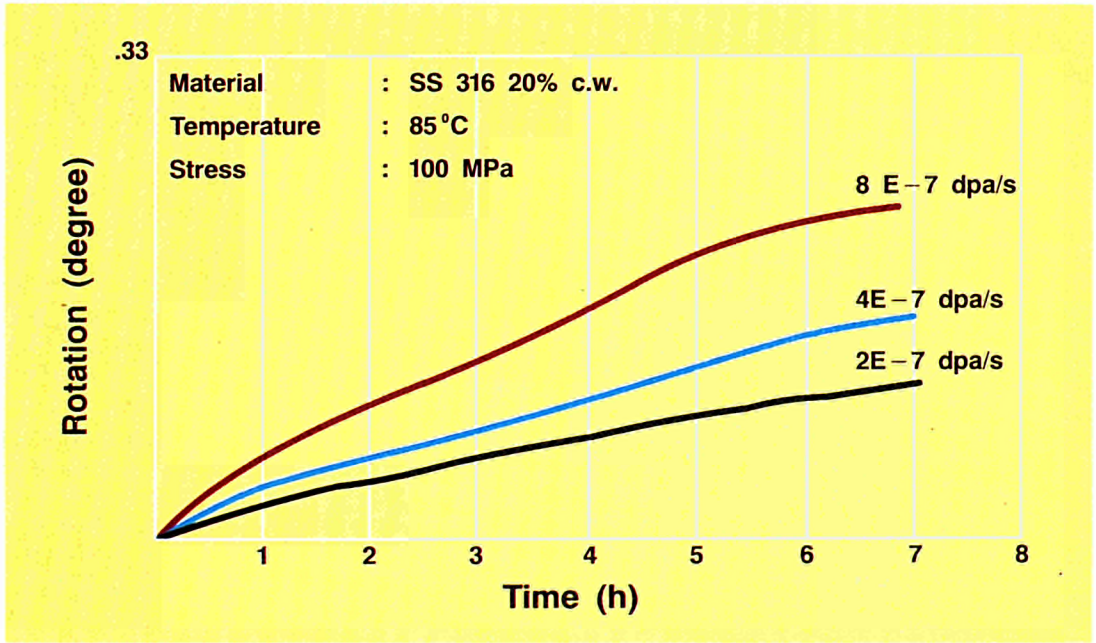
- the irradiation creep deformation is not negligeably small at temperatures T <100^{0}C,
- the irradiation creep rate slows down during the first 3-4 hours of irradiation and reaches thereafter a constant value for T <100^{0}C. The creep rate at the beginning of the irradiation was higher at 80^{0}C that at 400^{0}C (see figure page 92 above).
- For temperatures T >350^{0}C the irradiation creep rate stayed constant for the duration of the tests, if the sample had crept thermally for a sufficiently long time at the irradiation temperature, before the irradiation was started, such that thermal creep transients are suppressed.



In order to give an explanation for this unexpected irradiation creep behaviour at low temperatures, two types of experiments have been performed. In a first series of tests the irradiation creep rate of a 20% cold-worked 316 L stainless steel specimens has been determined as a function of the deuteron flux for a specimen temperature of 85°C. The damage rates ranged from 2E-7 to 8E-7 dpa/s. The results are given in figure below.

Above: *Strain time behaviour of a stainless steel specimen under 19 MeV deuteron irradiation for 80 and 380°C*

Below: *Strain time behaviour of a stainless steel specimen under 19 MeV deuteron irradiation for 3 different damage rates*



In figure below, the steady state irradiation creep rates are plotted as function of the damage rate K in a log-log display. This plot shows that a linear relation exists between log($^1/_s$) and log(K) with a slope s = .58, i.e. the irradiation creep rate is proportional to K.58.

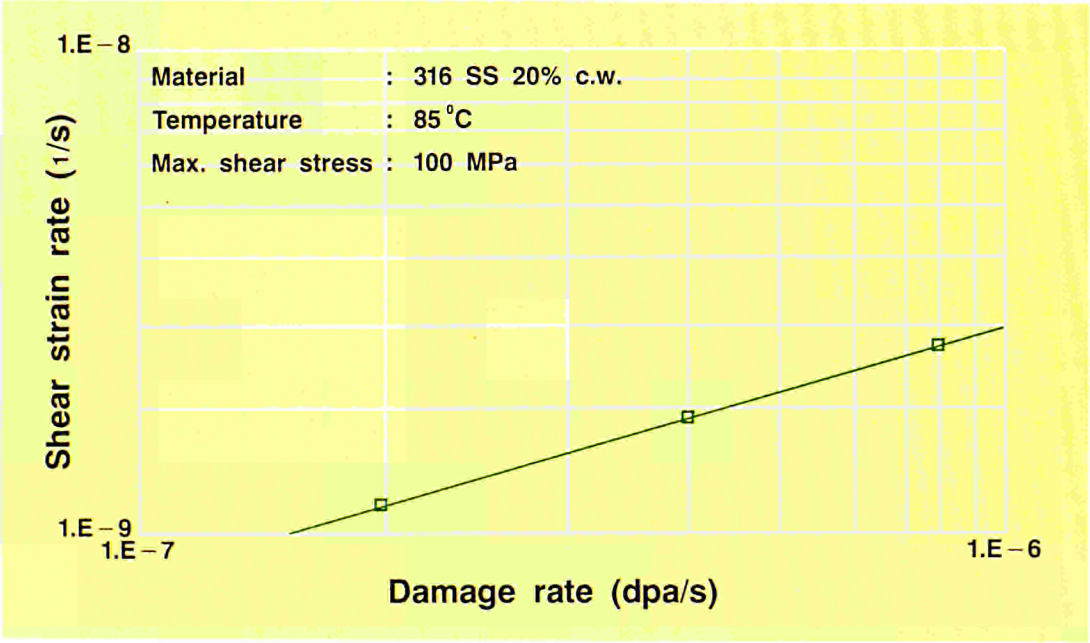
In a second series of tests a 20% cold-worked 316L stainless steel specimen was irradiated with 19 MeV deuterons at a temperature of about 80°C for 5 hours, until the irradiation creep rate had reached its steady state value. Then the specimen temperature was increased to 300°C for half an hour and turned back to 80°C, without changing the irradiation conditions. Thereafter, the irradiation creep curve at 80°C was measured, again, for about 5 hours. This procedure was repeated three times. The irradiation creep curves at 80°C were equal before and after each temperature drop from 300°C to 80°C, within the experimental error, i.e. at the beginning of the irradiation at 80°C, large transients were observed, during which the creep rate slowed before reaching its steady state value. An identical result was obtained when the irradiation was switched off before the temperature increase to 300°C, such that the specimen crept thermally at 300°C.

A qualitative explanation for the experimental results can be given in terms of the different magnitude of the point defect concentration, namely that of interstitial c_i and that of vacancies c_v, which are built up in the specimen at the different irradiation temperatures: assuming that dislocation climb is the basic microstructural process for the irradiation creep deformation 1). The dislocation climb rate v is proportional to the net point defect flux to the dislocation, which depends on c_v and c_i as follows

$$v \sim Z_i D_i c_i - Z_v D_v c_v \qquad (1)$$

where Z_i is the capture efficiency of dislocations for interstitials and
D_i is diffusion coefficient of interstitials,
Z_v and D_v are the analogous quantities for vacancies.

Below: *Shear strain rate during irradiation with 19 MeV deuterons as function of the damage rate in a log-log display*



The rate theory which is commonly used to calculate the point defect concentration as function of the temperature T, the particle flux K and the irradiation time t, predicts: the steady state concentration of interstitial and vacancies is directly proportional to the particle flux, when the annihilation at sinks, such as dislocations, is the predominant defect loss mechanism, there is a square root dependence c_i K and c_v K, when the point defects annihilate mainly by pair recombination. Since the steady state irradiation creep rate is proportional to K.58, one can conclude that pair recombination is the main defect loss mechanism for an irradiation temperature of 80°C and damage rates ranging from 2 E-7 to 8 E-7 dpa/s.

The creep transient which are observed at the beginning of each low temperature irradiation, may be related to the built-up times of the vacancy concentration. These built-up times are in the order of some seconds at 300°C, but may last hours at 80°C in accordance with the duration of the observed irradiation creep transients, assuming an activation energy for the migration of vacancies of 1.1 eV. Since the steady state vacancy concentration at 300°C is considerably smaller than that at 80°C, the vacancy concentration increases after each temperature drop from 300°C to 80°C. The interstitial flux to the dislocations is obviously higher, before the point defect concentrations reach their steady state values giving rise to the observed irradiation creep transients at low temperatures. In this context, it is of little importance, whether the specimen at 300°C is irradiated or not, which explains that the same creep transients are found for the irradiations at 80°C after thermal and after irradiation creep at 300°C.

Irradiation Creep-Fatigue Interaction in Torsion.

In a torsional creep experiment a cylindrical specimen is stressed in torsion and the resulting shear strain is measured as function of time. The torque is applied to the specimen by a current carrying coil inside a permanent magnet. Stress reversal, which is needed for the planned creep-fatigue tests, can be accomplished simply by inverting the direction of the coil current. The first tests under cyclic loading revealed the need for a modification in the torquing device in order to avoid grips slippage and specimen misalignment, which became evident in the fatigue tests after a number of cycles. To this purpose, the coil-specimen assembly including the specimen holders and the coil bearings have been modified.

The tests with the modified experimental set-up showed that the problems regarding the specimen mounting and the specimen alignment have been resolved so that the first fatigue experiments could be started. Fatigue tests were performed at room temperature on two 316L stainless steel specimens in 20% c.w. conditions. The number of cycles to rupture was determined for two different shear strain ranges. When the shear γ strain is transformed into its tensile equivalent ε with the relation $\varepsilon = \gamma/\Gamma_3$, the results are in agreement, within the error limit, to the corresponding data in the literature.

Neutron Induced Irradiation Creep and Damage Studies

In the reporting period results were obtained on:

i) irradiation creep at the HFR at Petten and
ii) phase transformations in manganese containing stainless steel alloys
iii) radiation enhanced diffusion in nickel single crystals.

Much of the data obtained were discussed and published. The main results were as follows:

i) The irradiation creep elongation in nickel or manganese containing stainless steel alloys investigated (in the HFR Petten) show a primary creep behaviour up to about 5 dpa. In some materials the creep elongation is negative due to the formation of precipitates at the beginning of the irradiation.
   This negative creep elongation effect increases with decreasing stresses and decreasing irradiation temperature.
   The irradiation creep rates obtained for US 316 type stainless steel alloys in ORR and EBR II and in HFR at Petten for ca 5 dpa are very similar, within a factor two.
   It is assumed that for the achievement of the dynamic precipitation and defect structures in the irradiated materials doses larger than 5 dpa are necessary.

ii) In the previous annual report it was reported that the phase field of manganese containing stainless steel alloys is much narrower than assumed previously.
The alpha-iron phase of deformed austenitic stainless steel alloys is formed during heating till about 350°C and dissolved during heating above this temperature, i.e. the alpha-iron phase is not stable below 600°C.

However, this phase is readily formed during high energy particle irradiation below 600°C.
Two-phase manganese containing iron-chromium steels are very brittle.
Plastically deformed materials break during heating up to 600°C.

iii) It was posible to determine the very small radiation enhanced diffusion coefficients in the next nearest surface region and in the bulk of nickel single crystals. The migration activation energies of interstitials and vacancies could be derived directly from the diffusion profiles. These activation energies decrease drastically with increasing irradiation flux.

It was further found that the rate determining diffusion mechanism near the surface (or sinks for point defects) is due to a vacancy diffusion mechanism, the rate of which decreases with increasing flux.
In the dislocation free bulk, the diffusion rate is determined by an interstitial diffusion mechanism, the rate of which increases with increasing irradiation flux.

These results are considered important for the formation and development of radiation enhanced and radiation induced structures especially if such irradiations are performed on thin foils for electron microscope studies or on plastically deformed materials.

It is concluded that the irradiation creep rate in a plastically deformed material is determined through a thermal point defect migration due to the large interaction fields between dislocations and point defects.

This means that the creep rate is almost independent of the irradiation temperature between 600°C and ambient temperature.

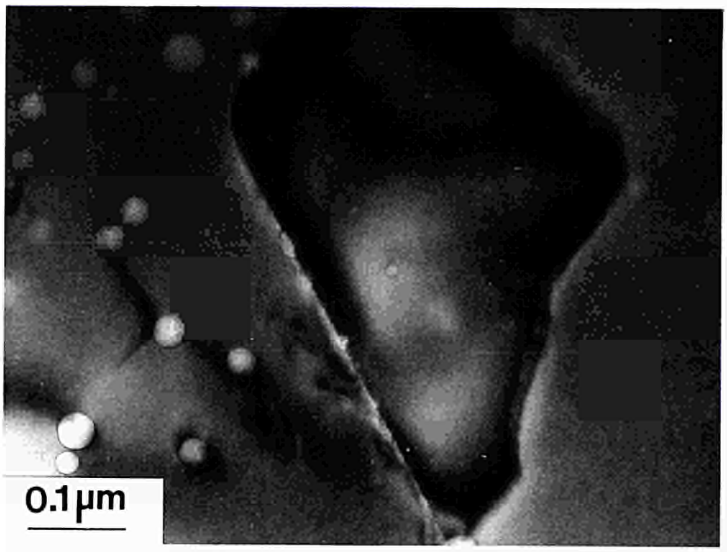
Microstructural Evolution in Cr-Mn Austenitic Steels Exposed to Alpha Particle Bombardment

Data on the phase stability of Fe-Cr-Mn alloys (simple ternaries and commercially produced) subjected to high neutron doses and displacement damage are available (Pacific Northwest Labs).
Up to the present time, no data were reported on the microstructural evolution of such materials subject to alpha-particle bombardment.
The structural changes in commercial Cr-Mn austenitic steels exposed to high doses have been studied over several years making use of the cyclotron technique which allows implantation of large amounts of He. It has to be pointed out that a particle bombardment in the cyclotron without a simultaneous heavy ion or neutron irradiation produces only a low level of lattice damage.
First results have shown that the microstructural changes in α-bombarded Cr-Mn steels depend on many parameters.

First of all one has to consider that in such materials both oversaturated C and N are present and rather insoluble He is introduced which precipitate at fusion relevant temperatures.
Therefore, for an understanding of the effects of the α-bombardment on the structural stability, a comprehensive study of the forming of precipitates, of He-filled cavities and of the dislocation structure has to be carried out, for which electron microscopy and associated analytical techniques are very suitable. In 1989 the study of three commercial Cr-Mn steels (Nitronic 32, AMCR and AMCR 003) implanted with 1000 atppm He at room temperature and subsequently aged at temperatures from 773 to 1073 K has been completed.

The study consisted of a quantitative or semi-quantitative analysis by TEM, EDS and EELS (in collaboration with the Institute of Physics of the University of Bologna) of the dislocation loop structure, the formation and growth of precipitates and the nucleation and growth of He bubbles. As the most significant result, a remarkable difference in the microstructural evolution was observed for materials containing either a high N or C content.

In the former case a strong interaction between interstitial Cr and N was observed (Nitronic 32).



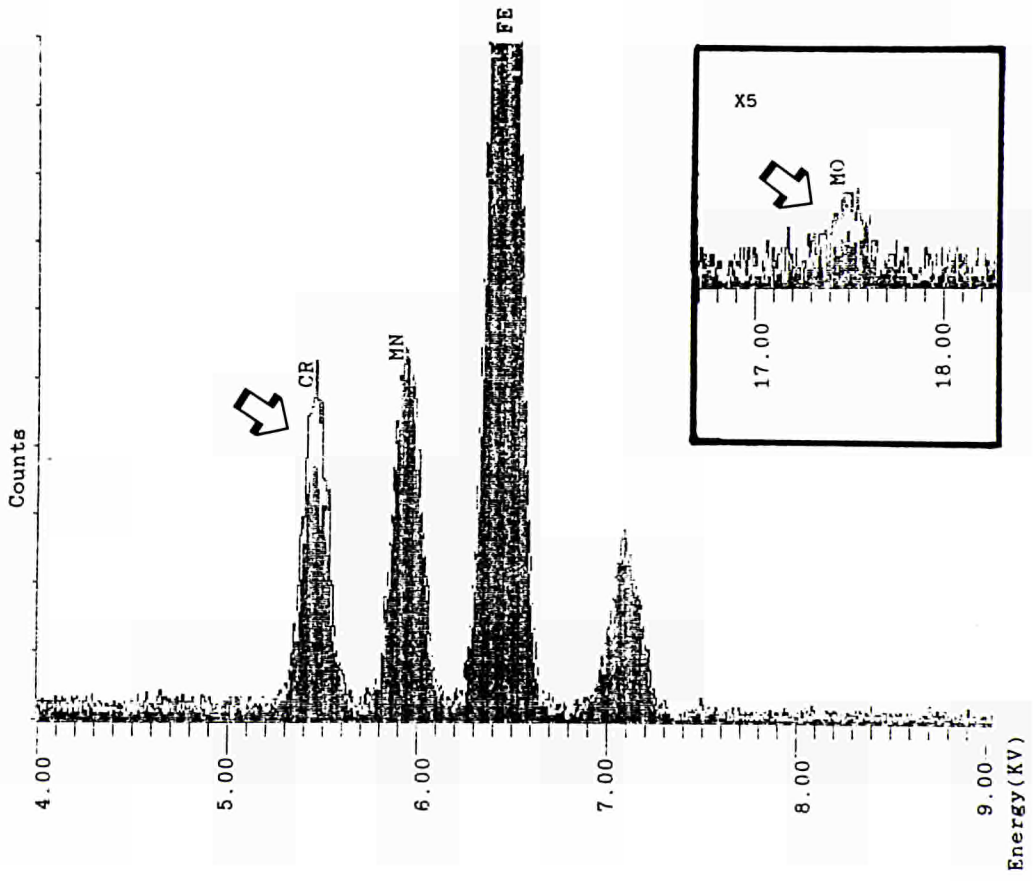
Above: *Formation of polygonal He bubbles in the matrix and at carbide interfaces in AMCR 0033 steel implanted with 1000 atppm He at room temperature followed by aging at 1023 K for 50 h*

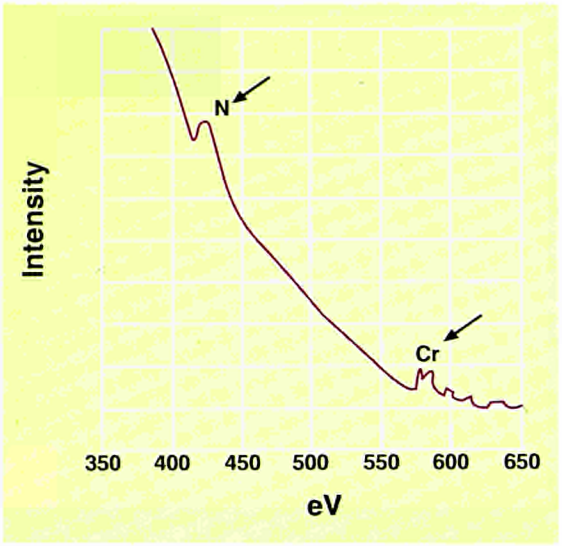
Below: *Superposition of EDS spectra obtained from intragranular He bubbles (bright) shown in the figure above and from matrix at 50 nm distance (dark). Note enrichment of Cr and Mo near bubble surfaces*

As a consequence slowly growing Frank interstitial dislocation loops are formed which act as nucleation sites for Cr_2N whose formation within the grains is otherwise very sluggish.

Simultaneously He bubbles are also formed which grow to a spectrum of sizes and are subject to segregation of Cr, N and Mo at their surface (Figures above, below and on page 97 above).

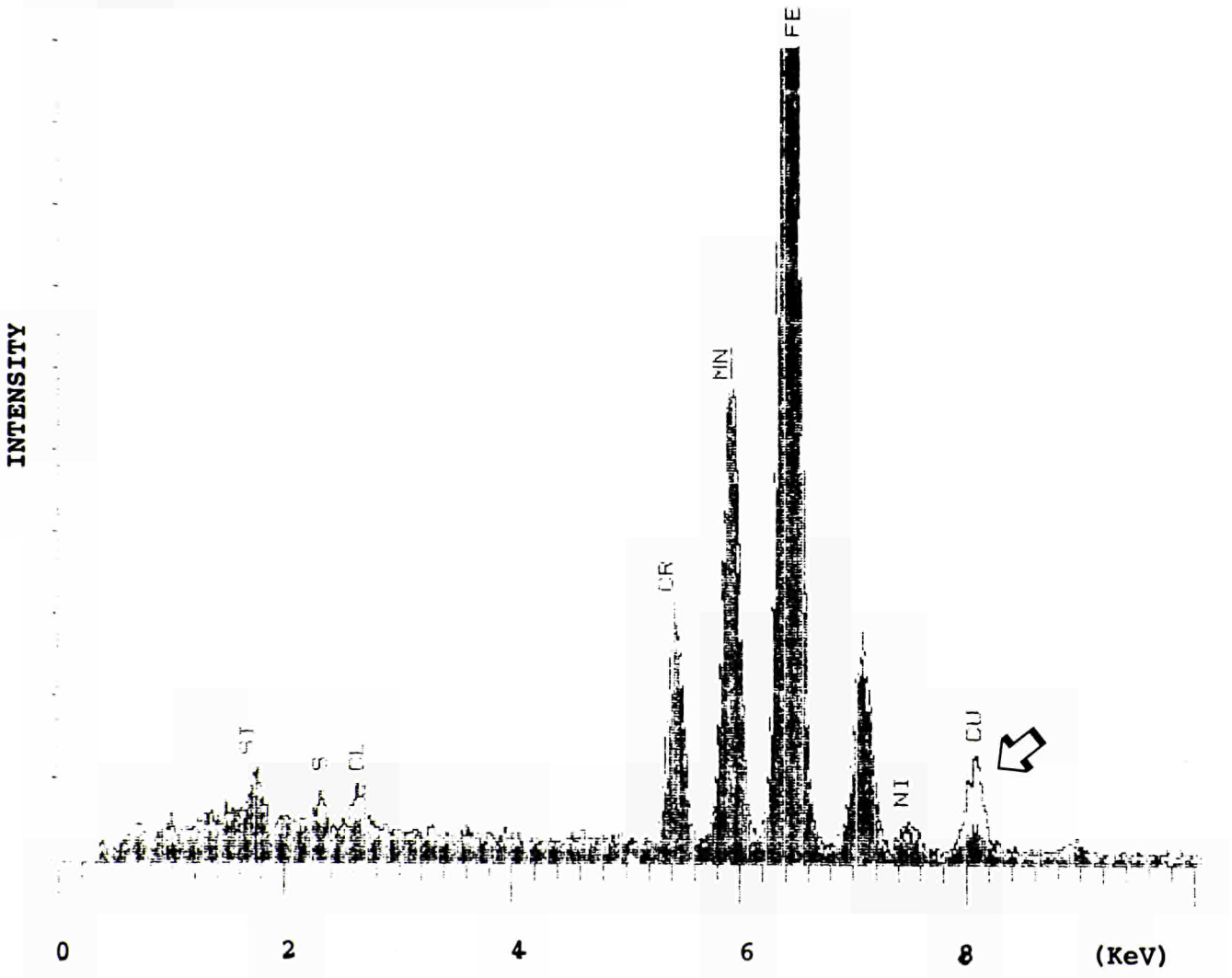
In the material high in C (AMCR), $M_{23}C_6$ carbides are nucleated at the Frank interstitial loops which on annealing at higher temperatures, grow and become reduced in size while He bubbles with a bimodal size distribution appear.
The class, being cube-shaped and having a larger size (~50 nm side length), showed an enrichment of Cu present as an impurity, at their surface (figure opposite below).





Above: *EELS spectrum intragranular He bubbles shown in the figure on page 96 above indicating enrichment of Cr and N near their surface*

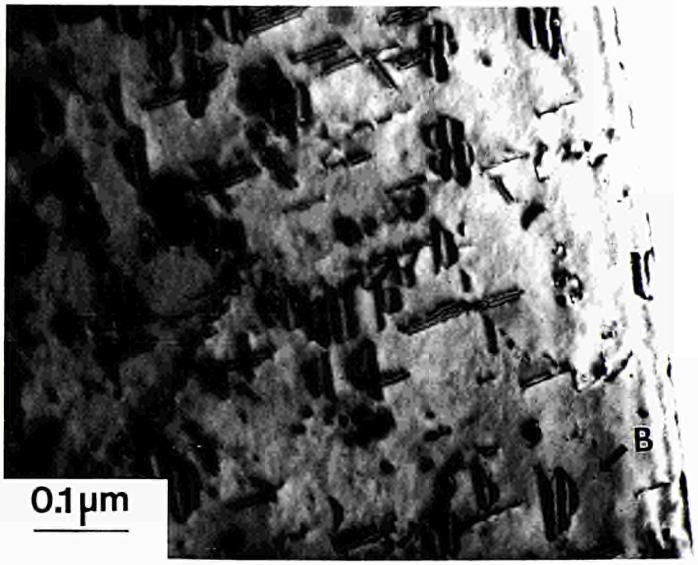
Below: *Superposition of EDS spectra obtained from square-shaped He bubbles (bright) and from matrix at 50 nm distance (dark) formed in AMCR steel implanted with 1000 atppm at room temperature followed by aging at 1073 K for 100 h*



In the steel containing an intermediate amount of C and N (AMCR 0033) finally, only the formation of He bubbles was observed, (apparently at the expence of C and N precipitation), exhibiting the enrichment of Cr, N and Mo at their surface.

