ANNUAL · REPORT · 92

Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

EUR 15125 EN

A N N U A L · R E P O R T · 92

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Published by the
COMMISSION OF THE EUROPEAN COMMUNITIES

Directorate-General
Telecommunications, Information Industries and Innovation

L-2920 LUXEMBOURG

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Cataloguing data can be found at the end of this publication

Luxembourg: Office for Official Publications of the European Communities, 1993

Catalogue number: CD - NA - 15125 - EN - C

Printed in the Netherlands

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I Introduction

Introduction

Here we report on progress at the Institute during the first year of the current three year JRC Programme. In the new Programme we respond to the evolving materials needs of industry. The central theme within the Advanced Materials Programme is: the extension and reliable use of advanced materials to the limits of their capabilities. That theme remains unchanged, but we have sharpened the focus by emphasising the importance of the pre-normative aspect and by reducing the number of research areas from ten to six. These areas are:

Materials for extreme environments
Reliability and life prediction
Measurement and validation methodologies
Surface modification technologies
Fusion Materials
Materials information and data management.

This refocusing of effort on core activities allows us to exploit more effectively our central areas of competence in order to help to promote materials research as an enabling technology for the benefit of European industry.

Institute budget and manpower resources

The budget and manpower available to implement this programme are:

Programme	Scientific Research Staff	Research Budget (Kecu)
1. Specific Programme		
Advanced Materials	104	2800
Fusion Materials	15	390
2. Support to the Commission	18	1195
3. Exploratory research	7	250
4. High flux Reactor:		
Complementary	40	200
Common	1	30
Totals	185	4865

Notes

1. In addition to the above resources, 12 research staff were engaged in contract work for third parties. Contracts to a value of more than 3.5 Mecu were signed in 1992 for execution in 1992 and later years.
2. The research budget for the HFR excludes the reactor running costs.
3. The above scientific staff were supported by 30 technical and 39 administrative personnel.

We note that whereas the current JRC Programme runs for three years until the end of 1994, the Supplementary Programme for the HFR was approved for four years and will run until the end of 1995. Germany and the Netherlands share costs of 75 million ECU on an equal basis, and a further 15 MECU should be generated from other sources such as contract research.
The impact of the unfavourable economic climate on new third party research contracts was less in 1992 than might have been feared. We remain confident that the level of expected income from industrial sponsorship will be maintained.

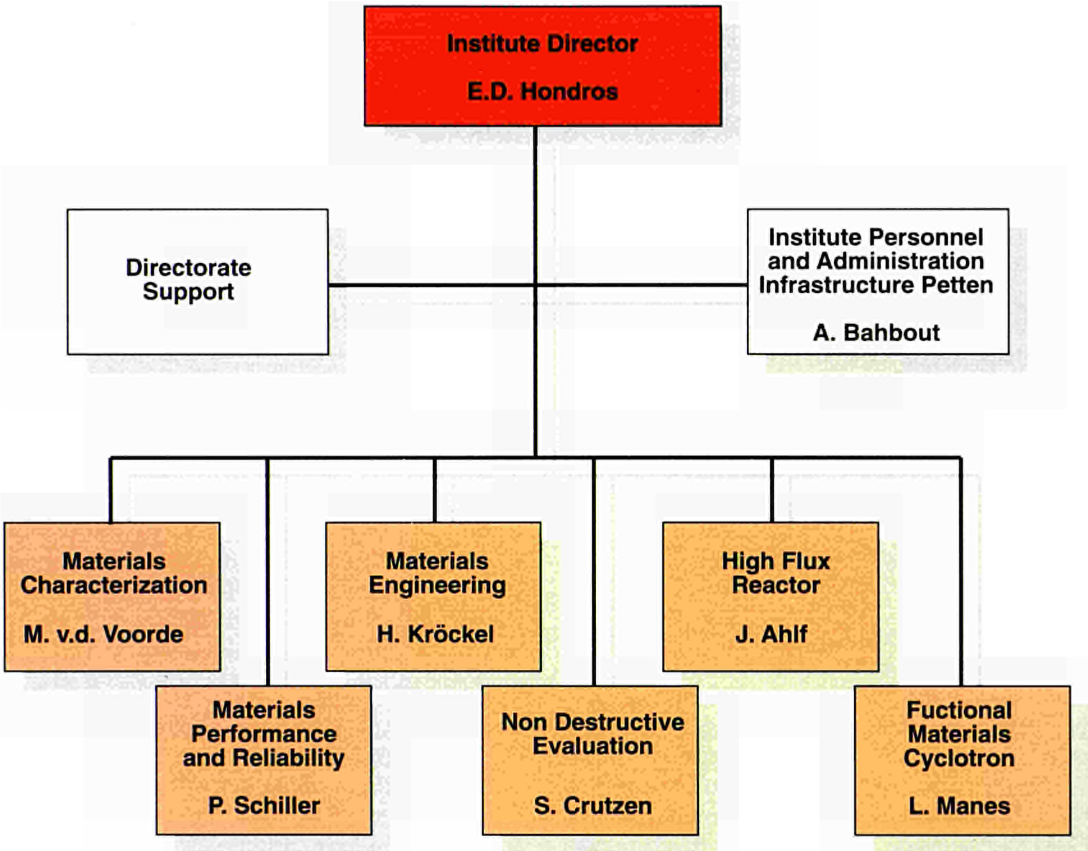
Adaptation of organigram to programme requirements