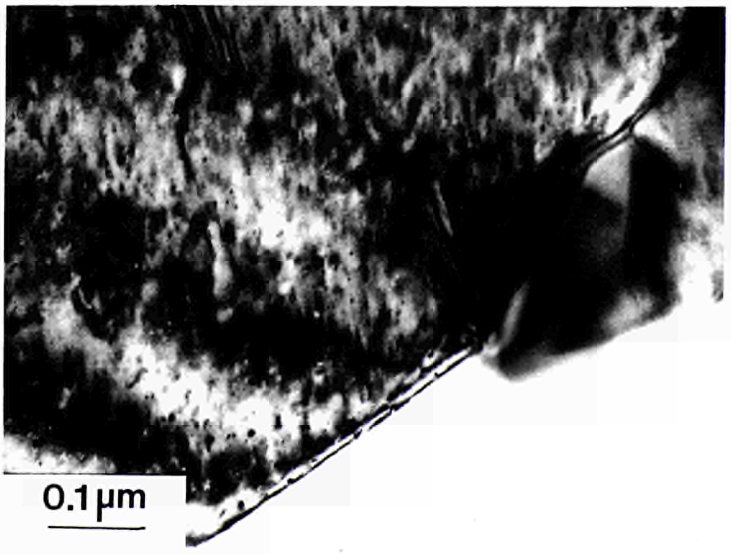
In October 1989 the examination of samples of the three steels, directly implanted at 773 K with 1000 atppm He, was initiated.

First results indicate that at this (fusion relevant) implantation temperature also, the microstructural evolution depends strongly on the N and C content. In the steels having a high N content (Nitronic 32 and AMCR 0033) large Frank interstitial loops due to the interaction of Cr with N together with very small He bubbles are formed whereas in the steels having a high C content (AMCR) the interstitial damage is annealed out and He bubbles and clusters are observed due to He- and C-vacancy interactions (figures above and below). The examination of these samples will be completed in 1990 together with a study of samples of the same steels α-implanted at 1073 K.



Above: *Formation of large Frank interstitial dislocation loops due to the interaction of interstitial Cr with N together with very small He bubbles (B) in AMCR 0033 steel implanted with 1000 atppm at 773 K.*

Below: *Formation of small He bubbles and clusters due to C- and He-vacancy interactions and annihilation of interstitial damage in AMCR steel implanted with 1000 atppm He at 773 K*



Mobilisation of Activation Products from Cr-Mn Austenitic Steels: Experiments in Flowing Air

There is a concern that in potential fusion reactor accidents larger amounts of radioactive Mn (^{56}Mn and ^{54}Mn forming from Fe and Mn in the Cr-Mn steels) could be released due to a combination of higher temperatures and oxidising conditions.

Since no data on the volatilisation of Mn in oxidising ambients are available, a few exploratory tests in air were performed in 1987 by researchers at INEL with the Cr-Mn steel AMCR 0033 supplied by the JRC-Ispra.
In these tests the samples were brought to the test temperatures very rapidly by induction heating. The release data obtained on this material at INEL showed a strong scattering.

A study of the oxide scales formed on these samples, subsequently carried out at Ispra, indicated that the observed losses (Fe, Mn and Cr in decreasing order) depend on the kind of oxidation of the Fe-Cr-Mn system. The observed scatter of the release data at INEL therefore could possibly be due to a diverse oxidation behaviour in the various tests.

In 1989 a study was initiated in collaboration with researchers of the University of Genova and ICFAM-CNR at Genova, of the oxidation behaviour of AMCR 0033 in flowing air by using a broader spectrum of experimental conditions with the aim to compare the results with those obtained for the same material in the INEL tests.

In our experiments coupons of AMCR 0033 with surfaces prepared in the same way as for the previous tests at INEL, were oxidized in a flowing air stream at temperatures from 1073 to 1473 K.

The samples were brought to the desired temperature in a resistance furnace with 100^0C/min and in an alumina furnace with 20^0C/min.
Test duration was up to 30 h.

As the oxidation kinetics showed, air oxidation behaviour of AMCR 0033 is strongly dependent on the initial experimental conditions and on the surface treatment.

In some experiments protective, metastable, oxide scales formed (Mn_2O_3 with Cr-Mn spinel underneath) while in others, performed in the same experimental conditions, the oxidation process was much less protective.

At the highest test temperature of 1473 K, the results were more reproducible and in accordance with the thermodynamic predictions.

In our work the time to heat up the samples was about 10 minutes and the oxygen pressure $\sim 10^{-3}$Torr before exposure to air.

Therefore in our study the heating up stage defined the following oxidation process whereas in the INEL experiments, due to the high vacuum and very rapid temperature increase, the preferential evaporation and compositional changes near the surface were the determining step for slow oxidation kinetics.

Transient regimes of different duration are therefore a possible explanation for the scattered results on mobilisation observed at INEL.

A paper summarizing these results, and in which a comparison with the INEL data is made, is in preparation and will be submitted to J. Nuclear Materials.

Blanket Materials: Influence of Pb-17Li on Properties of Materials

Susceptibility to Liquid Metal Embrittlement by Pb-17Li of AMCR welded structure.

This work is a continuation of the study on the possible embrittlement in Pb-17Li, at a temperature very close to the liquid metal melting point, of the candidate structural and blanket containing materials envisaged in the European Technology Programme.

Tensile specimens were machined from a welded joint of AMCR 0033 (shield metal arc electrode), they included welded and heat affected zone structures.

The tensile specimens, previously heat treated in Pb-17Li for 15 h at 773 K in order to achieve a good wetting, were tested at 523 K in Pb-17Li in a special device connected to an Instron machine that allows the testing of six samples, one after the other under vacuum or inert atmosphere.

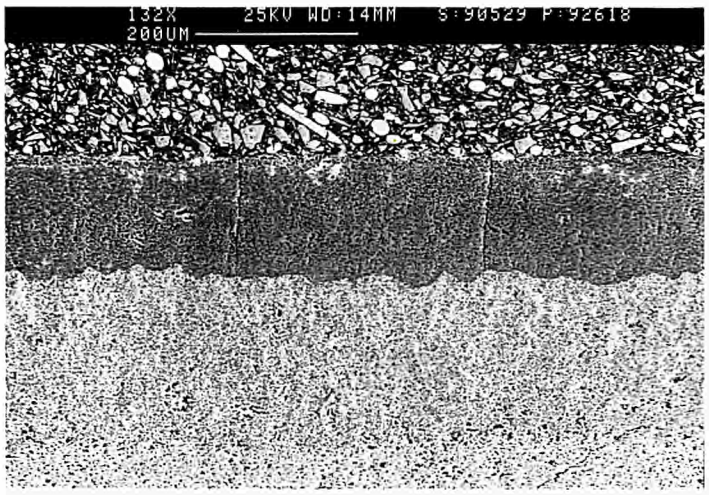
The total elongation values for the welded structure vary from 44 to 58%; the value for the HAZ is around 50%.
The reference value for a non welded specimen in the same experimental conditions is 62%.
At 723 K HAZ samples show an elongation of 44% in Pb-17Li compared to 40% in air.

Compatibility of Ceramics with Pb-17Li

The compatibility between different ceramic materials and the lithium lead eutectic Pb-17Li has been studied at 1073 K, for t = 1500 h. The experiments were conducted in electron beam welded molybdenum capsules.

From experimental as well as thermodynamic consideration it clearly appears that silicon carbide based materials could be used, at high temperature, as low activation materials to contain a Pb-17Li blanket.

The use of ZrC, TiN and MgO in a Pb-17Li environment is also possible at temperatures as high as 1073 K. Experimental results show that Al_2O_3 reacts intensively with Pb-17Li at 1073 K with the formation of both $LiAlO_2$ and $LiAl_5O_8$ (Figure on page 100 above).



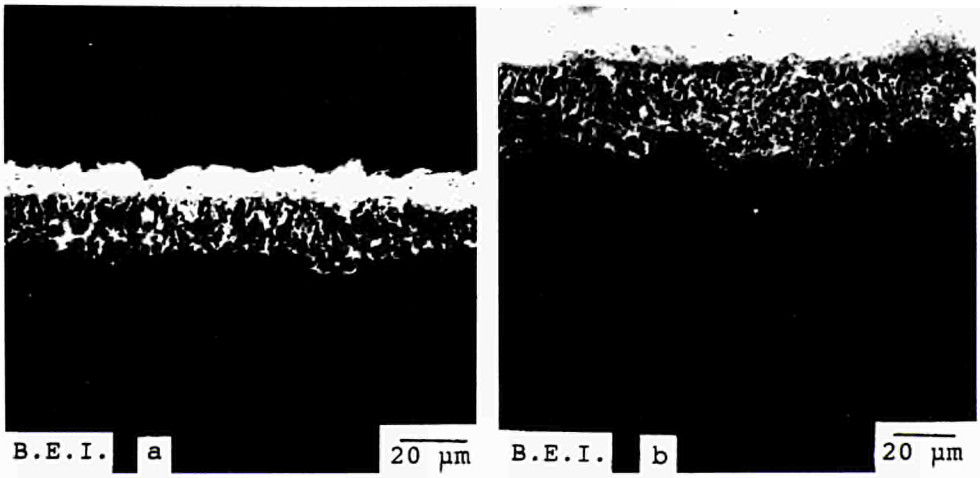
Corrosion of Cr-Mn based Austenitic Stainless Steels by Pb-17Li

The compatibility between the lithium-lead eutectic and different Cr-Mn steels has been studied in the temperature range 723 K in capsules in a rotating furnace for times up to 9500 h and in a thermal convection loop for 4200 h with a T of 50 K.

- The corrosion mechanism is essentially based on the dissolution of Mn and to a lesser extent Cr formation of a ferrite layer and penetration of Pb and possibly Li in this layer. Cold work enhances corrosion (Figures below).
- The behaviour of this family of steels in Pb-17Li is at least similar if not better than that of AISI 316.

Above: *Al_2O_3 heat treated in Pb-17 Li - 1500 h - 1073 K*

Below: *SEM micrographs of cross-section of AMCR 0035 heat treated in Pb-17Li at 723 K for 4200 h in AISI 316L T.C.loop. T = 50 K (a = long face, b = short face)*



Synergism between Corrosion and Mechanical Properties

Specimens of the Cr-Mn austenitic stainless steel AMCR 0033 and of type 1.4914 martensitic steel are heat treated in Pb-17Li under a constant uniaxial tensile load in Thermal Convection Loops at 723 K with a thermal gradient of 50 K.

AMCR 0035 wt %	Mn	Cr	Ni	Si
MATRIX				
Semi-quantitative EDX-microanalysis AVERAGE VALUE	19.80	14.23	0.00	0.77
CORRODED ZONE				
Semi-quantitative EDX-microanalysis AVERAGE VALUE	0.30	7.90	2.80	0.95

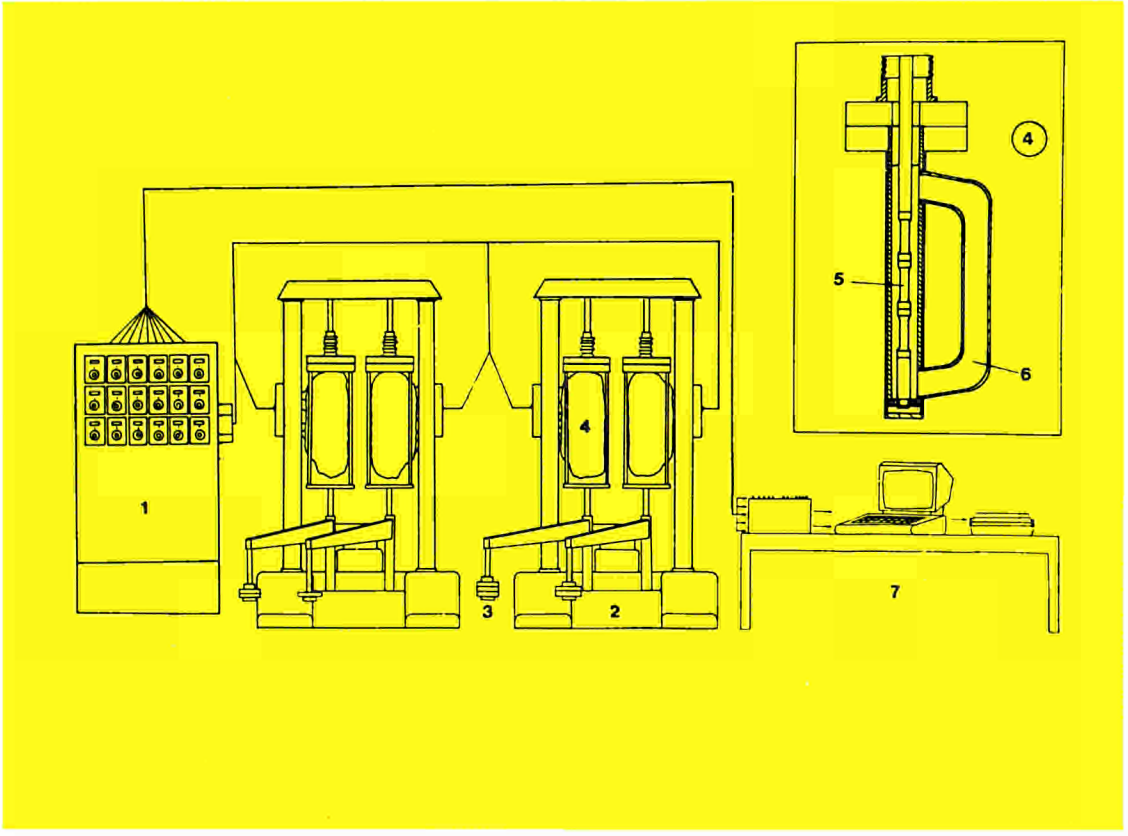
The experimental set-up is shown in figure below. Three loops, each containing three specimens, are presently running. The applied load varies from 250 to 280 MPa.
The loop containing AMCR has reached 9000 h while the other two are at more than 6000 h.
The experiments are scheduled to last for 15.000 h.

Effect of Oxygen on Corrosion of AISI 316 L by Pb-17Li

This study is a continuation of the work initiated some time ago in which it was shown that for times up to 300 h oxygen seems to enhance the corrosion of 316 steel by increasing the depth of the ferritic layer and the extent of chromium depletion within this layer. The apparatus in which the experiments are conducted consists of two resistance heated AISI 304 L containers filled with Pb-17Li (about 120 cm^3), situated in the same vacuum chamber, containing the samples to be tested. A known volume of 50% O_2 in Ar, at a continuously monitored pressure is passed through only one of the two test containers. The composition of the gas before and after reaction with Pb-17Li is constantly monitored by a quadruple gas analyser.

Below: *Device for Stress Corrosion Testing Specimens in Pb-17 Li in TCL:*

1 - Control Device
2 - Creep Test Machine
3 - Fixed Load
4 - TC Loop
5 - Tensile
6 - Data Logging



At intervals during the experiment and at the end, thermal and differential thermal analyses are carried out on the liquid alloy.

In the experiments carried out two samples of AISI 316L, one coated with $LiCrO_2$ and the other not, were inserted in each of the two Pb-17Li containers. The temperature was 873 K. The experiment had to be interrupted after 750 h due to plugging of the Argon-oxygen inlet tube. The final composition of the alloy in which the oxygen was bubbled was Pb-13Li while that of the reference pot was Pb-17Li.
The analysis of the result which necessitates an accurate Scanning Electron Microscopy analysis with quantitative determination of Cr and O contents is underway. In a first approximation it would seem that the coated samples are less attacked than the uncoated which would confirm that once $LiCrO_2$ is formed on the surface of the sample it acts as a protective barrier to corrosion by Pb-17Li.

The experiments will be repeated for longer times after modification of the experimental set-up to avoid further plugging.

Synergism between Effect of Hydrogen (Tritium) and Pb-17Li on the Properties of AISI 316 L

The purpose of this study is to heat treat in Pb-17Li tensile specimens previously hydrogen loaded at a specific H_2 partial pressure.

The samples will then be tensile tested in a LME device. For comparison the experiment is repeated at the same hydrogen partial pressure but with no liquid metal. In order to carry out this series of experiments the following device has been set up.

It consists of an electron beam welded AISI 316 L capsule containing 20 tensile specimens and the Pb-17Li. This capsule is inserted in a second container which is kept under a hydrogen partial pressure equivalent to that of the loaded samples. The system is resistance heated and placed in a vacuum chamber equipped with a quadruple partial pressure gas analyser which monitors possible hydrogen leaks which are then replenished.
In the experiment now underway (it has reached ~ 1800 h) the tensile specimens have been loaded at a hydrogen pressure of 1 bar, at a temperature of 723 K.

Simulation of Disruptions by Electron Beams

Installation and experience with the new electron beam gun

The new electron beam gun became operative in the course of this year and its characteristics have been determined and compared with specifications. The new machine allows the automatic (computer controlled) bombardment of metallic specimens with beam sizes from 3 up to 12 mm diameter. These beam sizes have been experimentally verified and the energy density of the spot has been analysed by scanning the electron beam over a pinhole while recording and storing the current. Afterwards, the information can be used in a P.C. to reconstruct a map of the energy density of the electron beam.

The main results of this analysis showed
1) the beam has not a perfect central symmetry, especially for larger spot sizes;
2) for small spot sizes up to Ø 4 mm, the current density increases towards the center of the beam giving a "Gaussian" current distribution;
3) for larger spot sizes (6 to 12 mm) the current density increases towards the center but dips at the center. This gives an annular energy deposition for the larger spot sizes.
4) The current density is not completely stable in time and depends on the adjustment and the aging of the cathode and other gun electrodes (current density seems also to depend on vacuum conditions).

The maximum beam energy of the gun has been determined and is equal to 30 KW (500 mA, 60 KV). The minimal practical beam energy is 1.5 KW (50 mA, 30 KV). The stability of power (voltage and current) has been measured and was found to be satisfactory: stabilization of current 0.14%, voltage 0.05%.
The beam of a given energy may effectively be switched on to a metallic specimen for times varying between 1 ms and 100 ms.

This experimental set-up can be used for disruption simulation studies and has now been operational (a first disruption study on stationary specimens is now completed and the results are being analysed).

In another experimental set-up a pulsed electron beam is used for temporarily melting the surface of a specimen fixed on a rotating specimen holder. The beam is switched on to the specimen for a predetermined time and follows the specimen in its movement. This set up is able to dump a given amount of energy, for a predetermined time on a metallic specimen, while the rotation of the specimen subjects the molten metal to centrifugal body forces. The purpose of this experimental set-up was to simulate electrodynamic body forces exerted on the wall material during a plasma disruption. This set-up has been tested on a limited set of specimens and its operation is entirely satisfactory.
Typical values obtained are

- bombardment area Ø = 8 mm
- Beam power : 20 KW
- Pulse time : 15 ms
- Body force : $0.85 . 10^6$ Newton/m^3.

Disruption simulation (with new EB gun).

Electron energy absorption

The first problem in experimental electron beam simulation is the exact determination of the energy absorption of electrons. The absorbed power E_a of an electron beam of power UI is

$$E_a = UI - \eta I . \vartheta_r$$

where ηI is the backscattered current

and ϑ_r is the average energy of the backscattered electrons.

The literature on the subject gives good values for η (the back scatter coefficient for electrons) but contrasting values for ϑ_r which is of course more difficult to measure exactly. For this reason we determined the energy absorption efficiency directly by calorimetric measurements.

$$\varepsilon = \frac{Ea}{UI}$$

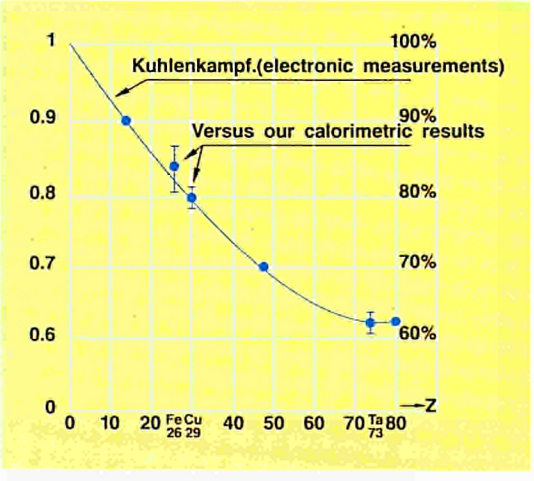
For this reason, a special copper calorimeter has been built with a conic hole. The opening of the cone has been calculated to absorb practically all electrons after multiple reflection, so that the calorimeter may be considered completely black for impinging electrons ~ 99.9% (energy absorption).

The impulse energy UIt (where t is the impulse time) was determined electrically and compared with calorimetric results for a range of currents and voltages.

The average deviation between electrical and calorimetric results is ~1.2%, which shows that UI can be determined with sufficient accuracy.

Afterwards, the conic electron collector was substituted by flat metallic specimens (stainless steel AISI 316, copper and tantalum). The absorbed energy E_a was measured by calorimetry. Figure below compares the experimental results with the results (continuous curve) calculated from the electron absorption factor n and the average energy of the backscattered electrons E_r obtained by Kuhlenkampf. This shows the validity of the values of Kuhlenkampf for calculating electron energy absorption and invalidates the values obtained by other authors (Brand, Sternglass deviation up to 40%).

Below: *ε = Efficiency of energy absorbtion for electrons (30~50 Kev)*
Z = Atomic number



Disruption simulation with monodimensional heat flow

During a disruption,a large amount of energy is deposited on a large surface of the first wall. This results in a monodimensional heat flow in the first wall. With the new machine it is possible to deposit comparable energies (1000 Joule/cm^2) on cylindrical specimens of 6 mm diameter. In this way monodimensional heat flow is created and the theoretical model for melt penetration can thus be tested.

For this reason 3 series of cylindrical specimens were prepared for disruption simulation experiments and bombarded with increasing energy density (from 100 to 1000 J/cm^2 impulse time 20 ms). The melt penetration was measured by metallographic methods and the weight loss was determined. The examination of the specimens is under way but the preliminary results show a good correspondence of experimental melt penetration and theoretical values obtained from the model described earlier.

High Emissivity Coating

In the NET (Next European Torus) project graphite tiles are proposed for protecting the AISI 316L vacuum vessel from plasma interaction. The coatings on the first wall act as selective absorbers for the energy radiating from the graphite tiles in the temperature range of 1000^{0}C - 2000^{0}C.
The coating increases the heat transfer and reduces the tile surface temperatures.

Coating Candidate

Considering the operational environment, Cr_2O_3, TiO_2 and Al_2O_3 have been proposed as absorbing coatings for their thermal and chemical stability.

Method for Producing the Coatings

Coatings have been produced by galvanic, flame spraying (FS), and air plasma spraying (APS) techniques. The effects of structure, chemical composition and surface morphology on emissivity at the different wavelengths have been studied. The thermal and chemical stability in vacuum and in hydrogen atmosphere at 500^{0}C have been checked. The adherence and thermal shock resistance have been examined using an electron beam gun.

Experimental work

Galvanic coatings.

Two types of coatings have been studied. Metallic Cr and black Cr. A Ni flash (Watts) increases the adherence of the coatings on 316 SS. The thickness range is between 5 and 10 µm.
The metallic Cr is not absorbing, it will be converted into Cr oxide by thermal treatment or by ionic implantation.
Black Cr, on the contrary, is very absorbing. It consists of a mixture of Cr and Cr_2O_3, (results from RBS analysis). The coatings are very hard and brittle and they are affected by transversal cracks which do not reduce the adherence. The amount of hydrogen in the coating and in the base material have been determined by RBS analysis.
Tests of annealing and thermal cycling (400^{0}C-100^{0}C) are now running. Their effects on the absorbance coefficient will be established.

Spraying Coatings

Due to its versatility, plasma spray coating seems to be a very promising method for producing thin and thick absorbing coatings. Tests started with the activation of stainless steel base material using sand blasting techniques. The grit blasting treatment to roughen and clean the surface which has be to covered is a prerequisite for good bonding.

Corindone 36, 40 mesh was used sprayed with dry air at 5 atm with an incidence angle of 45^{0}C.
The powders employed (Cr_2O_3, Al_2O_3, TiO_2) have been analysed by X-Rays before and after spraying. Using the correct parameters no chemical modifications have been produced during the deposition. The powder sizes have been determined by microscopic analysis and the heating energy has been chosen as a function of the powder grain size.

Flame Spray Coatings

Using an oxiacetylenic flame the following coatings have been deposited: Al_2O_3 85%, TiO_2 15% Cr_2O_3 50%, Al_2O_3 32%, SiO_2 10% and TiO_2 3%. The last film looks less adherent and very porous. This is probably due to the high melting point of the chromium oxide (2340^{0}C).
Better results have been obtained using propane instead of acetylene.

AISI 316

Figure above shows a metallographic section of a flame spray coating and figure below shows three absorption diagrams. For increasing the adherence of these ceramic coatings, bond films of NiCrSiB have been deposited on sand blasted steel. The results are very encouraging.

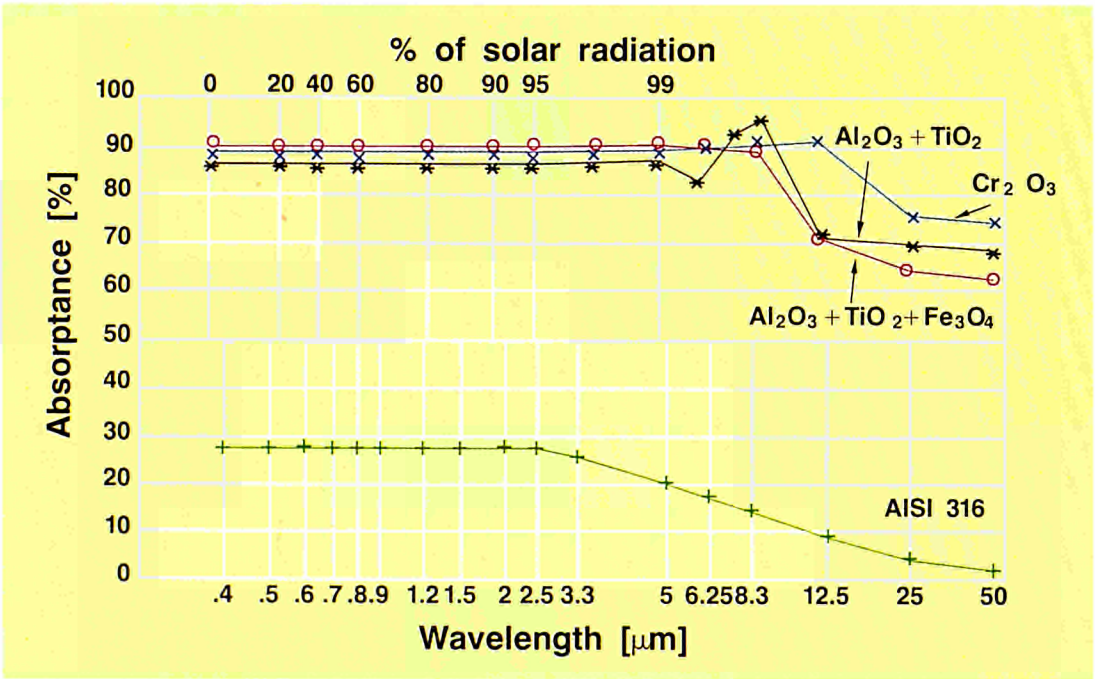
Air Plasma Spray Coatings

The conventional films produced by plasma spray are more dense and more adherent than FS coatings. This is due to the higher temperature and the higher speed of the deposition technique and they give higher bonding energy. Cr_2O_3 99% have been deposited by APS on sand blasted AISI 316.

Blended powder (Al_2O_3 87%, TiO_2 13% and Cr_2O_3 92%, SiO_2 5%, TiO_2 3%) APS deposited have been characterised with radiographic, metallographic and absorbance tests.

Above: *Cross-section of Cr_2O_3 film flame spray on AISI 316*

Below: *Hemispheral near normal spectral absorptance*



These specimens will now be studied under thermal cycling and stability tests at 400°C in a hydrogen atmosphere.
The work done up to now demonstrates that the grain size of the powder, its distribution and morphology play an important role in its flow through the plasma and the melting degree which have large affects on deposition rate, film density, adhesion, surface morphology and, consequently, absorption efficiency. For these reasons great care will be taken with the powder characterisation.

Fusion Reactor Components Development and Testing

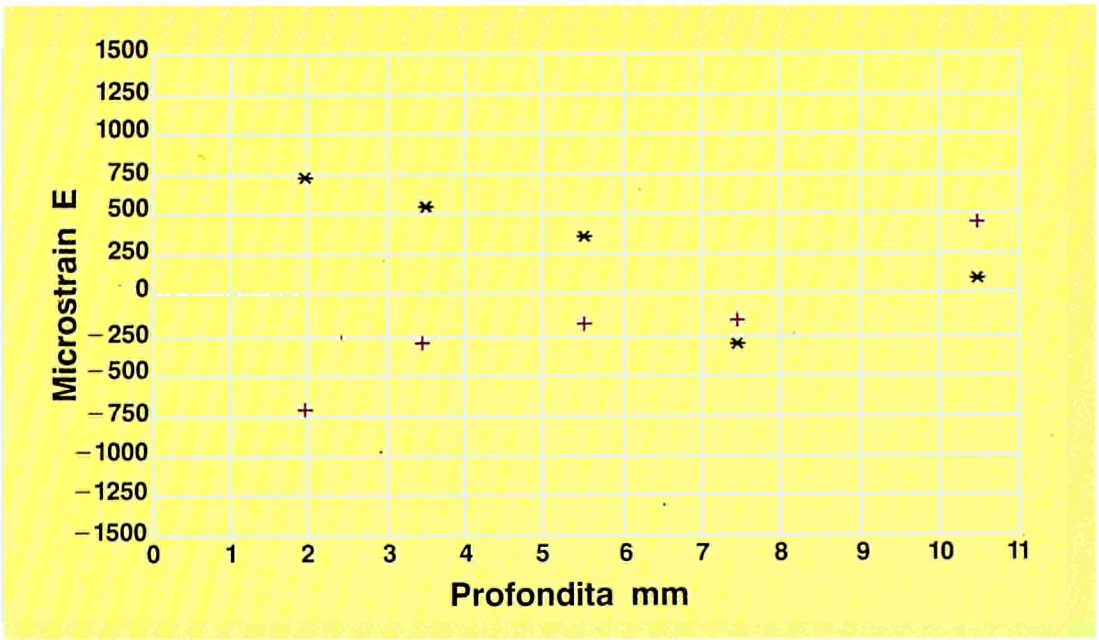
Thermal Fatigue of First Wall Components

The activity of thermal fatigue testing proceeded as scheduled with the exception of the building of the facility which is delayed because of infrastructure problems. Although no component reached failure during '89, many important achievements were reached, as detailed below.
The CAD for the two test lines were completed during March; the internal structures, the new instrumentation and the closed loop cooling circuit were purschased or put in fabrication. The completion of the facility improvements is expected by April '90. The hardware and software for data acquisition, reduction and analysis was installed.

The direct measure of the heat flux was carried out by a specially built probe based on a modified ASTM method. The data are under evaluation at present. The result seems to confirm the previous calculation of the heat flux based on the numerical thermal analysis.

The final report on the first completed test on the tubular specimen was presented at the ICFRM-4, Kyoto, December 89. Two benchmark specimens, B1 and B2,were tested up to 24,000 and 10,000 cycles, respectively. No evidence of fatigue damage in the highly stressed zones was detected by visual inspection, although failure occurred in the welds in the low stress region. On B1 failure the residual thermal strains were measured by neutron diffraction, in collaboration with Prof. Rustichelli, Ancona University, and M. Perrin, L.L.B., CEA Saclay.
The results are shown in figure below.

Below: *Neutron Diffraction*



On B2 the technique of geometric "Moire" was successfully applied to measure on-line the strains on the cold surface of the component. Due to lack of staff for of the FEM analysis, no comparison with the numerical results could be performed. The reseach agreement with IAEA was officially signed; the test on B1 and B2 will be the basis for the experimental validation of the ITER Lifetime Evaluation Benchmarck to be performed by stress analysis groups of the four participants.

The brazed Ansaldo 1 mock-up was tested up to 10,000 cycles. NDE by ultrasonic inspection revealed an increase in the size and in the number of the brazing defects already detected in the as fabricated component.

The Framatome 1 component was installed and the instrumentation checked by the manufacturer's technicians. The test started very recently. After the first thermal cycle, recorded by a thermocouple located at 1 mm from the heated surface, there was a sharp decrease in the peak temperature and a levelling off of this phenomenon in the successive cycles.
This effect could be attributed to one or to a combination of the following phenomena:

- training of the heating elements;
- displacement of the sensor tip due to the deformation;
- modification of the emissivity of the component surface;
- power supply drift;
- variation of the infrared absorptance of the inert gas environment.

Further investigation will be performed with improved instrumentation.

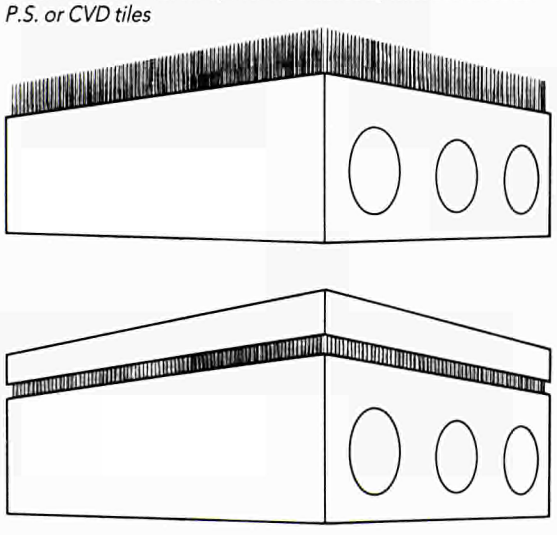
Development of an Innovative Divertor Plate Based on Fibre Composite Technology.

The problem of the bond between the protection material and the heat sink in a divertor plate has not yet been satisfactorely resolved. Both the mechanical attachments and the brazed joint present limitations as far as the power dissipation capacity and/or the lifetime under cyclic conditions are concerned. Figure below shows an innovative solution to this problem, based on fibre composite technology. The thermal and mechanical bond between armour and heat sink is realized through thin fibres, parallel to the thermal gradient, which link together two different materials leaving between them a small gap to avoid any direct mechanical coupling. The discontinuous nature of the interface avoids or greatly reduces the stresses due to the thermal expansion mismatch of the two dissimilar materials, yet allowing a substantial heat transfer by conduction through the fibres cross section. By a slight modification of well established metallic or carbon matrix fibre composite fabrication technology it is thus possible to obtain different combinations of heat sink and protection materials.
A microscale analysis was performed to assess the feasibility of the fibrous transition zone. The conservative criterium is to restrict the deformation of the most highly deformed fibre to the elastic region. The fibre stress was calculated on the basis of the difference in thermal expansion between the protection layer and the underlying composite materials. The temperature profile was derived from the F.E.M. calculations. The following analytical expression was used to optimize the fibre diameter D and volume fraction V and the gap A between tile and heat sink

$$S = (6*F*E*D)/(A**2) \qquad (1)$$

where S is the yield stress and E the fibre elastic modulus.

Below: *Brush Divertor a) with free fibres b) with continuous P.S. or CVD tiles*



The maximum displacement F is given by

$$(B*T2 - U*T1)*W \qquad 2)$$

where B and U are respectively the thermal expansion coefficient of the composite and of the tile, T1 and T2 the temperature of the composite and of the tile, W is the width of a divertor module. T2 is given by the expression:

$$T2 = T1 - (Q*A)/(K*V) \qquad (3)$$

where Q is the heat flux and K the fibre thermal conductivity. The results of the parametric study for both concepts shows that for a tungsten fibre of 0.025 mm diameter, a unit cell width of 54 mm and a fibre volume fraction of 50 percent, the gap should be at least 1.5 mm. These parameters were incorporated in both the above mentioned macroscale analysis with the optimized geometry and in the fabrication of the test piece for future experimental validation.

A prototype of a copper-tungsten divertor plate was fabricated starting from 25 micrometer diameter tungsten fibres plated with 5 micrometers copper. The fibres were hot isostatically pressed at 900^0C under a pressure of 1000 bar in a copper container.

5. Contribution to the Specific Programme:

NUCLEAR FUELS and ACTINIDE RESEARCH

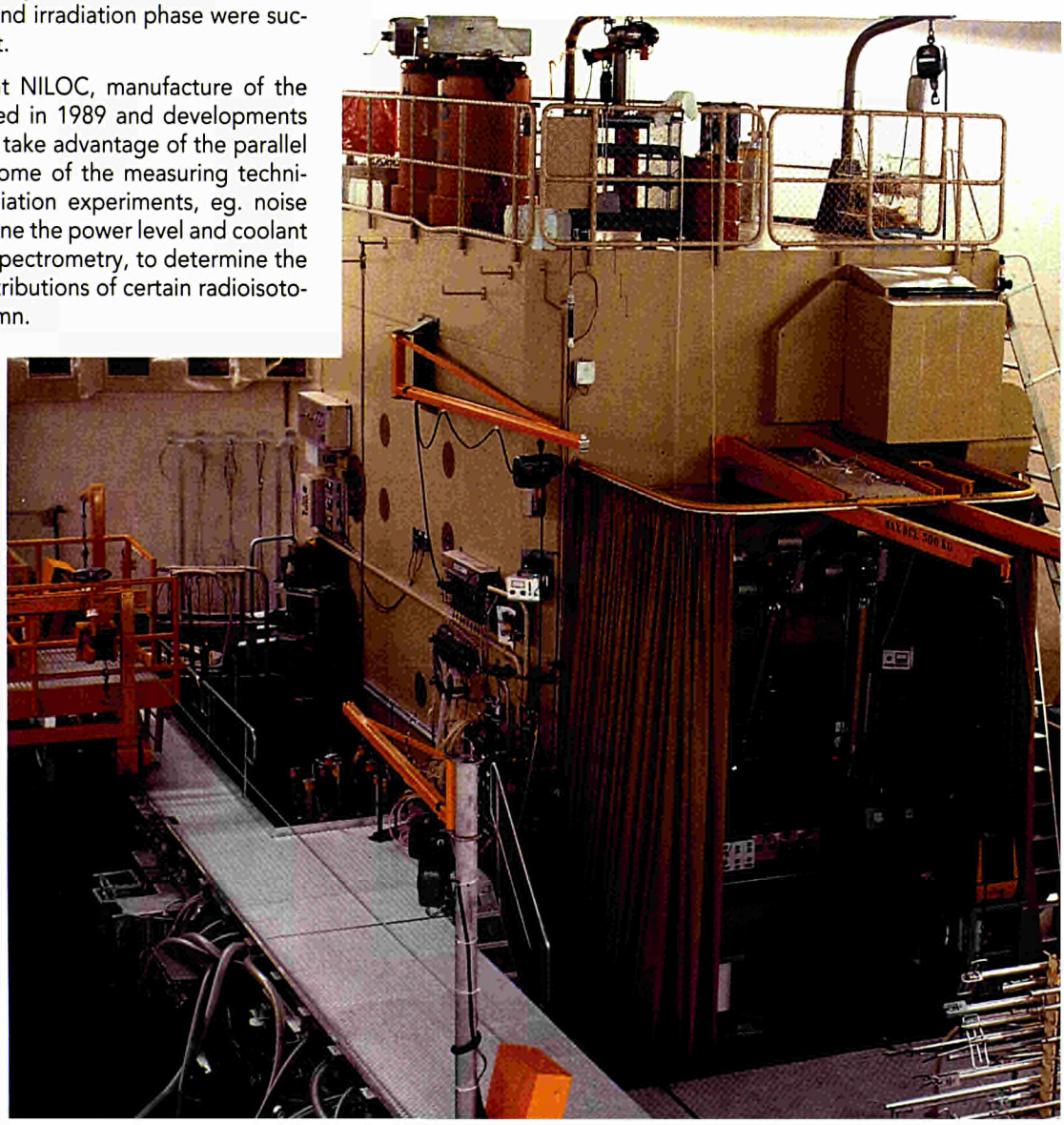
Irradiation Experiments in the High Flux Reactor

The development and improvement of the equipment and installations used in the irradiation of Advanced Fast Breeder Reactor Fuels, as sponsored by the Joint Research Centre at Karlsruhe continued throughout the year. During this time, the experiment BUMMEL completed its planned irradiation and the manufacture of the components for the coming experiments on mixed (U,Pu) nitride, POMPEI and NILOC, continued.

The experiment BUMMEL, which studies the formation and behaviour of fission a gas bubbles in UO_2, consisted of two short fuel pins of UO_2 pellets. It was required that prior to a second period of irradiation which would last just 2 hours, both fuel pins had to undergo heat treatment for 3 hours at 1300^0C. This necessitated the design of a specially built oven which, due to the fuel pin radioactivity, would need to be operated in the HFR hot cell (figure below). In July, both the heat treatment stage and the second irradiation phase were successfully carried out.

For the experiment NILOC, manufacture of the fuel capsules started in 1989 and developments are being made to take advantage of the parallel developments in some of the measuring techniques used in irradiation experiments, eg. noise analysis, to determine the power level and coolant flow, and gamma spectrometry, to determine the radial and axial distributions of certain radioisotopes in the fuel column.

Below: *The HFR hot cell, where the BUMMEL fuel pins were heat treated*



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 concentrate on studies of the behaviour under transient irradiation conditions of fuel rod segments pre-irradiated in commercial power reactors of both the BWR and the PWR line. In 1989 24 experiments were finalized by the post-irradiation examinations and final reporting. Considerable effort was put into the development of a "low power" boiling water fuel capsule, which is of high importance for new test objectives in the near future. Major progress was achieved with the development of an experimental set-up to study iodine solubility and degassing after a LOCA-scenario. The equipment was commissioned and out-of-pile testing was finalized. The experimental set-up was transferred to the HFR and the first pre-conditioning irradiation has been started.

Irradiation programmes related to the high temperature gas cooled reactor comprise graphite and fuel irradiations. The "fundamental properties graphite programme" which contributes largely to the data base on irradiation effects on graphite, progressed to schedule. Thirteen individual irradiations are under evaluation, in the reactor or under preparation. Within the graphite creep programme two irradiations were performed with their intermittent measurements of dimensional changes. A third experiment is under preparation. A long term temperature control test of spherical fuel elements was finished, evaluated and reported. A noteworthy achievement was the successful finalization of an irradiation experiment on fuel rods containing spherical coated fuel particles after 20 HFR cycles comprising nearly 450 full power days. The sophisticated tests specifications included temperature variations and the addition of pre-specified amounts of hydrogen to the sweep gas. Significant results on the effect of fuel hydrolysis on fission gas release were obtained.

Fast breeder reactor fuel irradiations were continued with an overpower equilibrium test and the conditioning irradiation for further fuel and cladding axial displacement experiments.

Considerable effort was put into the development of future SUPER-KAKADU and HYPER-KAKADU experiments which study fuel performance under power cycling conditions. Noise analysis techniques are being developed for application to fuel to cladding gap heat conductance measurements during transients.

Fusion related investigations comprised the irradiation of prospective constituant materials for super conducting magnets, investigation of tritium release kinetics from different ceramic lithium compounds under irradiation, and damage studies on different ceramics which are considered as candidate materials for the first wall protection of NET. A new objective is the irradiation of different nuclear graphites which are under discussion as alternatives for the first wall protection and other applications in NET.
For this programme the irradiation devices were developed, manufactured and assembled.

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

The FBR 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 are irradiated at different temperature and fluence levels. A rather extended programme to study fracture mechanics properties of irradiated steels, mainly compact tension specimens, at lower temperatures is under preparation.
In collaboration with other European research centres ECN is also contributing to the irradiation testing of lithium containing ceramics.
The main objective is to obtain data on tritium residence times of a variety of different zirconates, aluminates and silicates at different irradiation temperatures.

Three beam tubes have been used for nuclear physics experiments. At HB2 a polarized deuterium target is used to study details of the cross section for the reaction D(n,γ) from which information on the mesonic exchange currents can be obtained.

At HB7 the circular polarization of the gammas emitted in this same reaction is studied. The beam of HB11 which has been equipped with a system of curved mirrors, is used to study radiative capture of neutrons in He3.

Five beam tubes - HB1, 3, 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 parenthesis:

- 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.

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

References

/1/ *Annual Progress Report 1989, Operation of the High Flux Reactor EUR 12881 EN/1990*

7. S/T Support to the Service 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 pre-standards and the execution of research and development within European standardisation activities.
The concept for the development of standards for advanced ceramics at a European level resulted from recommendations of a Workshop on:
"Pre-Normative R & D for Advanced Ceramics", at JRC Petten in 1987. The workshop participants representing European industrial and research interests, invited the JRC to initiate a number of actions pertinent to standardisation and pre-standardization of advanced ceramics, on a European basis.
JRC Petten in cooperation with the Directorate General "Internal Market and Industrial Affairs" section: Standardization and Certification, initiated a review of the current European situation by supporting two actions:

- the commissioning of CEN to prepare an inventory among European Standard Organisations concerning needs and priorities for European Standards in the field of advanced ceramics;
- the organisation of a "Round Table" discussion which should
  * provide a European Forum for the presentation and discussion of up-to-date information regarding European standards for advanced ceramics;
  * present the Community infrastructure for standardisation;
  * review on-going activities in Europe, Japan and U.S., and in international co-operation schemes.

The Round Table discussion was addressed by a number of invited speakers representing the viewpoint of the major sectors of interest for establishing European Ceramic Standards including the European Commission, the European Standards Organisation: Comité Européen pour Normalisation (CEN) and the counterpart for the electrical/electronics industry (CENELEC) and European industry for both producers and users of advanced ceramics.

The Round Table expressed opinions on three aspects:

- The present situation of dispersed activities needs coordination, both in the field of standards and pre-standards. Such reorganisation will need funding.
- With respect to sponsorship, it was recognised that the Commission currently makes a reasonable contribution to standardisation, in parallel with the approach of most Member States. Substantial industrial investment is normal practice in many countries and should be expected on a European level.
- The initial phases of a European standardisation exercise will be operating at a pre-market status and additional support will be needed.

The Round Table discussion closed with unanimous support of need for a centralised coordination of standardisation activities on the European scale.

In parallel to the initiatives for the implementation of European standardisation, JRC Petten considered actions in the field of pre-normative R & D for advanced ceramics. An Ad-hoc Committee, members representing industry and research, was set up to provide a stimulus and a forum for the promotion of pre-normative R & D, to act as an interface between existing activities in Member States and relevant organisations, to investigate all possibilities for CEC support.

The Committee recommended prior to formulation of any R & D action to inventories the international situation. A review study on Standards for Test Methods for Advanced Ceramics was performed under contract by the National Physical Laboratory, U.K.

The study reviewed the actual international position in standardised testing of "advanced" ceramics of all types on the eve of the initiation of action within CEN to produce single standards for Europe. Discussed aims, driving forces and progress in each country known to be active in developing standards. The status of individual methods of testing was assessed from which areas in the following four categories were identified:

- methods which already exist and which can be straightforwardly rewritten suitably for "advanced" ceramics;
- methods which can be readily developed from the existing scientific or technical base;

- methods for which full standardisation is feasible, but which require extensive research in order to develop adequate practices or to ensure usability by test houses and other institutions;
- methods which would be desirable, but for which the prospects for standardisation in a full scientific sense are poor. Their use at a superficial level, e.g. for quality-control purposes under very restricted conditions of validity may be possible.

Further progress on the initiation of pre-normative R & D was obtained in co-operation with the Community Bureau of Reference (BCR). Two expert meetings discussed the requirements and procedures for round robin tests for advanced ceramics in the field of high temperature mechanical testing and hot corrosion.
The development of a Classification System of Advanced Ceramics in the frame of VAMAS was supported through the conception and preparation of a workshop to be organised in 1990 by the Institute for Advanced Materials and through the preparation of a questionnaire inquiry among industries of non-VAMAS member states.
Classification of advanced ceramics is considered as the 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 scope of the workshop will be:

- the identification and assessment of the issues inherent in developing a unified classification system for advanced technical ceramics;
- establishing a building block structure featuring critical elements necessary for international use;
- establishing mechanisms and institutional links as needed and appropriate, between national and international standards bodies (AFNOR, ASTM, BSI, CEN/CENELEC, DIN, ISO, JISC and others);
- enabling further system development and refinement to meet individual national and international industrial needs.

Experimental activities in the field of prenormative R & D were focussed during 1989 on two subjects relating to the development of test methodologies, i.e. hot corrosion and mechanical properties.

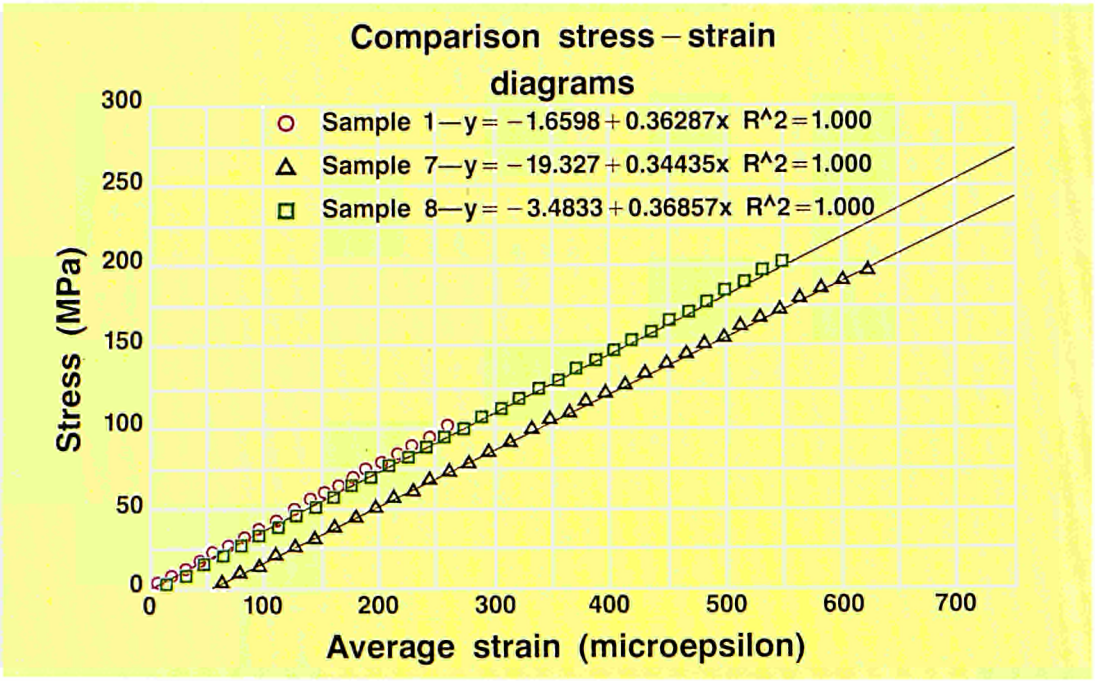
Hot corrosion:

A simple furnace test for hot corrosion of silicon-based ceramics has been developed, for predicting material degradation rates in combustion gases where sodium is the principle contaminant. The test incorporated novel methods for compensating experimental errors due to sodium evaporation.
The final report includes a "design rule" for predicting corrosion rates in practice as a function of the sodium deposition rate.
On the basis of this experience, a draft proposal for a standard test for hot corrosion of ceramics was submitted at the JRC inspired meeting at BCR to work towards standardizing hot corrosion testing of ceramics.

Tensile Testing:

The experimental activities focussed on the development of a facility for the accurate uniaxial tensile testing of both monolithic and long-fibre reinforced ceramics at high temperature. Factors such as specimen shape and dimensions, precision of machining, heating method, reliability of gripping, loading train alignment and the possibility to correct for it, which all affect the realization of a pure uniaxial tensile stress state in the specimen, have been critically assessed.