The Institute organigram was adapted to the objectives of the new Programme. The main change is that the six scientific Units - previously called Divisions - are now subdivided into Sectors corresponding to particular areas of research competence. Progressively, responsibilities and budgetary decision making are being devolved to the Sector level.

Growth of research resources

A number of developments have led collectively to a substantial growth in research capability, mostly in the Petten part of the Institute.
Thus, the completion of a new building in Petten has provided additional resources of laboratory and office space and offers more flexibility to respond smoothly to the challenges of the new Programme.

As part of the policy to increase the number of statutory staff at Petten towards a target level of 200, the decision was taken to transfer the NDE group from Ispra to Petten - including the transfer of a



High Energy X-ray Source for which a shielded building will be constructed.
The Advanced Coatings Centre was officially opened in November 1992. This Centre is constituted as a business unit and is a joint venture between the Netherlands Energy Research Foundation (ECN) and the Institute for Advanced Materials. It offers a wide range of coatings facilities and expertise to industry as well as support to ECN and IAM projects.

The main new investment is in a second x-ray diffractometer which will increase the throughput on phase analysis and extend our capabilities to the measurement of crystal orientation and residual stresses.
During this period, the Commission introduced a Programme on Human Capital and Mobility intended to promote scientific cooperation through the exchange of young researchers.
As a result, the Institute is benefitting from a considerable influx of post-doctoral workers arriving from most of the Member States of the European Communities.

Exploratory activities

Among the exploratory projects under way which could form the basis of future mainline activities, we note advances in the following:

- Laser spallation

Recent investment in a wide range of coatings technologies has stimulated new ideas for characterizing coatings. Laser-spallation is a generic method with potential for measuring the strength of adhesion of thin films to substrates. This technique uses the shock wave induced by a laser pulse to detach the coating from the substrate. The characteristics of the shock wave are measured by laser-Doppler interferometry and the stresses induced at the coating substrate interface are calculated by finite element analysis. The aim is to develop a reference test measurement technique for adhesion of coatings, as part of the pre-normative research drive.

- Boron Neutron Capture Therapy

The ultimate goal of the Boron Neutron Capture Therapy project is the treatment of brain tumour patients at the HFR. The project entered a new phase with tests on animals to establish the starting dose for clinical trials on humans. The trials, which were preceded by extensive irradiation calibration work with cell cultures, will continue into next year to establish the healthy tissue tolerance limits and to optimize treatment procedures.

Cooperation Networks and Initiatives

- CEFIR

The Institute is coordinating the EUREKA-CEFIR project with the initial objective of defining the needs and targets for high temperature ceramic fibres and of evaluating the technological and commercial prospects for their production in Europe. The participation of 22 industrial partners and 24 research institutes in this project is motivated by a perceived strategic vulnerability of the European aero-engine industry. The project achieved its objectives for the definition stage and a European scale R&D work programme was prepared. The project is now ready to proceed to the implementation phase.

- CEASI

This concerted European Action on Structural Intermetallics was catalysed by the Institute as a means of integrating the growing, but diffuse research activities in Europe. Support for the organisational aspects conducted by the Institute was given by DG XII/C. Thorough study and assessment by an ad-hoc group of scientists resulted in a proposal being accepted by BRITE/EURAM for a concerted Action, led by IRC at Birmingham University, UK. For this type of research, the BRITE programme funds the "enabling" costs, the research funding is the responsibility of the individual participants. The Action covers a wide range of intermetallics for potential application in aero-space and engine construction, as well as in power engineering, transport and other industries. The participating organisations total 87 including 34 from industry; they represent 8 E.C. countries and 5 other European countries.

- European Pre-normative Research Association

The establishment of a European Pre-normative Research Association (EPRA) was proposed and this is still at the discussion stage. The aim is to promote pre-normative research on materials i.e. research which is required for establishing European standards. This proposal was formulated under the guidance of IRDAC (Industrial Research and Development Advisory Committee) in association with national standards organizations and CEN (Committee Europeen de Normalisation). The plan foresees that the research work will be decentralized to a group of participating laboratories. The Institute for Advanced Materials will be one of these laboratories, but our primary role will be to act as Executive Agent for the daily management of EPRA and to act as a Reference Laboratory.