The options withheld at the current stage are flat rectangular specimens, induction heating and hydraulic gripping of specimens in the cold zone. Flat specimens are preferred for several reasons.
Often flat specimens are the only shape available with ceramic matrix composites. Moreover, a reduced sensitivity to machining flaws can be achieved by machining parallel to the loading direction.
Finally flat surfaces allow an easier optical access to the specimen for crack monitoring. Cold gripping is favoured since the alignment, which is being checked at ambient temperature, does not change when the central part of the specimen is heated to the test temperature.
The specimens are clamped hydraulically because this improves the reproducibility and thus partly eliminates the variability in test results due to differences in gripping. Also, the grips are provided with accurately machined inserts allowing a repeatable location of the specimen in the loading train.



Above: *Tensile test results on Al_2O_3 samples*

Induction heating is preferred because it offers the best compromise between access to the specimen, temperature stability, temperature homogeneity in space, thermal inertia and reliability.

Two routes are followed with regard to the alignment of the loading train: a fixed, very accurately aligned loading train, which does not allow for correction after mounting of the specimen, and a self-aligning version in which bending strains on the specimen after mounting can be corrected by adjusting the position of precisely machined knife edge supports. The first set-up is much more demanding in terms of specimen dimension tolerances and assumes very good reproducibility in specimen machining. Moreover, when the specimen shape is changed, the loading train must be completely realigned. The advantage, on the other hand, is that no friction, which is always associated with universal joints of every kind, has to be overcome and that an extension to tension-compression testing is very easy. Also because of its lateral stiffness this set-up is preferred for testing composite specimens. Currently, experiments to establish the capacity of both experimental set-ups are underway.

The following conclusions can be drawn from the work performed so far. Machining of monolithic flat specimens with a total length of 160 mm and a gauge length of 30 mm to close tolerances is possible. Thickness and width variations smaller than 2 µm can be reproducibly achieved. The main problem in machining specimens rests in assuring the parallelism of the gauge length and the main axis of the sample. Alignment control on specimens equipped with strain gauges on different cross sections has revealed that the largest contribution to the bending strain on the sample stems from the loading train and the grips when using the fixed loading train set-up. Using this set-up bending strains below 5% at stress levels of around 100 MPa can routinely be achieved. It still has to be confirmed whether a decrease of the bending strain to around 1% can be realized.

The results of room temperature tensile tests on an alumina ceramic are shown in figure above. On the basis of the size effect in terms of effective volumes (Weibull theory) and using the value of 400 MPa for the flexural strength provided by the manufacturer, a tensile strength of 237 MPa is predicted. As observed all the specimens failed at a lower stress. The low tensile strength of sample 1 is a consequence of poor alignment caused both by the loading train and by inadequate sample tolerances. This sample failed in the grip, outside the gauge length. The other two specimens reached stress levels of around 200 MPa and fractured within the gauge section.

Materials Databanks

The general objectives of this work are research and development which will assist the development of a market in information on materials used in engineering. A particular purpose is to increase customer acceptance of commercial information products in this sector.
Developments to make progress towards a European Materials Data System Network and work on data systems standards which could have worldwide impact are also included in this programme.

The main activity in the European Community on this subject over the last four years has been the Materials Database Demonstrator Programme. This Programme commenced in 1984 with an international workshop, held at Petten, which helped to determine the strategy and actions which were to be followed in the Programme. A set of ten online, computerised, materials databases were selected from four Member States and, with the HTM Databank at Petten, were used for research on the practical problems involved in the building, operation and application of such systems. During the Programme, the producers of the systems developed a Code of Practice for their operation and central systems were created for a Common Reference Vocabulary, a User Guidance System and an online directory of information sources.
For the last year there was a major effort to raise awareness and determine user opinions with seminars and workshops in every Member State of the EC. The Programme has also been independently assessed and evaluated.
Dr. N. Swindells, who coordinated the development of the Code of Practice and the subsequent stages of the Programme, joined the Institute in October as a Visiting Scientist to continue the contribution of Petten to work in this area.

The Programme has been concluded with a second international workshop, organised at Petten in December, which was attended by over 70 users and providers of materials information and representatives of public bodies and governments from 16 countries in and outside the EC, including the USA, Japan, Sweden and Finland.
Over the period of the Demonstrator Programme there has been a world wide expansion of activities in this field and the attendance at the workshop demonstrated the importance which this topic has attained. The workshop therefore reviewed the lessons learned in the Demonstrator Programme, considered these lessons in the light of what is happening elsewhere and worked out recommendations for future actions within the EC.
The conclusions of the workshop recognised the technical and commercial immaturity of the market sector and established the need for continuing the combined actions of the CEC, national governments and industries in the Member States.
The areas selected for further attention included: promotion of materials information products and their application, standardisation and integration of products and services, efforts on improving sources of data, the provision of user support agencies and international collaboration.
The group of producers of the systems which formed the core of the Demonstrator Programme have held a series of meetings to find a framework which would allow them to follow the expressed wish of the CEC that they should continue to provide a focus for development within the EC and these discussions will continue.

Arising partly from opinions expressed at the national workshops held in the Member States, work was commenced, by an external contractor to DGXIII/B, on a study of the problems involved in the substitution by local national standard materials of materials specified in the standards of another country. This topic is considered to be important for the operation of the Single Market after 1992.
The outcome of the contract was that further work to define a solution to this problem is required and this will be investigated at a meeting of a group of experts which will be held soon and coordinated from Petten.
It is expected that this activity will develop into the need for further work on both information and system development for this purpose.

Further progress on some of the other central developments of the Demonstrator Programme is still under consideration, as the lessons of the Programme and the recommendations from the Concluding Workshop are being studied, but work has commenced on research into system specification and testing to enable the Code of Practice to be strengthened and extended.

Two correlated definition studies contracted by the CEC to Cambridge University and Rolls Royce PLC have addressed the subjects of materials data interchange standardization and the integration of materials information into the computer aided engineering environment, respectively. The resultant reports include action plans broken down into specific tasks and time tables for subsequent pilot demonstrator projects. After having held some international seminars with interested partner invitations, Rolls Royce PLC have submitted an action plan for a project under ESPRIT sponsorship, while the materials data interchange study will see a follow-up with EC participation under VAMAS coordination.

It is expected that these developments towards a direct industrial involvement in the development of materials information concepts in computerized engineering and manufacturing systems will generate an increasing demand for a harmonized multilingual standard vocabulary.
The Common Reference Vocabulary (CRV) developed for the Demonstrator Programme has therefore been checked and compared with other terminology systems, and there are proposals for joint action with the therminology activities of ASTM committee E49.

Valorisation of J.R.C. Research Results: Spontaneous Downward Heat Transport

Twin plants

The purpose of the operation of the twin plants is to compare the performance of two equal solar systems, one conventional, i.e. with differential thermostat and circulation pump for heat transfer from the solar collector to the heat store, and the other fitted with the spontaneous downward heat transport system; i.e. without control devices and pump. This latter system is being tested with the double effect bi-stable float valve. Advantages of this valve with respect to the single-effect valve are: an expected higher efficiency in conditions of low irradiance or low water temperature, the capability of working also under reduced amounts of the working fluid (i.e. losses from the circuit), and in case of complete malfunction of the solar collectors, (dry out) avoidance of the build up of dangerous pressure in the circuit.

Many tests, lasting usually three or four weeks have been made, changing the operating conditions, i.e. the amount of water extracted from the heat store; the time at which the extraction is made and the temperature threshold of the water extracted. These conditions simulate the various situations met in the partical applications.

A sensible improvement over the performances of the single-effect valve has been found, although not enough yet to recover completely the advantage of the forced circulation system when the water is extracted from the heat store at low temperature ($<30^0$C). In most other cases the efficiency of the spontaneous system is higher than the forced circulation system. Due to these positive results it is foreseen to implement the double-effect bi-stable valve also in the industrial plants.

Plants for operation in mountain region

The monitoring of the plant installed at the "Rifugio Pastore" at an altitude of 1600 m has continued until the end of July. After the measurements done during summer and fall 1988 on the efficiency for heating the sanitary water, the main goal of the winter and spring tests was the determination of its behaviour as snow-melting device and its efficiency.

To that purpose the snowtank was filled every two or three days after discharge of the water produced from the previous charge of snow. This operation has been repeated 14 times. It was observed that the full thermal contact between the heating coil, at the bottom of the tank, and the snow, more or less impregnated with water, existed. The "bridging" of snow across the walls of the tank was never observed. As soon as the snow floats in the produced water, the temperature of the water at the

bottom of the tank reaches about 7-8^{0}C, i.e. the temperature at which its specific gravity is the same as that at 0^{0}C.

The overall thermal efficiency of the snow-melting process, intended as the ratio between the energy released in the snow and the incident solar energy on the collectors, was around 60%. During the following months, the system was used again for the heating of sanitary water; in these working conditions, with higher temperature of the solar collectors, the efficiency was around 40%. As this experimental run, at 1.600 m was successful, it was decided to move the plant into high mountains at 3.650 m, where the boundary conditions are clearly much harder. The results collected, for the first month of operation, confirm the previous results obtained at lower altitude. The efficiency remains unchanged because the negative effect of the lower external temperature is practically compensated by the higher irradiation values. The measured peak values of irradiance amount to 1300 w/m^2, in comparance with 1150 w/m^2 at 1600 m and to 1050 w/m^2 at Ispra level.
During the winter 89/90 the plant will remain in operation for snowmelting. The experience gained during the previous winter let us believe that the plant will operate the 89/90 winter without troubles.
For a sunny day this plant will produce about 250 dm^3 of water. All the results obtained up to now show the usefulness of the spontaneous system for the mountain applications. In order to demonstrate also its practical feasibility it has been agreed to build another plant, whose specific characteristic is that of a monobloc full unit, completely assembled and tested in the factory. A prototype, with 4 m^2 of solar collectors, is now in construction and will be put into operation in spring 1990 by the "Rifugio Omio" (Sondrio) at 2010 m of altitude. Above results show that solar energy devices can be profitably used in mountain applications. Our final aim is that industry will take the licence from the C.E.C. for the construction and commercialization of such plants in the alpine regions.

Participation to Exhibitions

In order to enlarge the number of industries interested in the system, an operational prototype was exhibited at "EURISKO 89 1er Salon Européen de l'Innovation" Paris, Jan. 89, and at "SIMAC 8th Building and Construction Exhibition" Lisbon, May 89. Particularly useful has been the participation to the Lisbon fair, where Portuguese firms expressed interest, for commercializing, building and selling the system. Negotiations for a licence agreement are conducted by DG XIII-C. Interest has also been shown by other firms from France, Australia, Israel and Uruguay.

8. Exploratory Research

Boron Neutron Capture Therapy (BNCT)

The topic of BNCT has generated a large interest throughout the medical and scientific community, especially in the last few years. Clinicians and radiotherapists agree that no single radiotherapy method is available for the treatment of all types of cancer and none that can successfully treat, for example, brain tumours. In the endeavour to be able to at least attempt to treat the different types of cancers, all current methods both in use and being developed, are being pursued. The method of boron neutron capture therapy (BNCT) appears the most promising and will initially aim to treat glioblastomas (brain tumours) and melanomas (skin cancer).

The underlying principle of BNCT is relatively straightforward. Stable nuclides with large cross sections for the absorption of slow (low energy) neutrons, eg. boron-10, are located in the tumour. The tumour is exposed to slow neutrons and the radiation emitted after neutron capture has a short range in tissue such that only the tumour cells are irradiated and destroyed. The relevant nuclear reaction for BNCT is:

$$^{10}B + {}^{1}n \rightarrow {}^{7}LI + {}^{4}He + 2.79\ MeV$$

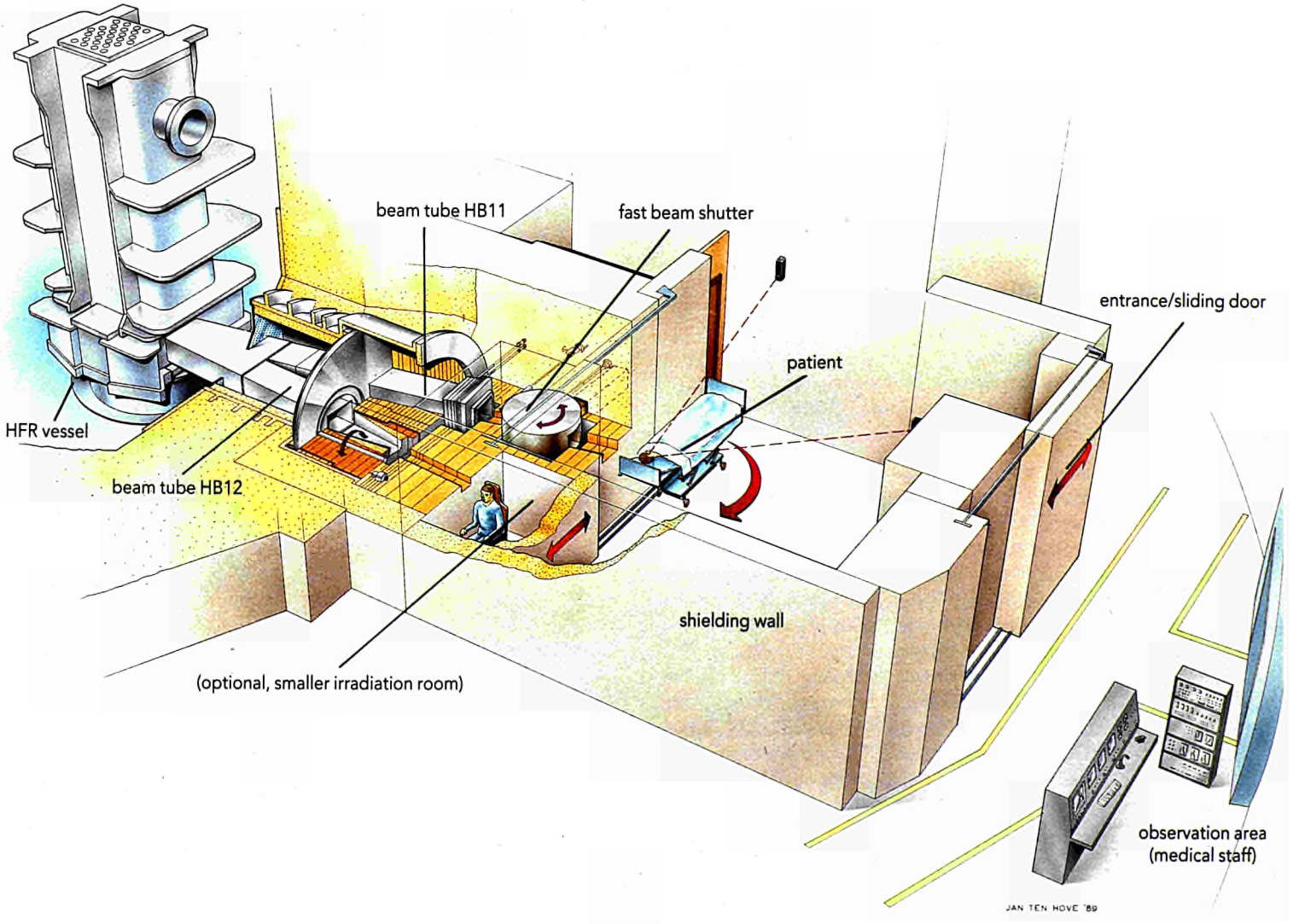
The alpha (^{4}He) particle and the lithium-7 nucleus have ranges in tissue of respectively 9 and 5 micron, which are of the same order as the diameter of a mammalian cell. Hence, heavily ionising radiation is deposited in a single cell, causing death of that cell.

During 1988, and as confirmed by calculations and measurements during 1989, it was apparent that a powerful BNCT facility could be built at the High Flux Reactor Petten. To realise this, it was necessary due to the multi-disciplinary nature of the project to collaborate closely with groups of experts throughout Europe, consisting of radiotherapists, neurosurgeons, radiobiologists, chemists, and clinical and nuclear physicists. During 1987, a local or Netherlands Petten-NCT Group was formed, consisting of JRC Petten (HFR Division), ECN Petten (Physics Department) and The Netherlands Cancer Institute, Amsterdam (Department of Radiotherapy). Within Europe, the Petten group joined the so-called European Collaboration Group on BNCT which became successful in 1989 in obtaining funding from the CEC in Brussels to form a Concerted Action on BNCT, with the prime aim to treat glioblastomas (brain tumours) at Petten before 1992. The work carried out at JRC Petten is concentrating on the design and implementation of the facility on beam tube HB11. During 1989, a rigorous campaign of design analyses using sophisticated Monte Carlo programs and measurements using a variety of neutron flux monitors were carried out on the reactor and beam tubes. These confirmed the expected results. At present, the final design work is in progress and will be completed in the beginning of 1990. In addition, work began on redesigning the present irradiation room at HB11 to create the necessary therapy room. A close-up view from a different angle of the therapy room is shown in the design on page 130.

Page 130 below: *The Petten BNCT treatment room*

During the year, the project has generated a lot of interest both locally in the Netherlands and in attracting scientists working in the field worldwide. Besides numerous experts from Europe, scientists from the USA, USSR and Australia visited Petten to discuss the BNCT project. Consequently, various articles have appeared in the Dutch national press, on the radio and television. A meeting on BNCT was organised at Petten in February 1989 and attended by over 40 people from 10 European countries. The two day meeting was the first meeting of the European Collaboration Group on BNCT in its CEC-funded capacity as a Concerted Action. In addition, various reports and presentations have been given at scientific and technical conferences in Europe and USA, and presented in scientific Journals.

The project of Boron Neutron Capture Therapy at the High Flux Reactor Petten continues and is well underway. It remains the intention to begin the first clinical trials in 1991/92.



Joining of Ceramics to Metals

Objective:

Almost all engineering applications of advanced ceramics require the development of a technology for joining these materials either to themselves or to metals. The anticipated applications very considerably and joints must be engineered to meet the specific service needs. The principal requirement for joints is that they are reliable.

This project has the aim to understand the fundamental mechanisms controlling interfacial behaviour and the influence of surface condition on joining, with the overall objective that ceramic surfaces and interfaces may be tailored to produce viable joints for high temperature applications under stress.

Methodology & Experiments:

To date the experimental work on solid state bonding and surface modification has been performed externally pending delivery and installation of bonding equipment (at Petten) and the operation of the Surface Engineering Lab (Ispra).

Surface, Interface Engineering:

A novel approach has been undertaken to tailor the key properties of the ceramic surfaces to be joined, utilising thin-film coating and ion-beam implantation technologies. Deposition of fine-grained metallic structures has been achieved by sputter or vapour deposition and prior or post (coating) ion-beam implantation treatments. Three commercial hot-pressed Si_3N_4 (SN) ceramics, are investigated. The first phase of the project will address SN/SN joining.

Joining and characterization:

Solid state bonding currently offers the greatest potential, for meeting the industrial service requirements for ceramic joints operating in high temperature structural applications. Joints will be evaluated by mechanical testing (strength, toughness, hardness etc.) and a variety of structural and chemical analyses techniques.

Results and discussion:

I. Solid State Bonding of Surface Modified SN with Metallic Interlayers.

In this main activity SN ceramics with Y_2O_3 and Al_2O_3 additives, are bonding using a Ni-Si alloy foil as an insert layer. Joining is performed by hot-pressing at temperatures between 1000 and 1350^{0}C in argon under uniaxial pressures in the range of 50 to 100 MPa. For the determined optimal joining conditions the average joint strength was large enough (>300MPa) for some industrial applications.
The effect on the bond quality of the state of the ceramic surfaces prior to joining eg. ground, polished, thermally annealed, Cr-coated and ion implanted has been investigated. By tailoring of the ceramic/metal interface the bond quality and reliability were greatly enhanced and compare very favourably with published results (table below).

Modification of the ceramic surface prior to joining	**Average strength MPa (4-pt bending, number of samples ≥9)**	**Weibull Modules**
As ground (average surface roughness, R_a = 0.3 µm)	309	2.3
Polished (average surface roughness, R_a = 0.05 µm)	365	2.65
Cr-coated (coating thickness, 1000Å)	384.5	2.93
Oxidized and Cr-coated	467	4.15
Cr-coated and ion-beam bombarded (Xe^+ ions, 400 KeV, $2x10^{16}$ ions/cm^2)	469	6.07

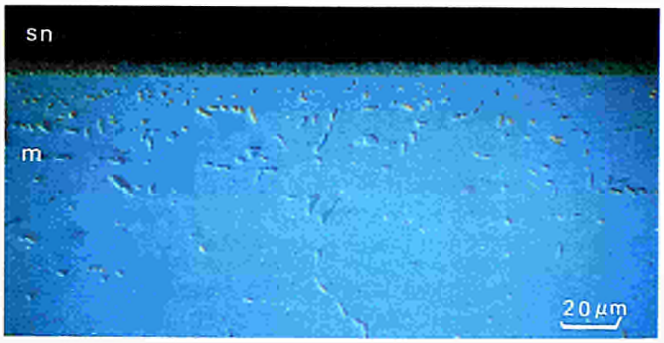
The major reaction products at the bond interface (see figure above) were shown to be Cr-nitrides; the oxidation resistance of the joints up to 1000^{0}C for 100 hrs. in air was good.

II. High Temperature Joining of SN under N Gas Pressure*.
This study extend an earlier project at GIRI Japan to develop joints using a carbonaceous film as an interlayer. Joining was carried out under systematically varied conditions of temperature, pressure time and atmosphere. The average joint strength (180-380 MPa) increased with increasing joining temperature and N gas pressure as well as with the use of protective powder bed during joining. However, joint strength is limited by degradation of the ceramic surfaces resulting from evaporation of the intergranular phases at the joining temperatures (1600-1750^0).

III. Ion Beam Mixing and Ion Implantation effects on the Mechanical Properties of SN Ceramics and SN/Metal Interfaces*.

Metallized and uncoated silicon nitride samples were implanted with inert gas and metal ions over a broad range of energy (130 Kev-2Mev) and doses ranging from 10^{15} to 10^{17}ions/cm^2. It is observed that no mixing takes place in the Ti/Si_3N_4 system but probably does in the Cr/Si_3N_4 system. Ion implantation improved the mechanical properties, i.e. surface hardness, surface fracture toughness and bulk fracture stress of the SN.

* Work performed jointly with Functional Materials Div. (IAM-Ispra), Aarhus Univ. (DK) and FOM-Institute (NL).



Above: *Microstructure of a joint interface; joining conditions: 1200^{0}C, 100 MPa, 1h, Argon (sn: silicon nitride, m: filler metal).*

Micro-Hydrodynamics of Laser Melted Pools

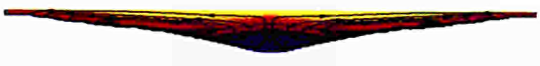
The incidence of a concentrated flux of energy onto a conducting substrate which leads to localized melting, as with laser beam striking a metal, is currently of interest in various material technologies, such as welding, in amorphous surface formation, as well as in the related problem of plasma disruptions on the first wall of fusion reactors. Here we have modeled the process and consider the contribution of Marangoni or capillary gradient convection to the steady-state molten zone configuration. In the small scale hydrodynamics involved in these phenomena and with high-surface-tension substances, Marangoni flow dominates over buoyancy flow.

This two-dimensional transient analysis refers to a model of a laser beam with Gaussian power distribution normal to a flat substance. Part of the incidence radiation is reflected, while the absorbed part raises the temperature and produces a molten pool. Because of the steep temperature distribution, the surface tension decreases or increases radially from the center depending upon the surface chemistry of the system. Classical Marangoni flow arises from the thermal or chemical capillary gradients at the fluid surface, the direction of flow being from a locality of low to one of high surface tension.

As a first step the behavior of the important stainless steel type AISI 316 is studied. This alloy is currently of interest as a candidate material for the first wall of the fusion reactor, where localized surface melting is expected to occur due to energy bursts resulting from plasma disruptions. The present alloy has been also studied for its welding behaviour and it has been demonstrated experimentally that a steel with high impurity content is associated with a deep weld pool while the converse holds for a "clean" steel. We apply the present model to test the notion that the role of sulphur is through its effect on capillarity, which in turn influences the flow hydrodynamics. In essence, we attempt through the present model to predict the resulting pool shapes from the surface thermodynamic data.
For a steel containing 140 ppm of sulphur and for a high power laser beam namely, 4kW with 20% absorption, the numerical model predicts the shape shown in the figure above. In general, a similar deep pool is predicted for all beam intensities, the fine detail of the profile depending on the actual power. For a "clean" steel, i.e. a steel having low sulphur impurities, the direction of the Marangoni flow at the surface is radially from the center to the periphery, from low to high surface tension as shown in the figure below. The surface currents transferred to the liquid bulk by shear forces now lead to a hydrodynamic flow pattern which tends to extend laterally the molten zone, leading to a broad shallow pool as represented.

Above: *Computed shape of molten pool for austenitic steel sample "B", having a high bulk impurity content of sulfur, a high level of surface-adsorbed sulfur and a positive temperature coefficient of surface tension: the streamline flow pattern, governed by surface Marangoni flow leads to a deep, narrow liquid pool.*
High-intensity beam (4 kW); pool depth 1 mm.
Temperature ranges from melting point (blue) to boiling point (yellow). Each of the 9 color shades represents a temperature step of about 125°C

Below: *Computed shape of laser-melted pool on high-purity austenitic stainless steel sample "A", having a negative temperature coefficient of surface tension: here, the direction of Marangoni surface streaming is reserved with respect to the figure above and the flow pattern leads to a shallow, flat molten pool. Laser power identical to the figure above, pool depth 0.09 mm. Color ranges as for figure above*



III Competences and Facilities

1. Skill Pools and Competence Areas

Materials Characterisations

Corrosion Properties

The group was set up towards the end of 1988 and therefore a large proportion of this year's effort was concerned with combining and rationalising the already existing separate alloys and ceramics corrosion test facilities as well as developing and extending these into new areas of competence.
The majority of the facilities is housed in a purpose-built Environmental Test Laboratory (ETL see page 162) in view of the hazardous nature of the atmospheres used.
Five vertical top-loading multispecimen autoclaves are presently operational with a temperature capability of 1100^{0}C and a capacity to expose up to 30 specimens simultaneously.
Two higher temperature rigs (1500^{0}C) of a similar design are available for studying ceramic materials whilst a further two are in the advanced stages of commissioning.
The kinetics and mechanisms of corrosive degradation are established during the interruption of the exposures.
Continuous kinetic studies are carried out in thermobalances which can operate at temperatures up to 1500^{0}C; another, with a temperature capability of 1700^{0}C, is being installed.

Two horizontal muffle furnaces have also been used in a separate laboratory for non-explosive SO_2 containing environments.
A technique has been developed whereby the conjoint action of gaseous environments coupled with synthetic and/or real ash deposits and molten salts, of the types found in F.B.C.'s, is studied.

The in-situ study of nucleation and growth processes occurring during corrosion in aggressive atmospheres is possible using a hot-stage microscope with an environmental chamber.
Structural changes are monitored using an in-built video camera.

A new laboratory containing a burner rig has been designed and the majority of the equipment ordered. This laboratory will become fully operational during 1990 and will enable dynamic tests to be carried out, complementing the simpler hot (salt) corrosion studies already possible using the conventional test rigs.

Mechanical Properties/Environment Interaction

It is common practice when designing a component to use mechanical property data and add an allowance for corrosion during the service life. This simplistic approach is adequate for some applications, but it ignores the synergisms which occur between the deformation/fracture and corrosion mechanisms. The interactions increase in importance when a design is aimed at making the most efficient use of materials or when they are being used near to their potential limit. Furthermore, the effects of creep/corrosion interactions increase in magnitude when the load, environment, temperature, time factors are in a critical range for the material concerned.

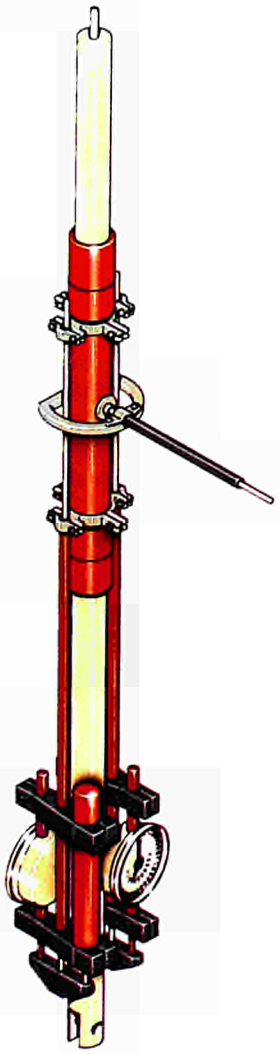
For this reason the group is concerned with the effect of creep stress or strain on corrosion and the converse effect of corrosion on creep deformation and fracture process. In total 12 uniaxial creep rigs are available for testing in air, 2 of them can be used up to 1250^{0}C and 4 up to 1100^{0}C, the remainder being limited to 1000^{0}C. For environmental testing 17 creep rigs are available, 3 of them have been modified to enable salt additions to be made during the test and 2 others can be used for variable H_2O additions. For all rigs which operate in controlled atmospheres the maximum test temperature is 1000^0 or 1050^{0}C with simple modifications. In addition to constant load uniaxial creep tests investigations are made with constant stress, or with cyclic temperature conditions. Creep crack growth (C.C.G.) is also studied. To extend knowledge about testpiece mechanical behaviour to components, advanced multiaxial testing techniques are required together with complex methods for analysing the time dependent (and time independent) stress-strain evolution in the component.

There are 4 test cells in which small tubular components can be pressurised up to 300 bar and the load maintained constant over long periods at temperatures up to 1050^{0}C with air, inert or aggressive gas.
Two of them operate with internal pressure alone, the circumferential strain being continuously monitored; the other two have in addition, facilities for axial loading, thereby allowing control over the multiaxiality.

Both axial and circumferential extensometry is provided, figure below. A number of computerised models are available for the prediction of deformation behaviour of tubular components operating under multiaxial creep stress using uniaxial data as the input. Assessment of the suitability of these models utilises the experiments as verification benchmark tests.

A test rig to enable uniaxial creep tests to be conducted in an aggressive environment with a system pressure up to 200 bar has been constructed. Techniques have also been established to allow C.C.G. to be followed in a tubular component and subsequently evaluated by fracture mechanics means. Details of these developments are given in the section: Alloys (page 23).

Below: *Longitudinal and axial extensometry on a multiaxially loaded tube*



Mechanical Properties

During the reporting period the groups' research effort stretched over the activities "Specific Programme", "Support to the Commission" and "Work for Third Parties", covering the research areas "Ceramics", "Metals and Alloys" and "Components and Thermal Fatigue".
Set up late in 1988, the group went through its starting-up phase in 1989 both in terms of launching of new projects and of geographic relocation of its equipment into the new ETL-S fatigue laboratory.

The ETL-S laboratory, built under the auspices of the infrastructural services, is a temperature and humidity controlled, state-of-art testing laboratory. Currently the laboratory houses six universal testing machines with their ancillary equipment, which were moved into the laboratory and recommissioned over the period April-June of 1989.
A further expansion with two testing machines is planned for early 1990.

The digital control of the universal testing machines and the digital acquisition and handling of test data has been a major concern of several of the group sections over the past years. In 1989 the task of conceiving and implementing an overall solution for automating the machine control and data acquisition functions of the mechanical testing laboratories was assigned.
The concept foresees in an individualized control and data acquisition per testing machine with networking to a central computer for the reduction and reporting of data.

Interfacial Engineering

In the field of "Processing of Engineering Ceramics" the group provides facilities for:

* the characterisation of powders with respect to particle size and size distribution, particle morphology, surface fotography etc.
* Green forming of powder compacts by cold isostatic pressing and slip casting, with characterisation of the rheology of slips and dispersions, viscometer, sedimentation behaviour, pH etc.
* densification by normal, low pressure sintering in controlled environments.

* densification by reaction sintering (reaction bonding - followed by low pressure sintering).
* joining of ceramics to ceramics or to metals by engineering of joint surface chemistry and/or microstructure followed by hot pressing (solid state bonding).
* characterisation of densified ceramics; measurement of density, porosity, microhardness, elastic modulus, residual stresses (e.g. in composite materials).
* machining of densified ceramics for fabrication of flat geometry test pieces and components, with in-situ measurement of triaxial grinding forces and continuous monitoring of test piece dimension.
* quantification of machining damage by measurement of rugocity, hardness, residual stresses in subsurface layers (X-ray diffractometric techniques).

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 to 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 effects, which depend on the dose rate.

   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 80^0C. 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. The changes in resistivity and the rate of change allow to develop additional information.

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 weldments. This knowledge gives the possibility to determine in a better way the behaviour of weldments 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. Weldments. Joining of metallic materials by different welding procedures in an essential part of all structures. The division has developed methods which allow to characterise weldments 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°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

Introduction

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.

Skills/Competences

Chemical sensors

The development of simple, reliable, long-lived, continuously monitoring sensors for the quantitative detection of components in gazeous atmospheres is of importance for both environmental as well as industrial purposes.
The Service has already 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 microprocessed 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, but has available for future use the Surface Modification Centre's Facilities (ion implanter, laser and electron beam heating etc.).

Facilities

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

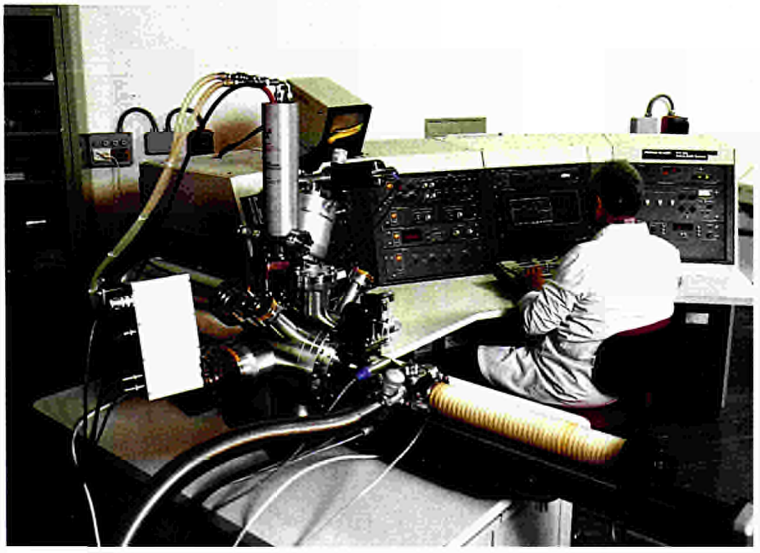
Surface and interface analytical methods.

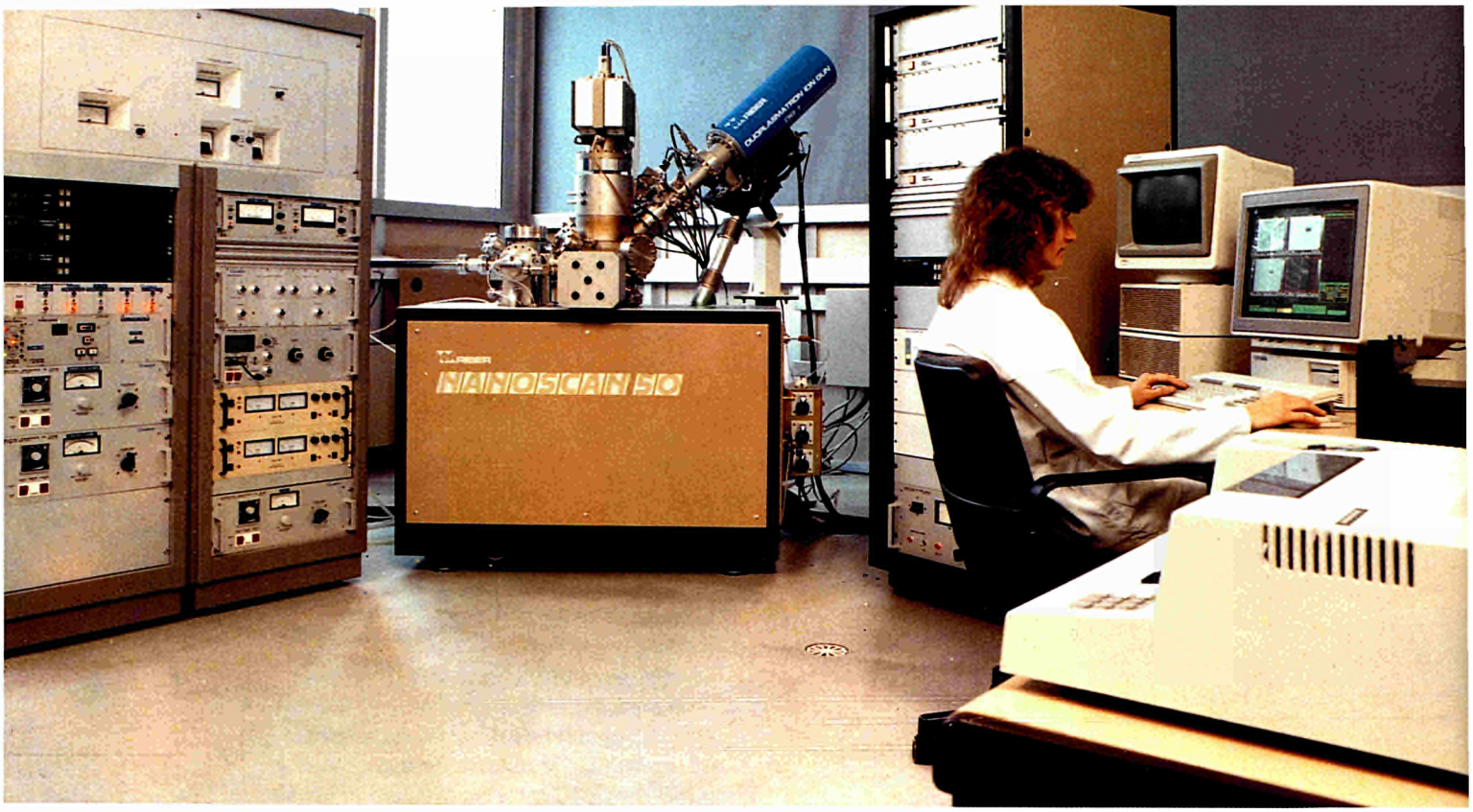
Chemical analysis

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 15uA), 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)

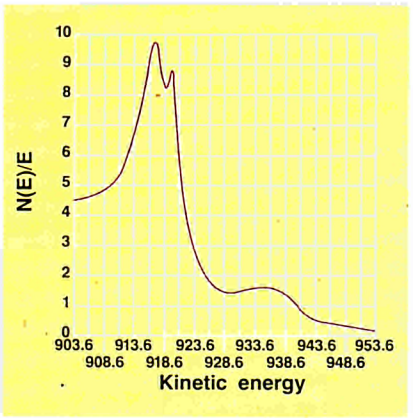
Above: *Riber Scanning Auger Microscope*

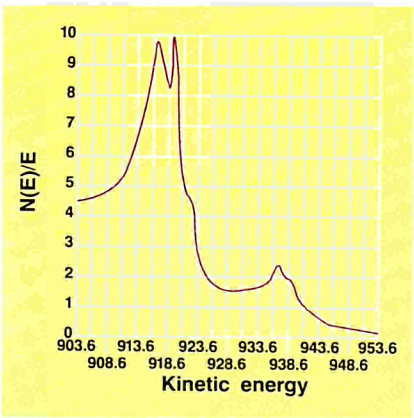
Below: *Auger LVV spectra of COPPER in the Printed CIRCUIT BOARD treated in vapour phase at 220°C for welding process*

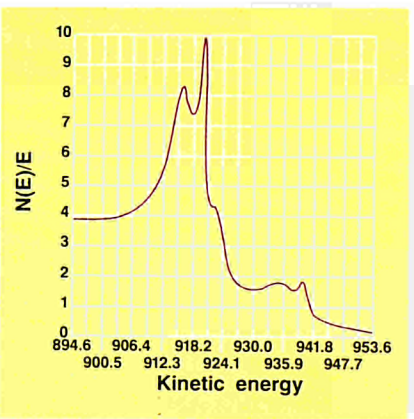
1 *Auger LVV spectrum of copper after treatment in vapour phase at 220°C after ≈ 10 Å of sputtering*

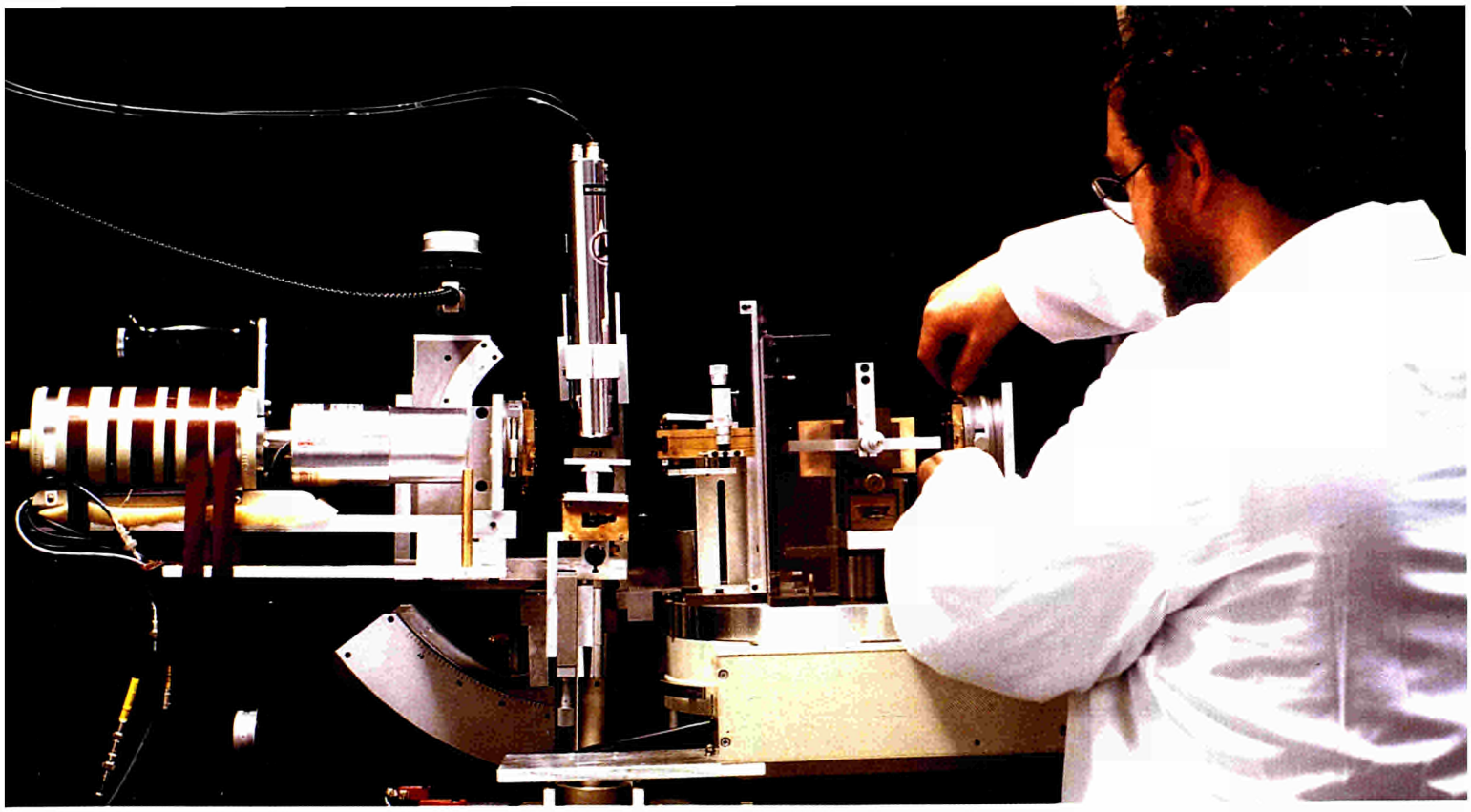
2 *Auger LVV spectrum of copper treated in vapour phase and sputtered for ≈ 20 Å*

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









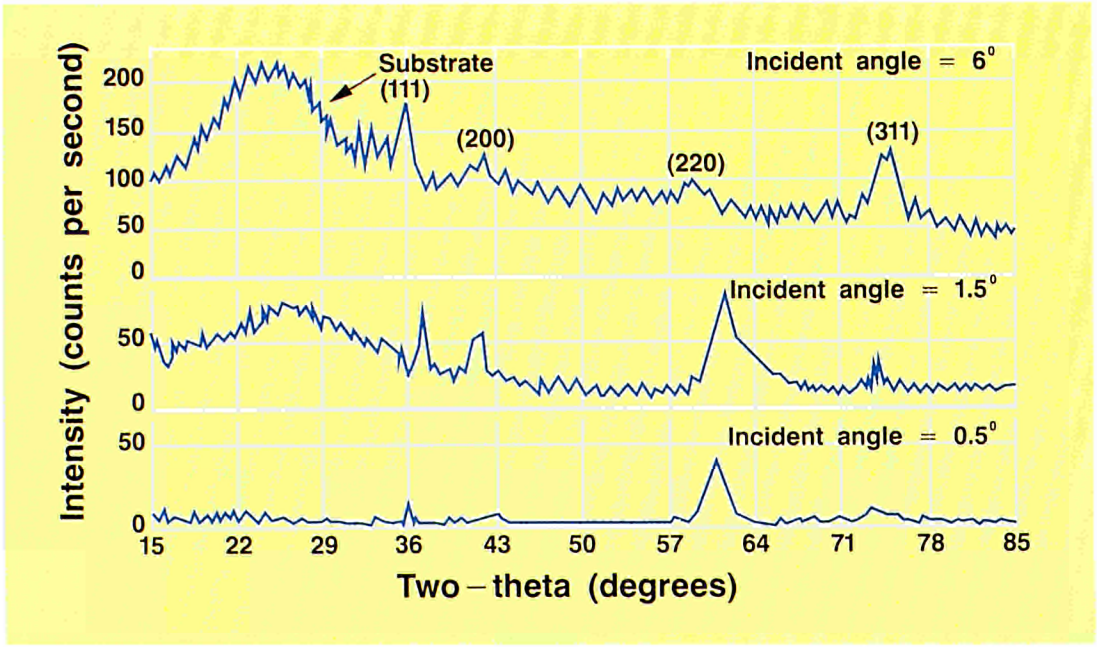
Structural analysis

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 glacing 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) XANES:

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

Access to:

- Synchroton Radiation (methods: REFLEXAFS, Glancing Angle Diffraction, Surface) - Daresbury, UK.

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.

f) A nanoindenter for measuring microhardness and Young's modulus of films in the submicron range.

Non Destructive Testing

Facilities

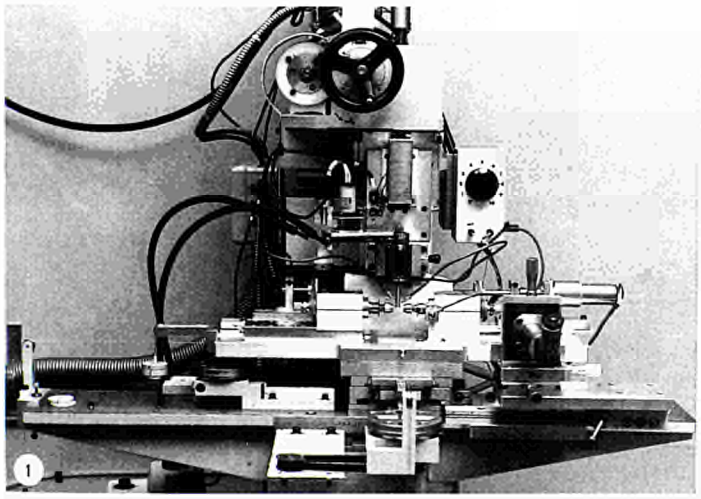
- {} Laboratory for the conception and fabrication of reference defects, using micro spark erosion, milling, punching and drilling techniques (Figure 1 and 2).
- {} Laboratory for the conception and fabrication of special ultrasonic transducers (Figure 3).
- {} Laboratory for the characterization of 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.

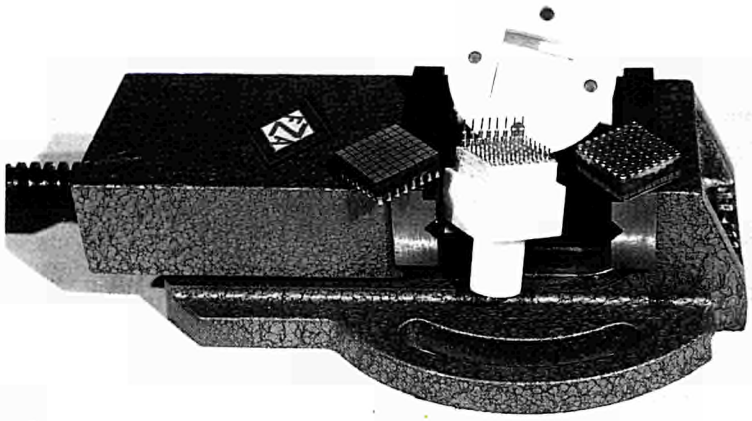
Expertises and technical services:

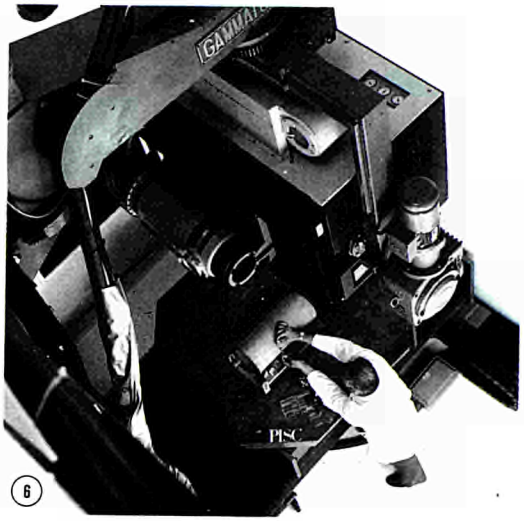
- {} Conception, fabrication and validation of reference defects (micro and macro) and of reference blocks.
- {} Conception and realization of special ultrasonic instrumentation for particular uses.
- {} Characterization of X-rays and ultrasonic instrumentation for particular applications.
- {} Studies 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.

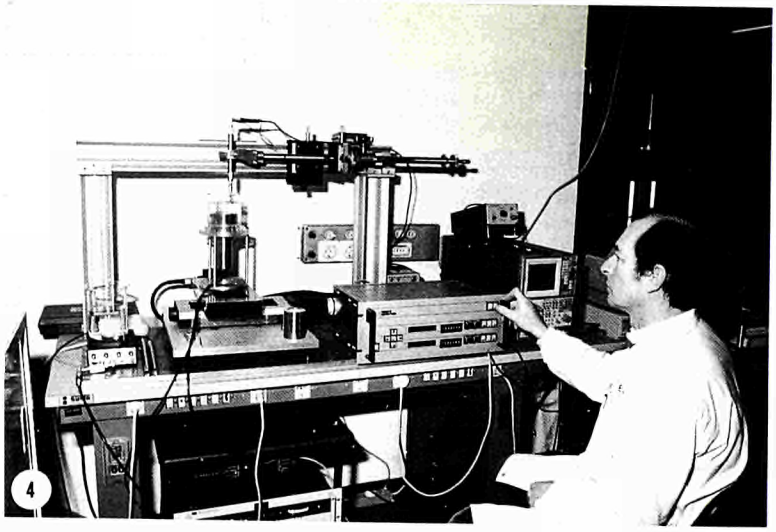
Figure 1: *Micro-spark erosion*

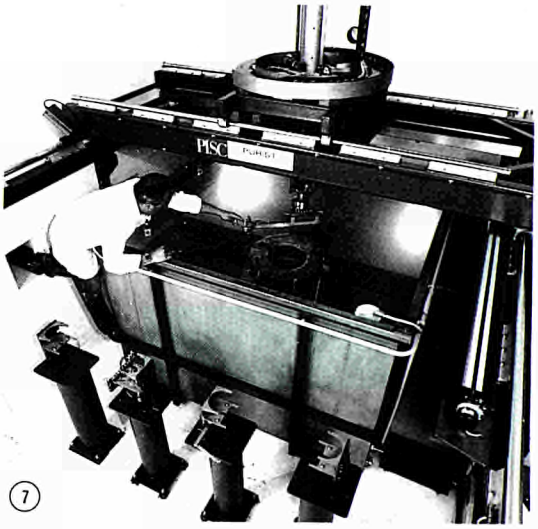
Figure 2: *Fabrication of a micro test-piece in austenic steel (diameter: 15µm, length: 10 mm)*











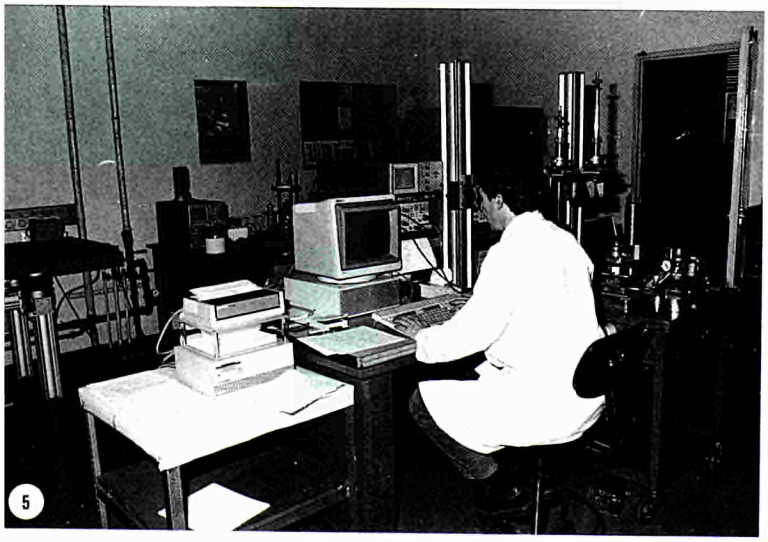


Figure 3: *Examples of a transducers array with 64 elements (NDE laboratories patent); each square element is an ultrasonic transducer, having 2.5 mm size and a nominal frequency of 5 MHz.*

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*

Materials Databanks

Materials databank development is a field of outstanding attention within the Institute for Advanced Materials. Research and development in this field are based on materials and informatics competences contributed by both the Petten and Ispra Establishments.
The joint development of online capability for a computerized materials data system has allowed the Institute to build up a unique experience on the operation of a materials databank with remote access via public networks.
This experience is highly useful in the forthcoming operation of the HTM Databank for materials data services to external online users.

The acquisition of data from published literature and laboratory records for databank input involves selection, assessment and formatting procedures which have been developed and implemented by the Institute. Using interactive PC-based menu techniques and thesaurus control, these procedures provide optimum informatics support to the most demanding task within the provision of machine-readable data.
The main effort within this area has since more than ten years been concentrated upon the development of the High Temperature Materials Databank.

The High Temperature Materials Databank

The High Temperature Materials Databank (HTM-DB) is an on-line databank of mechanical property test results for metallic materials with a unique modelling and evaluation system relevant to high temperature service.

The HTM-DB mechanical property data are related to materials used in components of conventional and nuclear power plants, steam and gas turbines. These data have been collected from open literature and publications, from tests in our own laboratories, from tests from partner laboratories and from European and world wide joint projects such as VAMAS, COST 50, COST 501, COST 505 and BRITE 1209 for which the access is restricted to collaborating parties.
The HTM-DB stores data on the following types of TEST: Creep, Creep Crack Growth, Tensile, Relaxation, Fatigue, Fatigue Crack Growth, Charpy-V Impact and Fracture Toughness.

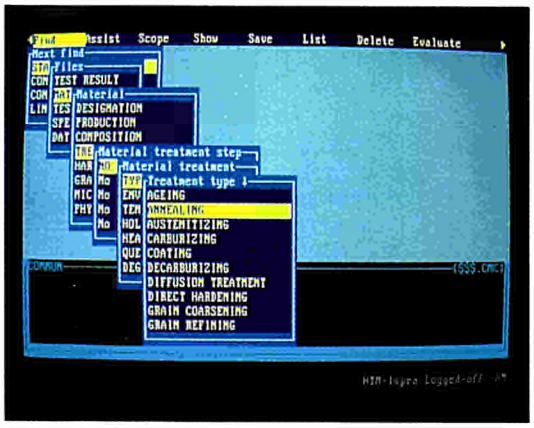
The Databank is hosted on the Amdahl mainframe of the Joint Research Centre in Ispra and accessed via the public packet switched network. Searching is menu-assisted and linked to a menu-driven application programme library which offers options for evaluation and modelling of the retrieved data. Output is obtained immediately in the form of reports, tables and graphs.

The PC-based Interface.

The data retrieval methods have been extended with a multi-purpose PC-based interface for extra user-friendliness. 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 interface has been designed in such a way that the user can store all commands and all retrieved information, both text and graphics, of a session for later use. It can be tailored to fit other databases as well, and it can be further developed as an interface to the proposed European materials databanks network.

Below: *Formulating a query by means of the PC-based interface*



Data Evaluation.

Datasets retrieved from the HTM-DB can be submitted to the modules of the Evaluation Programme Library for further elaboration. This library contains more than 70 Fortran programmes which can be selected from a menu system.

The output obtained from the evaluation programmes is a combination of reports on calculated values and colour graphics displays.

The library contains a wide variety of programmes. Some of the routines are simple spline or linear regression programmes, whereas other modules of the Evaluation Programme Library allow the calculation of constitutive equations with user guidance through the different programme steps. Most of the evaluation programmes calculate actual material parameters for design analysis. This capacity enables the HTM Databank to provide the materials input to, e.g., finite element methods. The integration of computer-based engineering analysis with computer-based materials information systems therefore is among the long-term objectives of this development.

2. Large Facilities

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 LWR fuel irradiations under steady and transient conditions the boiling water fuel capsule system has been developed. It can be operated in in-core and in pool side facility positions. The capsule can be used to irradiate fresh as well as pre-irradiated test fuel rods. A variety of instrumentation options is available, from which the following are of particular note: measurement of fuel rod length, internal pressure, central temperature, application of axial load, power control to simulate transient conditions. At the present time an advanced design for LOCA tests is under development. Future activities will concentrate on measurement of fission gas release during transient tests with re-instrumented fuel rods and on in situ profilometry on irradiated fuel rods.

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.

Irradiation testing of coated fuel particles and full pebble elements for HTR's is performed in rigs which can be operated between 600°C and 1500°C. The capsules are controlled by full instrumentation and attached to a sweep gas system for the quantitative assessment of volatile fission products. 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 Watt/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 shutdown 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.

Irradiation rigs have been developed for the study of creep of metals under constant load.

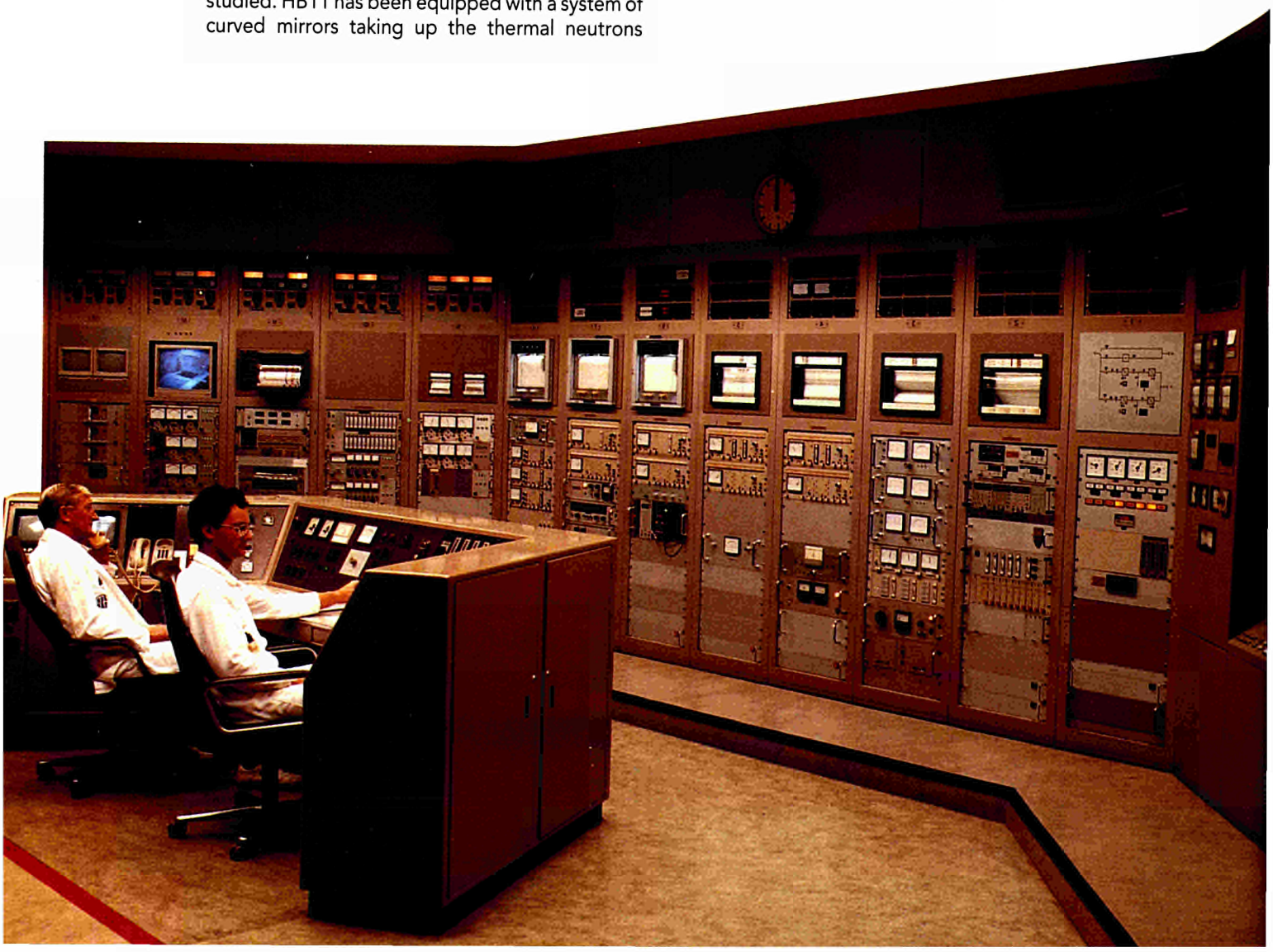
Measurement is either intermittent in-pile or after intervals of irradiation. Current applications are for first wall fusion materials where the temperature is in the range 300°C to 500°C. A device for studies on thermal fatigue combined with irradiation creep on first wall materials is under development.

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.

The present nuclear physics programme makes use of the beam tubes HB2, HB7 and HB11, which have been equipped with mirror systems to create a neutron beam in the low energy tail of the thermal spectrum. At HB2 and HB7 magnetized mirrors are being used in order to polarize the beam. At HB2 a polarized deuterium target is used. At HB7 the circular polarization of gammas can be studied. HB11 has been equipped with a system of curved mirrors taking up the thermal neutrons from the whole surface of the reactor core and focussing them on to the target area.
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 Au-198. 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.

Below: *The control room of the HFR at Petten*



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.

Neutron radiography has become a powerful tool for non-destructive testing for some high value components. Services in this field are offered jointly by JRC and ECN under the label "Petten Neutron Radiography Services".

Facilities include the submerged camera and one dry beam tube at the HFR and facilities at the Low Flux Reactor (LFR). Development activities address the introduction of electronic imaging systems including tomography for static and dynamic radiography.

A design study is being performed in preparation for setting up a clinical facility for boron neutron capture therapy at one of the large cross-section beam tubes.

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 progress 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*

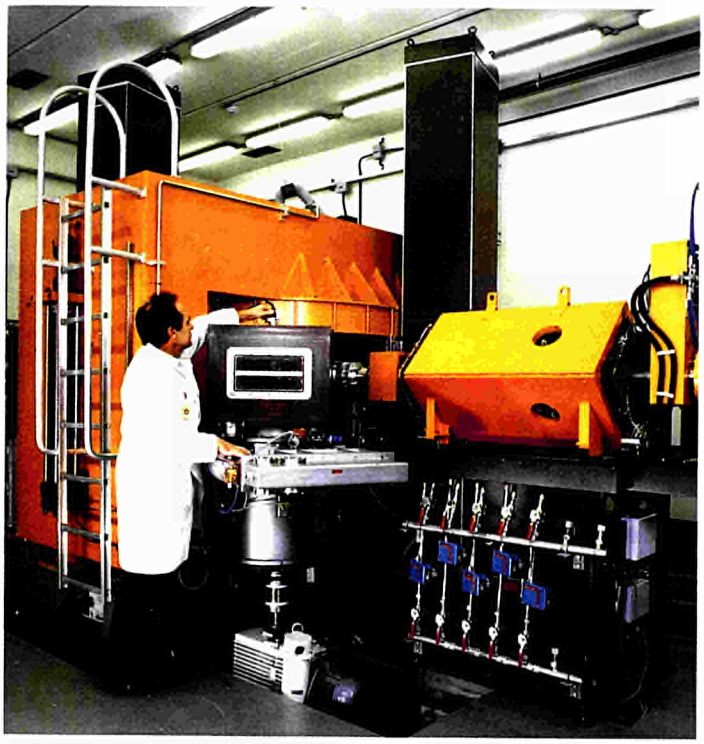
/2/ *Annual Progress Report 1989, Operation of the High Flux Reactor EUR 12881 EN/1990*

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 $\lambda/4$ 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 or indirectly by water-cooled support plates.
Temperatur measurement is 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 current 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.

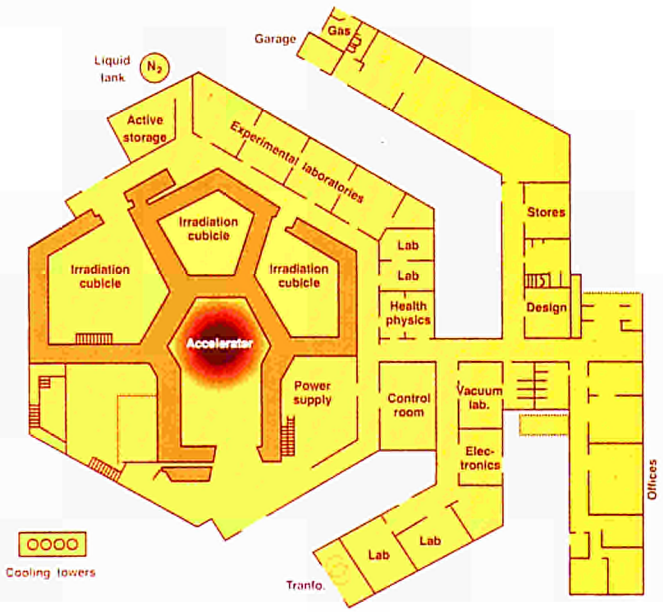
Utilisation

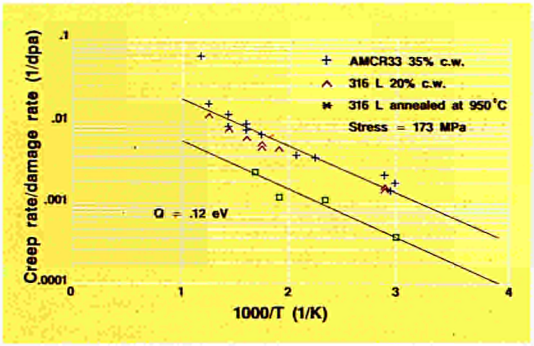
Fusion Reactor Materials:
The Ispra Cyclotron has been employed mostly as a facility for studies of radiation damage in fusion reactor materials, both as displacement and gas (hydrogen and helium) production damage. A large number of material tests in the scope of the European Fusion Technology Programme are presently under way (see also page 87).

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*

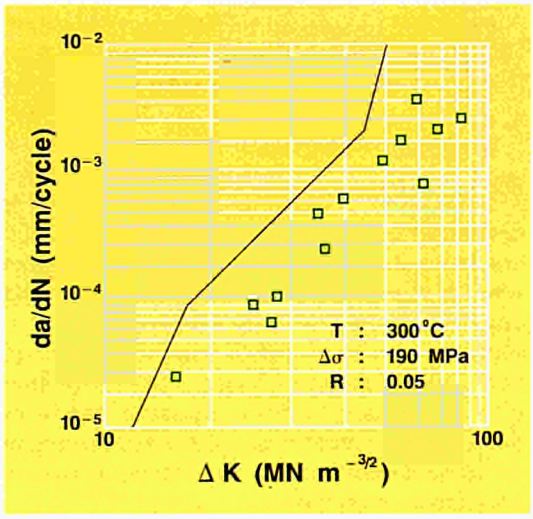


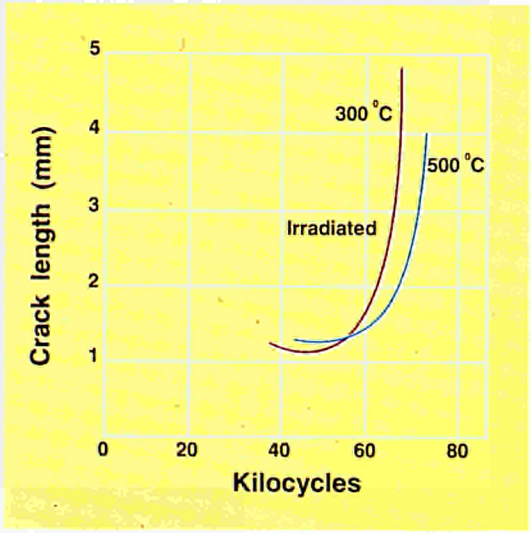


- The temperature dependence of the irradiation creep rate has been determined for type 316L stainless steel in both 20% cold worked condition and after annealing at 950^{0}C. The AMCR sample material was 35% cold worked. The results are shown in figure above which shows;
  - for all three materials 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^{0}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^{0}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^{0}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 above in the crack growth curves which shows that fatigue lives under irradiation at 300 and 500^{0}C are not very different.
  This preliminary result indicates a slightly shorter fatigue life at 300^{0}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^{0}C*





Above: *Fatigue life under irradiation at 300°C and 500°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 largets 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. As a first step we have successfully tested a production station for 67-Ga, produced from a natural zinc target. In this case a maximum current of 54 μA was used. Production of other radioisotopes of medical interest is foreseen. In particular 123-I, 201-Tl, 81-Rb, 111-In and 18-F are of principal interest and in increasing use in the European Community countries.
Medical research by proton irradiation of selected organs in laboratory animals has already been performed. In an experiment made in collaboration with a biological Institute male rats were irradiated on the cortex with 60 Gy Bragg peak protons in order to open the blood-brain-barrier, permitting the study of pathogen factors which can be responsible for epilepsy in man.

In the frame of a third party contract, 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.
In 1989, 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 are under way to check the samples for radionuclide impurities and for the relative intensity of 68-Ga.

The main development for the coming three to four years will be the set-up of radioisotope production for medical and scientific purposes.
In this context the possibility to produce high purity 123-I and 201-Tl is being studied in collaboration with radiopharmaceutical firms. 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) In preparation for a third party commitment, irradiations were carried out to determine rare earths in minerals.
3) The thin layer activation method is a powerful tool for the sensitive and precise determination of wear in lubricated mechnical components. The possibility of wear measurements in vital parts of engines in the aeronautical sector (helicopters) is being studied in cooperation with an external firm.
4) 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.

Environmental Testing Laboratory

The Environmental Testing Laboratory was conceived and constructed to enable the 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. The laboratory was designed in such a way that all experimental rigs could 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.

The test gases are supplied from batteries of premixed gas held in external gas stations and piped around the ETL. The gas compositions used in 1989 are shown, by way of example, in table below.

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.

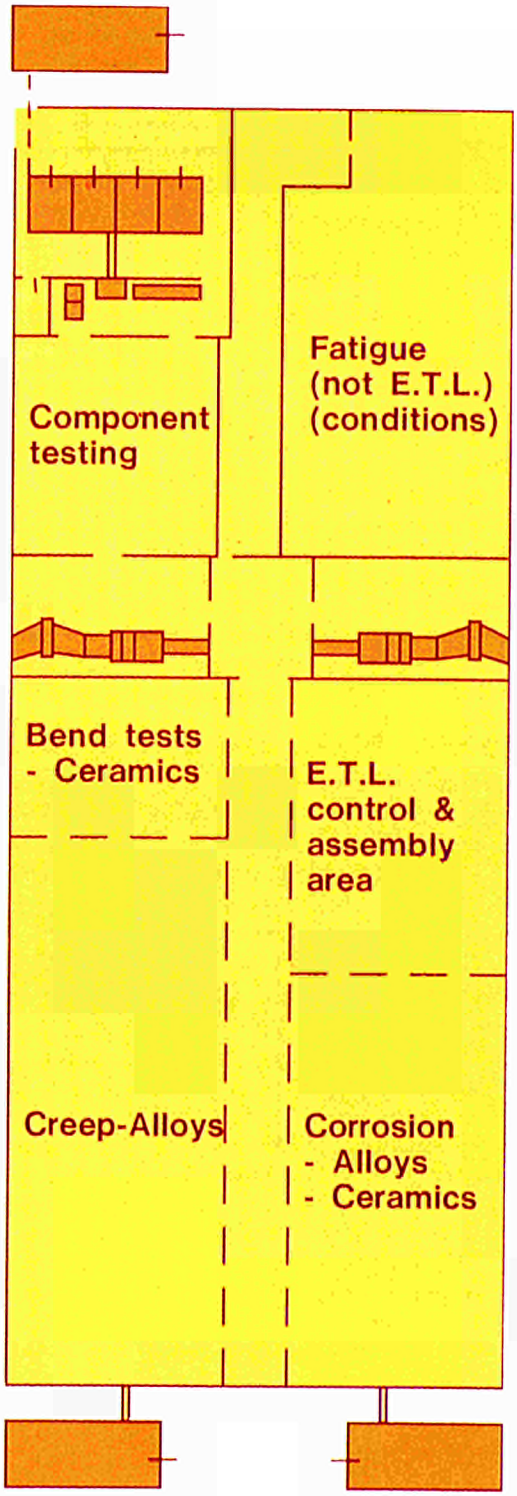
The space in the ETL is currently allocated between various test disciplines as shown in the figure right. A total of 47 test points are available which are used for work within the Specific Programme and for third-party contract research.

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. Hence it is dedicated to the work of the Component Testing sub-group and is described in more detail in chapter: Alloys.

Types of test gas used in E.T.L. during 1989

Gas mixture vol.%
2,0 H_2S + 7CO, bal. H_2
0,6 H_2S + 7CO, bal. H_2
0,4 H_2S + 7CO, bal. H_2
0,2 H_2S + 7CO, bal. H_2
0,1 H_2S + 2OH_2, bal. A
0,4 H_2S, bal. H_2
7CO , bal. H_2
205Vpm SO_2, bal. Air
99,9999A for inert gas tests
99,996A for safety blankets
Including the argon safety gas, 14 lines available for experimental use.

Below: Distribution of test areas in E.T.L. during 1989



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).

IV List of Publications

Scientific -Technical Articles and Monographs

J. Ahlf, H. Röttger;
The HFR Petten, Prospects and Future Utilization; Proceedings of a Colloquium held in Petten (NL), 20-21 April 1989, EUR 12522 (1989)

J. Ahlf, R. Conrad, M. Cundy, H. Scheurer;
Irradiation experiments on High Temperature Gas-Cooled Reactor fuels and graphite at the HFR Petten. Journal of Nuclear Materials 171 (1990). ART 28782

J. Bressers, M. Steen;
Fatigue and Microstructure in Austenitic High Temperature Alloys.
Art. in Res Mechanica - not yet published.
ART 29142

E. Bullock;
Research and Development of High Temperature Materials for Industry.
EUR 12374

E. Bullock;
European Standards for Advanced Ceramics.
Ceramic Forum International,
DKG 66, No. 7/8, 1989

R. Buschert, P. Gibson, W. Gissler, J. Haupt;
Glancing Incidence X-Ray Studies of Titanium Nitride Thin Films Using a New Multipurpose Laboratory Spectrometer. Intern. Conference on Surface and Thin Film Studies Using Glancing-Incidence X-Ray and Neutron Scattering.
CNRS. 31 May - 2 June 1989, Marseille (F),
Journal de Physique ORA/ART 34710

R. Buschert, P. Gibson, W. Gissler, J. Haupt, A. Manara, X. Jiang, K. Reichelt;
Comparative Study of TiN Films Deposited by Reactive Ion Beam Sputtering at 77 and 300 K.
ECASIA 89, Soc. Française du Vide,
23-27 October 1989, Antibes (F). Surface and Interface (Special Issue), ORA/ART 34919

V. Coen, H. Kolbe, L. Orecchia, M. Della Rossa;
Corrosion on Cr-Mn based austenitic steels by the lithium lead Pb-17Li. Fusion Engineering & Design - Special Issue on Pb-17Li in Breeding Blankets of Fusion Reactors (to be published).

G. De Beni, R. Friesen;
Impianto solare per la produzione di acqua calda di processo con trasporto spontaneo del calore verso il basso. Fase 2: risultati sperimentali.
Energie Alternative HTE, anno 11, N. 59 (1989), pp. 201-206

S. Crutzen, P. Jehenson, R. Nichols, N. McDonald;
The Major Results of the PISC II RRT.
Nuclear Eng. and Design. Vol. 115 (1989), 7.21

M. Farag, G. Piatti;
Superplastic Deformation and Cavitation of a 14Cr-20Mn Stainless Steel.
Materials Science and Engineering. ART 28572

G. Fischer, W. Goll, J.F.W. Markgraf, I. Ruyter;
Verhalten von DWR-U/Pu-Mischoxid-Brennstäben bei Leistungstransienten. KTG Jahrestagung 1989, Düsseldorf, 9-11 Mai 1989

M. Forte, C. Zeyen;
Neutron Optical Spin-Orbit Rotation in Dynamical Diffraction. Workshop on Fundamental Physics with Slow Neutrons.
ILL. 8-11 March 1989, Grenoble (F), Nuclear Instruments and Methods, ORA/ART 34646

P. Gibson;
Thin Films: Characterization with X-Rays.
Encyclopedia of Materials Science and Engineering. Supplement 2. Pergamon Press, Oxford, R.W. Cahn, W.S. Owen, M.B. Bever (Eds.), ART 28577

V. Guttmann;
The Influence of Sulphidation and Carburisation upon Creep. Art in Materials Science and Technology - not yet published. ART 28871

V. Guttmann, J. Timm;
On the Influence Thermal Pretreatment of Alloy 800H. Art. Zeitschrift für Metallkunde - not yet published. ART 29104

R.G. Hale, J.F. Norton;
CAD System brings a New Dimension to High Temperature Corrosion Research and Development Studies. Werkstoffe und Korrosion, 1989, vol. 40, pp. 193-197. ART 28094

E.D. Hondros, E. Bullock;
Materials for the Next Millenium. Angewandte Chemie (Advanced Materials), no. 8 (1989), p. 260.

E.D. Hondros, H. Kröckel;
Computerized Representation of Data and Knowledge on High Temperature Corrosion and Corrosion Control. Materials Science and Engineering, A121 (1989), pp. 685-691

T.D.A. Kennedy, J.F.W. Markgraf, S. McAllister, I. Ruyter;
Development of a Two-dimensional Computer Code for the Prediction of Two Phase Heat Transfer in Experimental Light Water Reactor Irradiation Capsule. To be published in The Journal of the British Nuclear Energy Society, DG XIII reference ART 29142

H. Kröckel;
Advanced Materials Data Systems for Engineering Scientific and Technical Data in a New Era, CODATA, 1989

H. Kwast, R. Conrad, S. Preston;
EXOTIC Annual Progress Report 1988, ECN-224 (1989)

E. Lang (Ed.);
The Role of Active Elements in the Oxidation Behaviour of High Temperature Metals and Alloys: Proceedings of the European Colloquium, Petten, 12-13 December 1988 - EUR 12327, Elsevier Applied Science, London 1989

E. Lang, V. Lupinc, A. Marucco;
The Effect of Thermo-mechanical Treatments on Short-range Ordering and Secondary Phase Precipitation in Ni/Cr based Alloy. Article in Materials Science and Engineering - A114 (1989) 147-157. ART 28378

F. Lanza;
High-level radioactive waste disposal: Safety Aspects. Supplement 2 of the Encyclopedia of Materials Science and Engineering, Pergamon Press

A. Lucia, M. Franchi, C.A. Marozzi, R. Fontana;
Three-Dimensional Strain Field Measurement on Cylindrical Vessel by Computer Analysis of Laser Interferograms. Experimental Mechanics, June 1989, 132-137. ART 28054

A. Manara, V. Sirtori;
Surface Investigation of Copper in the Printed Circuit Board. ECASIA 89. Soc. Française du Vide, 23-27 October 1989, Antibes (F). Surface and Interface Analysis (special issue). ORA/ART 34745

L. Manes, F. Lanza, V. Dallacasa, R. Feduzi;
Interpretation of Tc Dependence on Carrier Density of High Temperature Superconductors by a Generalized BCS Theory. Physical Review Letters. ART 29125

J.B. Marriott;
Future Materials Requirements for High Temperature Power Engineering Components. Art. in High Temperature Technology - Vol. 7, no. 3, August 1989, pp. 129-135. ART 28640

A.S. McAllister, T.E. Chung, R.C. Hurst;
Modelling of the multiaxial creep behaviour of Alloy 800H; Accepted for publication in Res Mechanica, 1989. ART 28419

J. Mukhopadhyay, G. Piatti, A. Mukherjee;
Extended Ductility and Cavitation in a High Manganese Stainless Steel.
Journal of Materials Science, Vol. 25 (1990), pp. 781-788. ART 28298

M. Nakamura, S.D. Peteves;
Joining of Silicon Nitride Ceramics under Nitrogen Gas Pressure.
J. Mat. Science Letters, 8 (1989), pp. 1228-30.

M. Nakamura, S.D. Peteves;
Solid State Bonding of Silicon Nitride Ceramics with Ni-Cr Alloy Interlayers. J. Amer. Ceramic Society, October 1989, ART 29130

C. Olagnon, D. McGarry, E. Nagy;
The Effect of Slip Casting Parameters on the Sintering and Final Properties of Si_3N_4.
British Ceramics Trans. J. 88 (1989), 75-78.
ART 28379

K. Ostijn, S.D. Peteves, A.C. Vinckier;
Joining of Advanced Ceramics for High Temperature Applications, in: Research and Development of High Temperature Materials for Industry. Elsevier Applied Science, p. 307, 1989

S.D. Peteves;
Designing Interfaces for Technological Applications, Ceramic-Ceramic, Ceramic Metal Joining: Proceedings of the European Colloquium, Petten, 20-21 April 1988. Elsevier Applied Science, London, 1989, EUR 12148

S.D. Peteves;
Joining of Silicon Nitride Ceramics under Nitrogen Gas Pressure. J. of Materials Science Letters, 8 (1989) 1228. ART 28560

S.D. Peteves, P. Tambuyser, P. Helbach;
Microstructure and Microchemistry of the Al/SiC Interface. Art. in Materials Science
- not yet published. ART 29131

G. Piatti, M. Vedani;
Relation between Tensile Properties and Microstructure in Type 316 Stainless Steel SA Weld Metal,
Journal of Materials Science, ART 28652

D. Quataert, F. Brossa, P. Schiller;
Melting and conduction during short electron beam pulses. Fusion Engineering and Design

M.D. Rogers, J. Jovicevic;
Proceedings of the Joint Colloquium on Advanced Metallic and Ceramic Materials, 12-14 September, Sarajevo, Yugoslavia - EUR 12274

P. Schiller, S. Ishino, A.F. Rowcliffe;
Needs and Requirements for a Neutron Irradiation Facility for Fusion Materials Testing, to be published on J. of Fusion Energy

G. Solomos, A. Lucia;
Markov Approximation to Fatigue Crack Size Distribution. Fatigue & Fracture of Engineering Materials & Structures, ART 28575

M.F. Stroosnijder, J.F. Norton, V. Guttmann, M.J. Bennett, J.H.W. de Wit;
Effect of cerium implantation on the corrosion of Alloy 800H in a S-O-C environment. Materials Science and Engineering, A116(1989), pp.103-110

M.F. Stroosnijder, V. Guttmann, T. Fransen, J.H.W. de Wit;
Corrosion of Alloy 800H and the Effect of Surface-applied CeO_2 in a Sulphidizing/Oxidizing/Carburizing Environment at 700 ^{0}C.
Not yet published, ART 29003

G.P. Tartaglia, H. Kröckel, H.H. Over;
The Integration of a FEM Code with a Materials Properties Databank. Art in Journal of Nuclear Materials. Not yet published. ART 28780

G.P. Tartaglia;
Irradiation Facilities for Thermal Fatigue and Biaxial Creep Experiments.
Art. in Journal of Nuclear Materials.
Not yet published. ART 28781

G. Tsotridis, H. Rother, E.D. Hondros;
Marangoni Flow and the Shapes of Laser Melted Pools. Art. in Naturwissenschaften.
Not yet published. ART 28508

G. Walz, K. Hornberger, H. Stamm;
An Implicit Integration Algorithm for Plane Stress Viscoplastic Constitutive Equations.
Computers & Structures. ART 29070

Technical EUR Reports

J. Ahlf, H. Röttger;
Annual Progress Report 1988.
Operation of the High Flux Reactor.
EUR 12271 EN (1989)

H. Arenz, R. Buschert, P. Gibson, W. Gissler, J. Haupt, A. Manara;
Ion Beam Mixing and Characterisation of a Copper/Silver Three-Layer System.
CEC, EUR 12192 EN (1989)

J.H. Baard, W.L. Zijp, H.J. Nolthenius;
Nuclear Data Guide for Reactor Neutron Metrology. EUR 12354 EN

G. Barbi;
Solid State Electric Gas Sensors.
CEC, EUR 12373 EN (1989)

E. Borloo, F. Daniele, P. Jehenson, R. van den Bergh, F. Violin;
The ATFI facilities for the PISC III programme.
PISC III Report No. 2.
EUR Report No. (not yet defined). November 1989

E. Bullock;
Annual Progress Report 1988.
High Temperatures Materials. EUR 12272 EN

M. Certo, T. Cola, G. Mariani;
Studi parametrici dell'effetto delle caratteristiche dei difetti sulla loro rivelazione e dimensionamento mediante ultrasuoni. CISE, PISC III Report No. 5, OECD-CEC, EUR 12432 EN (1989)

J.C. Domanus;
Neutron Radiography Working Group:
Test Programme. EUR 12121

Ph. Dombret;
PISC Parametric Study on the Effect of Defect Characteristics on Immersion focussing Probe Testing Results. Vinçotte, PISC III Report No. 6, OECD-CEC, EUR 12433 EN (1989)

E.D. Hondros;
Institute of Advanced Materials - and the Materials Challenge. EUR 12275

E. Lang, B. Hudson, A.T. Tuson, M.J. Bennett;
Thermal Fatigue Damage of Fecralloy Co-extruded Tubing. EUR 11967 EN, 1989

R. Murgatroyd, P. Highmore, S. Burch, T. Bann, A. Ramsey;
Flow characterisation using the tandem and tofd techniques. UKAEA, RNL, PISC III No. 4, EUR 12431 EN (1989)

W. Schule;
Considerations on the validity of the various radiation damage models.
To be published as EUR report.

Contributions to Conferences

F. Andritsos;
Non-linear Model of the Central Solenoid of a High Field Tokamak. First Italian ABAQUS Users Group Conference. Italian ABAQUS Users Group.
18-19 December 1989, Bologna (I).
Proceedings of the Conference. ORA/PRO 35254

J. Bakker, A. Baritello, M. Beers, J. Bordo, R. Conrad, H. Hausen, H.P. Leeflang, J.F.W. Markgraf, R. Moss;
Neutron Radiography of HFR Irradiation Devices. Colloquium of the HFR Petten, Prospects and Future Utilization, Petten, April 20-21, 1989.
Proceedings EUR 12522, page 355 (poster)

N.J. Barrett, D. Cunningham, M. Dos Santos, R.A.H. Edwards, E.N. Gibson, A. Manara, K.J. Roberts;
Characterizing of sulphidized steel surfaces using sychrotron radiation.
Poster presented at Conference on XSR, Rome (I), October 1989 (to be published in "Il nuovo cinvento")

C. Birac (CEA), P. van Leeuwen (RTD), Y. Miyake (Mitsubishi), G. Maciga (ENEL), H. Herkenrath (JRC Ispra);
Steam Generator tubes and defects used for the PISC III RRT. Paper presented at the Seminar on Reactor Safety Research, November 20-24, 1989 Varese (I)

E.J. Bleeker, H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
Experience with the HB-8 facility at the HFR Petten for thermal and subthermal neutron radiography. The Third World Conference on Neutron Radiography, Osaka, May 14-18, 1989

D. Bolsch, J. Bressers;
High Temperature Uniaxial Alternating Fatigue Testing of Engineering Ceramics. First European Ceramic Society Conference, 18-23 June 1989, Maastricht, The Netherlands

E. Borloo;
PISC Round Robin Test on the Measurement of UT Instrument and Transducer Characteristics (MITC). Presented at the 12th World Conference on Non-Destructive Testing, Amsterdam, April 23-28, 1989

E. Borloo, H. Herkenrath;
Full Scale Vessel Components Testing in PISC III. Pt. 2: Nozzles and Dissimilar Metal Welds (NDW) action. Paper presented at the Seminar on Reactor Safety Research, November 20-24, 1989, Varese (I)

F. Brossa, R. Pietra, E. Sabbioni, M. Gallorini;
CVD Coatings to prevent Metal Release in Medical Applications; 7eme Conference Europeenne sur les Depots Chimiques à partir d'une Phase Gazeuse. Univ. Perpignan, 19-23 June 1989, Perpignan (F).
Proceedings of the Conference ORA/PRO 34094

R. Buschert, P. Gibson, W. Gissler, J. Haupt;
Glancing Incidence X-Ray Studies of Titanium Nitride Thin Films Using a New Multipurpose Laboratory Spectrometer. Intern. Conference on Surface and Thin Film Studies Using Glancing-Incidence X-Ray and Neutron Scattering.
CNRS, 31 May - 2 June 1989, Marseille (F),
Journal de Physique, ORA/ART 34710

R. Buschert, P. Gibson, W. Gissler, J. Haupt, A. Manara, X. Jiang, K. Reichelt;
Properties of Ion Beam Sputtered Titanium Nitride Films (Poster Presentation). In: Proceedings of the Intern. Congress SURTEC '89, AMK,
11-13 October 1989, Berlin (FRG).
Oberflächentechnik, Carl Hauser Verlag, Wien,
H. Czichos, L.G.E. Vollrath (Eds.), ORA/PRO 35059

M. Cambini, S. Crutzen, X. Edelmann, P. Jehenson, R. van den Bergh;
Progress Report on Real Reactor Pieces Testing. Paper presented at the Seminar on Reactor Safety Research, November 20-24, 1989, Varese (I)

P. Clerici, P. Davoli, G. Piatti;
Fatigue Behaviour of a High-Mn Stainless Steel for Thermonuclear Fusion Purposes.
Intern. Conference on Evolution of Structural Materials. AIM, 31 May - 2 June 1989, Milano (I), Proceedings of the Conference (1989), pp. 281-288, ORA/PRO 34767

V. Coen, H. Kolbe, L. Orecchia, M. Della Rossa;
High Temperature Compatibility of Ceramics with the Lithium Lead Eutectic Pb-17Li.
Oral Presentation at the Workshop on High Temperature Corrosion of Technical Ceramics, JRC Petten, June 1989

R. Conrad, L. Debarberis;
Irradiation Facilities for Testing Solid and Liquid Blanket Breeder Materials In-Situ Tritium Release Measurements in the HFR Petten.
ICFRM-4, Kyoto, 4-8 December 1989, ORA 35039

R. Conrad, L. Debarberis, V. Coen;
Irradiation of Liquid Breeder Material Pb-17Li with In-Situ Tritium Release Measurements in LIBRETTO 1 and 2 Experiments.
ICFRM-4, Kyoto, 4-8 December 1989, ORA 35040

F. Costa Oliveira, R.A.H. Edwards, R.J. Fordham, F.L. Riley;
High Temperature Corrosion of Si_3N_4-Y_2O_3-Al_2O_3 ceramics in $H_2S/H_2O/H_2$ reducing environments. Paper presented at European Colloquium on High Temperature Corrosion of Technical Ceramics, Petten (NL), June 1989 (to be published in proceedings)

F. Costa Oliveira;
Corrosion of some Silicon Nitrides in $H_2/H_2O/H_2S$ Gas Mixtures at 1200-1300 ^{0}C. First European Ceramic Society Conference, Maastricht, 18-23 June 1989, ORA 34629

S. Crutzen, M. Certo, Ph. Dombret, R. Murgatroyd;
Summary of the PISC Parametric Studies of the Effect of Defect Characteristics (EDC).
Paper presented at the International Conference, NDE in the Nuclear Industry, 9th International Conference ASM, Tokyo, April 25-28, 1989.
PISC III Report No. 8, OECD-CEC, EUR 12435 EN (1989)

S. Crutzen, P. Jehenson, H. Herkenrath;
Effectiveness of NDE in relation to Structural Integrity. Paper presented at the International Conference on Structural Failure, Product Reliability and Technical Insurance, Vienna, July 10-12, 1989

S. Crutzen, P. Jehenson, R.W. Nichols, N. McDonald;
PISC III - Status Report. Paper presented at the International Conference SMIRT 10, Report G 11/5, Los Angeles, August 14-18, 1989

S. Crutzen, M. Certo, Ph. Dombret, R. Murgatroyd;
PISC III Parametric Studies on the Effect of Defect Characteristics. Paper presented at the Seminar on Reactor Safety Research, November 20-24, 1989, Varese (I). PISC III Report No. 8, OECD-CEC, 1989

S. Crutzen, P. Jehenson, R.W. Nichols, N. McDonald;
The PISC III Programme. Paper presented at the Seminar on Reactor Safety Research, November 20-24, 1989, Varese (I).
PISC III Report No. 1, OECD-CEC, 1989

M.R. Cundy, D.R. Parramore, J. Schinkel;
In-Service Inspection at the High Flux Reactor (HFR) Petten, Purpose, Methods and Evaluation.
International Symposium on Research Reactor Safety, Operation and Modifications, Chalk River, Canada, 23-27 October 1989, ORA 34690

M.R. Cundy, M.I. de Vries;
Results from Post Mortem Tests with Material from the Old Core Box of the High Flux Reactor (HFR) Petten. International Symposium on Research Reactor Safety, Operations and Modifications, Chalk River, Canada, 23-27 October 1989, ORA 34689

R. Denis;
PISC Programme. International Colloque A.PE.N.D., Toulouse 17-22 October, 1989

R.A.H. Edwards;
The Interaction between Downtime Corrosion and High Temperature Sulphidation/Oxidation of Alloy 800H in simulated Coal Gasifier Environments. Proceedings of the 9th European Congress on Corrosion, Utrecht (NL), October 1989, Paper EG 258

M. Farag, G. Piatti;
Superplastic Behaviour of 18Cr-11Mn-2Ni Stainless Steel. Intern. Conference on Evolution of Structural Materials. AIM, 31 May - 2 June 1989, Milano (I). Proceedings of the Conference. AIM Bologna (1989) 289-295, ORA/PRO 34699

P. Fenici, G. Casini;
JRC fusion materials irradiations in HFR: present status and prospectives. Oral Presentations at the Meeting: The HFR Petten, Prospects and Future Utilization, JRC Petten, April 1989

B. Fischer, G. Fischer, J.F.W. Markgraf, S. McAllister, D.J. Perry†, I. Ruyter;
Power Ramp Testing of LWR Fuel Rods at the HFR. Colloquium on the HFR Petten, Prospects and Future Utilization, Petten, April 20-21, 1989. Proceedings EUR 12522, page 343 (poster)

G. Fischer, W. Goll, I. Ruyter, J.F.W. Markgraf;
Verhalten von DWR-U/Pu-Mischoxid-Brennstäben bei Leistungstransienten. ORA 34554

R.J. Fordham, J.B. Veyret, F. Costa Oliveira;
Corrosion of Engineering Ceramics. Proceedings of Seminar "Corrosione ad alta temperatura in gas e prodotti di combustione di componenti per sistemi energetici" organised by ENEL-CRTN, Milan, February 1989 (invited contribution)

M. Forte, C. Zeyen;
Neutron Optical Spin-Orbit Rotation in Dynamical Diffraction. Workshop on Fundamental Physics with Slow Neutrons.
ILL, 8-11 March 1989, Grenoble (F), Nuclear Instruments and Methods A. ORA/ART 34646

S. Garribba, A. Servida, G. Volta;
Generalized Structures for the Composition of Evidence. Third IFSA Congress, IFSA, 6-11 August 1989, Seattle, Wash. (USA), Proceedings of the Congress, ORA/PRO 35041

S. Garribba, A. Lucia, A. Servida, J. Bressers;
Damage Assessment and Residual Lifetime Estimation. An Artificial Intelligent Approach to Structural Safety Analysis. ICOSSAR '89. International Assessment Structural Reliability, ASCE, 7-11 August 1989, San Francisco, Cal.(USA), Proceedings of the Conference, ORA/PRO 34888

P. Gibson, N. Barrett, D. Cunningham, R. Edwards, A. Manara, K. Roberts;
Synchrotron Radiation for the Characterisation of Sylphidised Steel Surfaces. Second European Conference on Progress in X-Ray Synchrotron Radiation Research, Ist. Naz. Fisica Nucleare, 2-10 October 1989, Roma (I), Proceedings of the Conference, ORA/PRO 34958

V. Guttmann;
The Influence of Sulphidation and Carburisation upon Creep. Symposium on Oxidation and Mechanical Properties, The Institute of Metals, London, March 1989

V. Guttmann, M. Schütze;
The Interaction of Corrosion and Mechanical Properties at Elevated Temperatures. Presented at the 1989 European Conference on High Temperature Materials in Engineering-Metal, Ceramics, Plastics.
London, 27-28 April 1989, ORA 34817

V. Guttmann, H. Schönherr;
Zum Kriechverhalten der Legierung G-X 40 NiCrNb 35 25 nach thermischer Vorbehandlung. 12. Vortragungsveranstaltung der FUW Düsseldorf, 1 December 1989, ORA 35144

H. Hausen, W. Schüle, M.R. Cundy;
Irradiation Creep Experiments on Fusion Reactors: Candidate Structural Materials. 10th International Conference on Structural Mechanics in Reactor Technology.
Anaheim, USA, 14-18 August 1989, ORA 34518

E.D. Hondros, H. Kröckel, R. Streiff;
Computerized Representation of Data and Knowledge on High Temperature Corrosion and Corrosion Control. Second International Symposium on High Temperature Corrosion of Advanced Materials and Coatings.
European Corrosion Fed., May 1989

K. Hornberger, H. Stamm;
Analysis of Structures with Viscoplastic Material Behaviour. ABAQUS Users Meeting, Hibbit, Karlsson & Sorensen Inc., 24-26 May 1989, Stresa (I), Proceedings of the Meeting, (1989) 111-125, ORA/PRO 34698

C.P. Hunter, R.C. Hurst;
Creep Crack Growth Verifiation Testing in Tubular Components. Proceedings of the 10th International Conference on Structural Mechanics in Reactor Technology, August 1989, Anaheim USA

C.P. Hunter, J.M. Church, R.C. Hurst;
Creep Crack Growth Measurement on Notched Components. 4th International Conference on Creep and Fracture of Engineering Materials and Structures, Swansea, 1-6 April 1990, ORA 34819

V. Khong;
Inference Engine Building & A Case Study. International Course on "Expert System in Structural Safety Assessment", MPA, CEC, 2-4 October 1989, Stuttgart (FRG), Lecture Notes in Engineering, Springer Verlag, K. Kussmaul, A. Jovanovic, A.C. Lucia (Eds.) (1989) 422-440, ORA/PRO 35077

H. Küpfer, I. Apfelstedt, R. Flükiger, R. Meier-Hirmer, T. Wolf, H. Scheurer;
The Influence of Neutron Irradiation on Inter- and Intragrain Critical Currents and Related Function Properties of $YBa_2Cu_3O_{7-x}$. TMS Annual Meeting, Las Vegas, 27 February - 3 March 1989, ORA 34688

H. Kwast, R. Conrad, P. Kennedy, G. Verstappen, W. Roux, J.D. Elen;
Tritium Residence Times of Lithium Zircontaes Irradiated in the EXOTIC exp. 4 and 5.
ICFRM-4, Kyoto, 4-8 December 1989, ORA 35038

E. Lang, V. Lupinc, A. Marucco;
The Effect of Thermo-mechanical Treatments on Short-range Ordering and Carbide Precipitation in a Ni-Cr Based Superalloy. EUROMAT '89, Aachen (FRG), 22-24 November 1989, ORA 35145

F. Lanza;
Exploratory studies of JRC on the D-D reaction in solid materials. Oral presentation at the Workshop "Understanding of Cold Fusion Phenomena, Varenna, September 1989

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
The Petten Neutron Radiography Services.
Colloquium on the HFR Petten, prospects and future utilization, Petten, 20-21 April 1989, Proceedings EUR 12522, p. 353 (poster, identical with nb. 10 poster)

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
Application of image processing techniques in dimensional analysis. The Third World Conference on Neutron Radiography, Osaka, 14-18 May 1989

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
Comparison of optical density profiles from a travelling microdensitometer and a PC-based image analysis system. The Third World Conference on Neutron Radiography, Osaka, 14-18 May 1989 (contribution and poster)

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
The Petten Neutron Radiography Services (PNRS), facilities and methods. The Third World Conference on Neutron Radiography, Osaka, 14-18 May 1989 (contribution and poster)

H.P. Leeflang, J.F.W. Markgraf, K.H. van Otterdijk;
Experience with the HB-8 Facility at the Petten High Flux Reactor (HFR) for Thermal and Subthermal Neutron Radiography. The Third World Conference on Neutron Radiography, Osaka, 14-18 May 1989, ORA 34749

H.P. Leeflang, J.F.W. Markgraf;
Neutron Radiography Activities in Europe.
First Canadian Neutron Radiography Workshop on Neutron Radiography and its Applications, Chalk River, 12-13 December 1989

Y.Z. Li, M.C. Nguyen, H.H. Over;
Creep and Creep Crack Growth Evaluation within the European High Temperature Materials Databank, Petten. Fourth International Conference on Creep and Fracture of Engineering Materials and Structures, Swansea, April 1990

A. Lucia;
Developing Expert Systems for Structural Diagnostics and Reliability Assessment at JRC.
International Course on "Expert System in Structural Safety Assessment, MPA, CEC, 2-4 October 1989, Stuttgart (FRG), Proceedings of the Course, Sperling, K. Kussmaul, A. Jovanovic, A.C. Lucia (Eds.), ORA/PRO 35080

G. Maciga, P. Papponetti, S. Crutzen, P. Jehenson;
Application of the PISC results and methodology to assess the effectiveness of NDT techniques applied on non-nuclear components.
Paper presented at the Seminar on Reactor Safety Research. 20-24 November 1989, Varese (I)

L. Manes, V. Dallacasa;
A New Theory on the Superconducting State by Spacing Statistics. Application to High Tc Superconductors. Conference International Supraconductivité, Inst. Industrial Technology Transfer, 23-24 November 1989, Paris (F), Proceedings of the Conference, ORA/PRO 34917

J.F.W. Markgraf;
The Practical Utilization of Nitrocellulose Film in Neutron Radiography. Third World Conference on Neutron Radiography, Osaka (Japan), 14-18 May 1989, ORA 34746

J.F.W. Markgraf;
Neutron Radiography in Europe for Industry and Research, Techniques, Facilities and Applications.
International SITEF NDT symposium 1989, Toulouse, 17-19 October 1989

J.F.W. Markgraf;
Neutron Radiography in Europe for Industry and Research. Avancées Européennes en Essous Non Destructifs, Toulouse, 17-22 Octobre 1989, ORA 34622

R. Matera, W. Janssen, Y. Crutzen, F. Farfaletti-Casali;
Design Optimisation. Study of an innovative concept divertor plate based on fibre composite technology. Oral presentation at the 4th International Conference on Fusion Reactor Materials, Kyoto, December 1989, to be published in Journal of Nuclear Materials

R. Matera, M. Merola, M. Biggio, E. Cicchetti;
Behaviour of First Wall Components under Thermal Fatigue. Oral Presentation at the 4th International Conference on Fusion Reactor Materials, Kyoto, December 1989, to be published in Journal of Nuclear Materials

R.L. Moss;
Progress towards Boron Neutron Capture Therapy at the High Flux Reactor Petten. Workshop on Neutron Beam Design, Development and Performance for Neutron Capture Therapy, MIT, Boston, 30-31 March 1989

R. Murgatroyd, S.J. Crutzen, C. Vinche, H. Seed;
Retrospective Analysis of PISC II Plate No. 3 Inspection. Paper presented at the International Conference, NDE in the Nuclear Industry, 9th International Conference ASM, Tokyo, 25-28 April 1989

J.F. Norton, S. Canetoli, F. Gandrey, K. Schuster, R. Santorelli;
Oxidizing/Sulphidizing Corrosion at 600^{0}C of some Superalloys. Proceedings of Seminar of "High Temperature Corrosion of Power Plant Components", organised by ENEL/CRTN, Milan, February 1989, invited lecture

C. Olagnon, E. Bullock;
The Influence of Whisker Morphology on Processing of Silicon Nitride Matrix Composites. Second International Conference on Ceramic-Ceramic Composites, Mons (Belgium), 17-19 October 1989, ORA 35053

H.H. Over, B. Buchmayr;
Collection and Evaluation of Fatigue and Fracture Mechanics Data According to the Standard of the European HTM-Databank Petten. ASTM Committee E9 Symposium "The Application of Automation Technology of Fatigue and Fracture Testing", Kansas City, May 1989

S.J. Perry†, B. Fischer, J.F.W. Markgraf, P. Puschek, K.W. de Haan;
Re-Instrumentation Technique of Irradiated Fuel Rods for Pressure Monitoring during Irradiation at the HFR Petten. Annual Meeting of the EC Working Group on Hot Cells, Karlsruhe, 27-28 September 1989, ORA 35069

S.D. Peteves, M. Nakamura, D. McGarry, E. Nagy, A.W.J.J. Gallee;
Influence of Machining on the Strength of Hot-pressed Silicon Nitride. First European Ceramic Society Conference, Maastricht, 18-23 June 1989, ORA 34974

G. Piatti, P. Clerici, P. Davoli;
Fatigue Behaviour of a High Mn Stainless Steel for Thermonuclear Fusion Purpose. Oral Presentation at the International Conference on Evolution of Structural Materials, Ass. It. di Metallurgia, Milano, June 1989

G. Piatti, M. Vedani;
Metallurgical and Mechanical Characterisation of a Submerged Arc Welded Joint in a 316 Type Stainless Steel. Oral Presentation at the Conference on Reactor Safety Research, Varese, September 1989

T. Pradell, P. Glaude, S.D. Peteves, A.W.J.J. Gallee;
Determination of Residual Stresses caused by Grinding of Silicon Nitride Ceramics. First European Ceramic Society Conference, Maastricht, 18-23 June 1989, ORA 34973

E. Ruedl;
Materials Problems in Fusion Technology as a Challenge for Electron Microscopy.
Oral Presentation at XVII Congresso di Microscopia Elettronica, Lecce, October 1989

E. Ruedl;
Ossidazione di un acciaio austenitico al Mn tra 800 e 1200^{0}C. Oral Presentation at XVII Congresso di Microscopia Elettronica, Lecce, October 1989

E. Ruedl;
Compositional and Structural Changes near the Surface of Mn-Cr austenitic steels due to Oxidation, Evaporation or Liquid Li-corrosion.
Oral Presentation at the Int. Conf. on Fusion Reactor Materials, Kyoto, December 1989,
to be published in Journal of Nuclear Materials

E. Ruedl, G. Valdre;
Study of the Formation of Dislocations, He-bubbles and Precipitates in Alpha-implanted Mn-Cr austenitic steels. Oral Presentation at the Int. Conf. on Fusion Reactor Materials, Kyoto, December 1989,
to be published in Journal of Nuclear Materials

R. Santorelli, J.F. Norton, S. Canetoli, F. Bregani;
A Study of the Corrosion Behaviour of two Nickel-based Superalloys exposed in complex Sulphur-containing Atmospheres at 600^{0}C.
Proceedings of the Int. Conf. on "Evolution of Advanced Materials", AIM and American Soc. of Metals-Europe, Milan, May 1989, pp. 499-509

P. Schiller, F. Brossa, D. Quataert;
Qualitative and Quantitative Observations during Simulated Plasma Disruptions on Metallic Surfaces. Oral Presentation at IAEA Consulting Meeting on Particle Surface Interaction Data for Fusion, Vienna, April 1989

P. Schiller, K. Ehrlich, J. Nihoul;
History, Present Status and Future of Fusion Reactor Materials Research in the EC and other European Countries. Invited Paper at the 4th Int. Conf. on Fusion Reactor Materials, Kyoto, December 1989, to be published in Journal of Nuclear Materials

R. Scholz;
Blank Profiles in Tracer Diffusion under Irradiation.
Oral Presentation at the Conference "Diffusion in Materials", Aussois (F), March 1989

W. Schüle;
Radiation enhanced Self-diffusion in Nickel.
Oral Presentation at "Fruhjahrstagung der Deutschen Physikalischen Gesellschaft", Münster, April 1989

W. Schüle, E. Lang;
Studies of Phase Transformation in Fe-Mn-Cr Alloys. Oral Presentation at "Moderne Entwicklungen in Materialwissenschaft und Materialtechnik", Karlsruhe, June 1989

A. Servida;
ARTIC: A Knowledge Engineering Approach to Damage Assessment of High Temperature Pressurized Components. Invited presentation at the International Course on "Expert Systems in Structural Safety Assessment", 2-5 October, 1989, MPA University of Stuttgart, Germany.
Proceedings to be published by Springer-Verläg in its series "Lecture Notes in Engineering and Design"

A. Servida;
Reasoning about Data: The Combination of Multiple and non-homogeneous Sources of Knowledge. Invited presentation at the International Course on "Expert Systems in Structural Safety Assessment", October 2-5, 1989, MPA University of Stuttgart, Germany.
Proceedings to be published by Springer-Verläg in its series "Lecture Notes in Engineering and Design"

A. Servida;
Fuzzy Uncertainty Measures. A Tutorial Approach. Invited presentation at ISPRA Seminar on "Structural Reliability", 11-15 May, 1987, Ispra, Italy. To appear in the Proceedings of the Ispra Seminar edited by A.C. Lucia, Reidel Publishing 1989

G. Solomos, V. Moussas;
A Time Series of Approach to Fatigue Crack Propagation. European Mechanics Colloquium EUROMECH 250, Univ. Pavia, 19-23 June 1989, Como (I), ORA 34801

H. Stamm, K. Hornberger;
Analysis of Structures with Viscoplastic Material Behaviour. Oral Presentation at ABAQUS Users' Meeting, Stresa, May 1989

H. Stamm, G. Walz, K. Hornberger;
Numerical Investigation of Crack Tip Fields in a Viscoplastic Material. Oral Presentation at COMPLAS II, Barcelona, September 1989

M.F. Stroosnijder, V. Guttmann, R.J.N. Gommans, J.H.W. de Wit;
Influence of Creep Deformation on the Corrosion Behaviour of a CeO_2 Surface Modified Alloy 800H in Sulphidizing/Oxidizing/Carburizing Environment. 2nd International Symposium on High Temperature Corrosion of Advanced Materials and Coatings, Les Embiez, France, 22-26 May 1989, ORA 34818

M.F. Stroosnijder, M.J. Bennett, V. Guttmann, J.F. Norton, J.H.W. de Wit;
The Effect of Ce Implantation on the Corrosion of Alloy 800H in a Sulphidizing/Oxidizing/Carburizing Environment at 700^0C.
Gordon Research Conference "Frontiers of Science - Corrosion". Colby Sawyer College, New London, New Hampshire, U.S.A., 17-21 July 1989 (invited contribution)

M.F. Stroosnijder, V. Guttmann, J.H.W. de Wit;
Corrosion of a CeO_2 Surface Modified Alloy 800H in a Sulphidizing/Oxidizing/Carburizing Environment under Mechanical and Thermal Cycling. 9th European Congress on Corrosion, Utrecht, 2-6 October 1989, ORA 34975

G.P. Tartaglia, H. Kröckel, H.H. Over;
The Integration of a FEM Code with a Material Properties Databank. International Conference for Fusion Reactor Materials ICFRM 4, Kyoto, Japan, December 1989

O. van der Biest, C. Weber, L.A. Garguet;
Role of Oxidation in Creep and High Temperature Failure. 3rd International Symposium on Ceramic Materials and Components for Engines, Las Vegas, Nevada, USA, 27-30 November 1988, ORA 34479

C. van der Sijp, O. van der Biest, E. Nagy, D. McGarry, S.D. Peteves;
The Influence of Carbon on the Processing and Properties of Silicon Nitride. Proc. 2nd International Conference on "Ceramic Powder Processing Science", Berchtesgaden, October 1988

Ph. Vannson;
Besoins en Normalisation: Traveaux du Groupe VAMAS. VAMAS-meeting 8-9 November 1988, ORA 34480

K.H. van Otterdijk, H.P. Leeflang, J.F.W. Markgraf;
Applications of Image Processing Techniques in Dimensional Analysis. Third World Conference on Neutron Radiography, Osaka, Japan, 14-18 May 1989, ORA 34750

M. Vedani, G. Piatti, C. De Gregorio;
Damage Evaluation by Microstructural Observation and Tensile Testing in a Cast Al-SiC Metal-Matrix Composite, MECAMAT 1989, UNIREC, 15-17 November 1989, St. Etiènne (F), Proceedings of the Conference, ORA/PRO 34992

J.B. Veyret, R.J. Fordham, M. Billy;
Gaseous Corrosion of some Silicon Nitrides in SO_2-Air Mixtures at High Temperature.
First European Ceramic Society Conference, Maastricht, 18-23 June 1989, ORA 34628

J.B. Veyret, M. Billy;
Oxidation of Hot-pressed Silicon Nitride: Modelling. First European Ceramic Society Conference, Maastricht, 18-23 June 1989, ORA 34859

A. Walz, H. Stamm, K. Hornberger;
Numerical Investigation of Crack Tip Fields in a Viscoplastic Material, COMPLAS II, Univ. Politecnica de Catalunya & Univ. College Swansea (UK), 18-22 September 1989, Barcelona (E), Proceedings of the Conference, Pineridge Press, D.R.J. Owen, E. Hinton, E. Onale (Eds.) (1989), 1159-1170, ORA/PRO 35002

W.L. Zijp, J. Ahlf;
Upgrading Activities for the HFR Petten.
International Symposium on Research Reactor Safety, Operation and Modifications, Chalk River, Canada, 23-27 October 1989, ORA 34691

Patent

A.R. Korko, J.F.W. Markgraf;
A Pump with an Electronically Controllable Stroke Volume. European Patent granted under number EP 0 202 714 B1, 1989

V Glossary

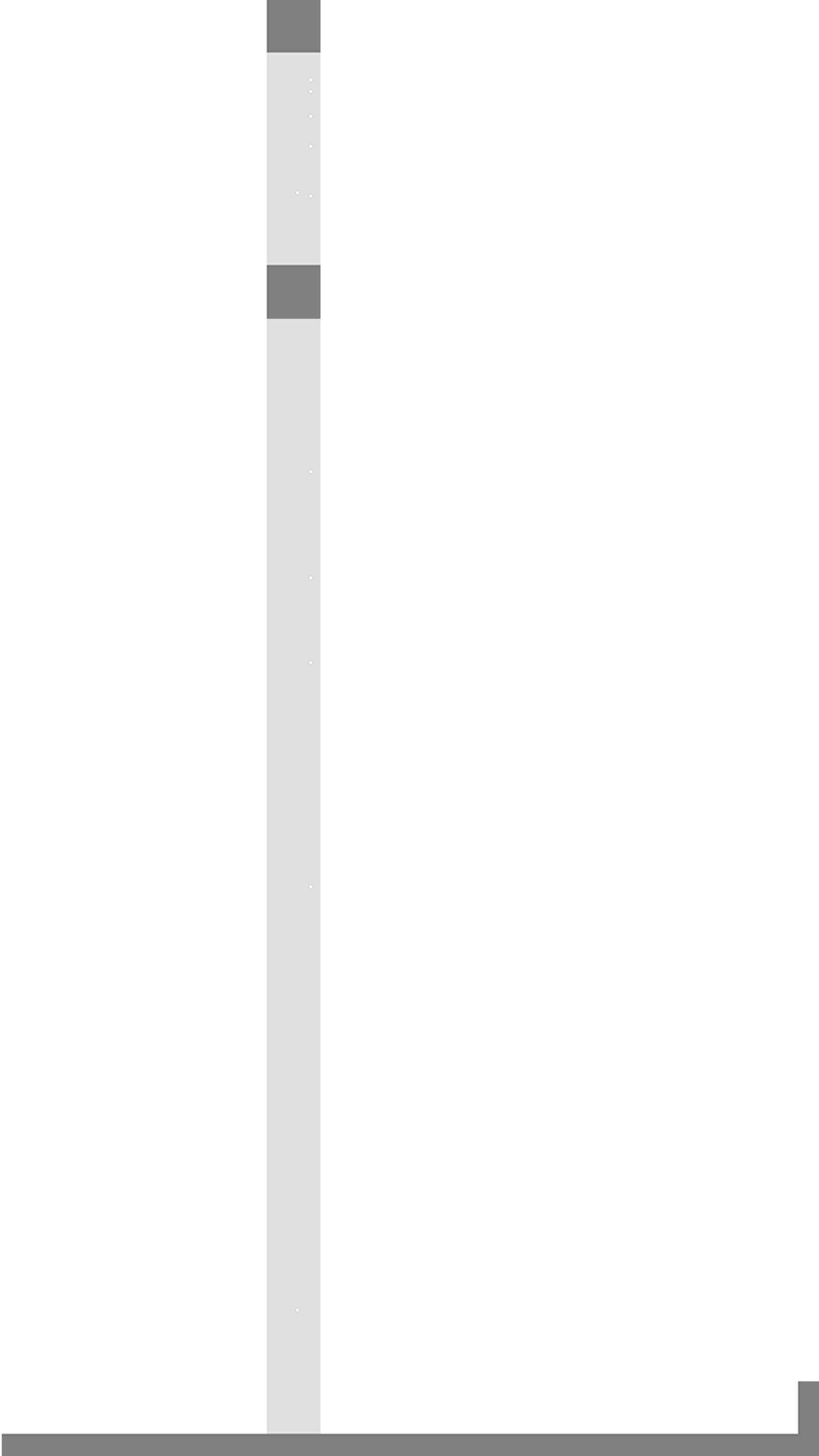
Glossary

ABAQUS	Finite Element Code
AES	Auger Electron Spectroscopy
AISI	American Iron and Steel Institute
AMCR	Acier Mangan Chrome (Low activation materials)
APS	Air Plasma Spraying
ARTIC	Expert System
ASME	American Society for Mechanical Engineers
ASTM	American Society for Testing and Materials
BCS	Bardin Cooper Schrieffer (Theory)
BISCO	Bismuth-Strontium-Calcium-Oxygen Compound
BN	Boron Nitride
BNCT	Boron Neutron Capture Therapy
BRITE	Basic Research in Industrial Technologies for Europe
BUMMEL	Bubble Mobility Measurement Level
BWR	Boiling Water Reactor
CAD	Computer Aided Design
CCG	Creep Crack Growth
CEC	Commission of the European Communities
CEGB	Central Electricity Generating Board
CNB	Chevron Notched Beam
CNR	Centro Nationale di Ricerche
COST	European Cooperation in the Field of Scientific and Technical Research
COST 50	Materials for Turbines
COST 501	Advanced Materials for Power Engineering Components
COST 505	Materials for Steam Turbines
CRKPRO	Crack Propagation
CRV	Common Reference Vocabulary
CSNI	Principal Working Group on Primary Circuit Integrity
CVD	Chemical Vapor Deposition
DB	Data Bank
DG	Directorate General
DPA	Displacement per Atom
EB	Electron Beam
EBR	Experimental Breeder Reactor
EC	European Communities
ECN	Energieonderzoek Centrum Nederland
ECU	European Currency Unit
EDC	Effect of Defect Characteristics
EDS	Energy Dispersive System
EELS	Electron Energy Loss Spectroscopy
EGF	European Group of Fraction
EMF	Electromotive Force
ENEA	Ente Nazionale Energie Alternative
ENEL	Ente Nationale di Energia Electrica
ERD	Elastic Recoil Detection
ESPRIT	European Strategic Programme for Research & Development in Information Technology
ETL	Environmental Testing Laboratory
FAFNIR	Fatigue in First Wall Nuclear Irradiation Rig

FBC	Fluidised Bed Combustion
FBR	Fast Breeder Reactor
FE	Finite Element
FEM	Finite Element Method
FS	Flame Spraying
HAZ	Heat Affected Zones
HIFIR	High Flux Irradiation Reactor
HFR	High Flux Reactor
HT	High Temperature
HTM	High Temperature Materials
HTMDB	High Temperature Materials - Data Bank
HTR	High Temperature Reactor
IAM	Institute for Advanced Materials
ICFRM	International Conference for Fusion Reactor Materials
IIF	Indentation Induced Flaw
IR	Infra Red
ISE	Institute for System Engineering (JRC Ispra)
ISI	In Service Inspection
ISO	International Organization for Standardization
ITER	International Thermo-nuclear Experimental Reactor
JOULE	Joint Opportunities for Unconventional or Long-term Energy Supply
JRC	Joint Research Centre
KAKADU	Kamin Kasel-Duo (Twin capsules for fuel pin irradiation)
KECU	Kilo European Currency Units
KFA	Kernforschungsanlage Jülich
KFK	Kernforschungsanlage Karlsruhe
LCF	Low Cycle Fatigue
LEFM	Linear Elastic Fracture Mechanics
LFR	Low Flux Reactor
LMFBR	Liquid Metal Fast Breeder Reactor
LOCA	Loss of Cooling Accident
LPPS	Low Pressure Plasma Spray
LWR	Light Water Reactor
MA	Minor Actinides (Np,Am,Cm)
MAN	Machienenfabriek Augsburg-Nürnberg
MECU	Million European Currency Units
MITC	Measurement of UT Instrument and Transducer Characteristics
MMC	Metal Matrix Composite
MPA	Staatlich Materialprüfungsanstalt (Stuttgart)
MPR	Materials Performance and Reliability
NDE	Non Destructive Evaluation
NDW	Nozzles and Dissimilar Metal Welds
NET	Next European Torus
NILOC	Nitride Fuel, Low Oxygen and Carbon
NNE	Non-Nuclear Energy
ODS	Oxide Dispersion Strengthened
OECD	Organization for Economic Cooperation and Development
ORR	Oak Ridge Reactor

PET	Positron Emission Tomography
PISC	Project for the Integrity of Steel Components
PIXE	Proton Induced X-Ray Emission
PNA	Proton Nuclear Activation Analysis
POMPEI	Pellets Oxide Mixte, Petten Irradiation
PVD	Physical Vapor Deposition
PWR	Pressurized Water Reactor
RBS	Rutherford Backscattering
RF	Radio Frequency
RRT	Round Robin Test
RTNS	Rotating Nuclear Source
RWE	Rheinisch Westfälische Elektrizitätswerke
SEM	Scanning Electron Microscopy
SIMS	Secondary Ion Mass Spectrometry
SMC	Surface Modification Centre
SOC	Sulphidizing/Oxidizing/Carburizing
TEM	Transmission Electron Microscopy
TRIO	Irradiation Device With Three Thimbles
UKAEA	UK Atomic Energy Authority
US	Ultrasonic
UTS	Ultimate Tensile Strength
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
YS	Yield Strength

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.
- Components and Thermal Fatigue: J. Bressers.
- 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.
- Characterization of High Tc Superconductors: L. Manes.
- Low Activation Steels: D. Boerman.
- Composite Materials Properties Improvements: G. Piatti, M. Airola, D. Boerman, F. Brossa, F. Dos Santos Marques, M. Vedani.
- Chemical Sensors: L. Manes.
- Surface Treatments for Improved Performance: P. Schiller, O. Gautsch, D.G. Rickerby.
- Databanks: F. Franck.
- Information Centre: M. Merz, E. Bullock, R.J. Fordham.
- COST Secretariat: J.B. Marriott.
- Project on the Integrity of Steel Components (PISC): P. Jehenson, S. Crutzen, R. Denis, H. Herkenrath.
- Safety of Final Storage in Geological Formation: Materials Research Aspects: F. Lanza.
- Materials Integrity: P. Schiller, F. Brossa, V. Coen, Mo Dawei, P. Fenici, W. Janssens, F. Lanza, B. Looman, R. Matera, G. Mercurio, G. Orabi, D. Quataert, M. Quériaud, D.G. Rickerby, E. Ruedl, R. Scholz, W. Schüle, Shi Suolang, G.P. Tartaglia.
- Nuclear Fuels and Actinide Research: R. Moss.
- Operation of the High Flux Reactor: J. Ahlf.
- Standards for Advanced Ceramics: M. Merz, J. Bressers, R.A.H. Edwards.

- Materials Databanks: H. Kröckel.
- Valorization of JRC Research Results: Spontaneous Downward Heat Transport: G. De Beni, R. Friesen.
- Boron Neutron Capture Therapy (BNCT): R. Moss.
- Joining of Ceramics to Metals: S. Peteves.
- Micro-Hydrodynamics of Laser Melted Pools: G. Tsotridis.

III INSTITUTE COMPETENCES AND FACILITIES

- Materials Characterization: J. Bressers, E. Bullock, R.J. Fordham, V. Guttmann, J.F. Norton.
- Materials Performance and Reliability: P. Schiller
- Functional Materials: L. Manes.
- Non Destructive Testing: P. Jehenson.
- Materials Databanks: H. Kröckel.
- High Flux Reactor Petten: J. Ahlf.
- Cyclotron: L. Manes.
- Environmental Testing Laboratory: J. B. Marriott
- Surface Modification Centre: P. Schiller

EDITING AND COORDINATION: M.R. Cundy, M. Merz.

European Communities - Commission

EUR 12272 — Institute for Advanced Materials, Annual Report 1989

M. Merz, editor

Luxembourg: Office for Oficial Publications of the European Communities

1990 - 196 pages - 21.0 x 29.7 cm

Series: Physical Sciences

EN

ISBN 92-826-1329-1

Catalogue number: CD-NA-12272-EN-C

Price (excluding VAT) in Luxembourg: ECU 15

Abstract

The Annual Report 1989 of the Institute for Advanced Materials of the JRC is the first of its kind since the Institute has been set up in 1988. The report highlights the Scientific Technical Achievements and presents the Institute's Competence and Facilities available to industry for services and research under contract.

The Institute executed in 1989 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**
Lay-out and Photosetting: **J. Manten**
Printing: **Trio Goemans B.V.,** Sassenheim, 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. (0221) 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) 275 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 12 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) 78 98 80 (bestellingen)
Fax (070) 47 63 51

PORTUGAL

Imprensa Nacional
Casa da Moeda, EP
Rua D. Francisco Manuel de Melo, 5
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
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

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
Shiniuku-ku
Tokyo 160-91
Tel. (03) 354 01 31

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

MAGYAR

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 veb ofset 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

Privrednivjesnik
Rooseveltov Trg 2
41000 Zagreb
Tel. 44 64 28
44 98 35
43 32 80
44 34 22
Teleks 21524 YU

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

2/90

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.

16 CD-NA-12272-EN-C

Price (excluding VAT) in Luxembourg: ECU 15

OFFICE FOR OFFICIAL PUBLICATIONS
OF THE EUROPEAN COMMUNITIES
L-2985 Luxembourg

ISBN 92-826-1329-1