Materials Events

The Royal Society organized a soirée in London on May 6th on "Science into Industry" to which the Institute was invited to contribute. The broad aim was to promulgate science and its applications to a wide audience from industry, commerce, politics, education and the media. Our contribution emphasized the European dimension of research on aero-engine turbine materials. The Institute organized a seminar in Aveiro, Portugal, jointly with the University of Aveiro on "The Role of Surfaces and Interfaces in Materials". Our objective in holding the seminar, and in supplying a large number of presentations, was to help stimulate awareness of and interest in the ubiquitous importance of interfaces in advanced materials. For us, this was something of an experiment in technology diffusion, and we were very pleased with the response it generated.
The introduction of the HCM programme this year has had the indirect effect of some what reducing the intake of PhD students for training in the Institute. We must acknowledge the value of the contributions of these students to the Institute research projects. The high quality of the research is particularly evident in the field of high temperature mechanical properties of alloys where three successive students have won awards for their work.

II Scientific - Technical Achievements

1. Specific Programme: Advanced Materials

Research Area: Materials for Extreme Environments

Assessment of Research Area Progress and Achievement: Materials for Extreme Environments

Materials operate at the limit of their capabilities and efforts are necessary to develop new materials for reliable use in demanding operating conditions for example in aeroengines, power plants etc.
A valuable data base is to be generated for these newly developed metallic materials, engineering ceramics and composites in order to ensure safe and economic design. It is therefore necessary to investigate failure mechanisms which requires knowledge of the relationship between materials properties and microstructure.

The research work is elaborated through three strategic lines:
i) the development of modern test equipment,
ii) the evaluation of HT materials behaviour and the understanding of degradation mechanisms in industrial simulative environments,
iii) the improvement of high temperature materials.

The high temperature (up to 1400^{0}C) corrosion, creep and fatigue properties are studied on new metallic materials (ODS superalloys, intermetallics) and Si_3N_4 - SiC based ceramics and composites.

i) Development of modern test equipment

The installation of unique ultra high temperature equipment for specimen and sub-critical components testing (corrosion burner rig - multi purpose modern HT corrosion/mechanical test machine) was completed and set to work. This modern equipment catalyses the setting up of networks of European Laboratories around the development of industrial materials standards. Future efforts will concentrate on projects having good subsidiarity such as "unique testing on modern ceramics/composites".

ii) Study of materials degradation mechanisms

A final report on the H.T. mechanical behaviour of typical iron and some nickel base ODS superalloys was prepared; some boundary conditions for safe operation were formulated with respect to component life time, stress/strain ranges, corrosion etc. These results are sufficiently conclusive to be extrapolated to other alloys and test environments.
Failure modes in model type monolithic Si_3N_4 and 2D continuous fibre reinforced ceramic matrix composites are being investigated and first attempts made to determine the boundary conditions for reliable industrial operation. The knowledge in this new but challenging field is rather poor and may require a stronger European collaboration between material producers, industrial users and materials scientists.

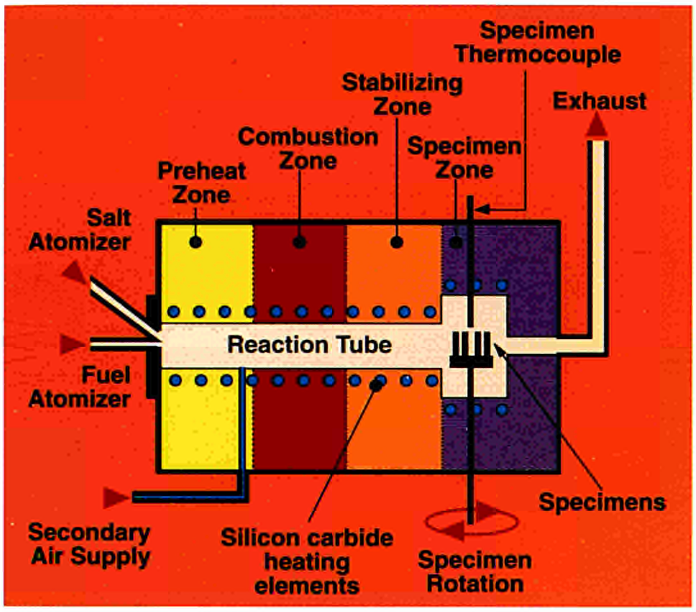
A data base of creep and damage behaviour on a typical newly developed intermetallic Ti_5Si_3/Ti_3Al was generated. This material shows good creep strength, but has poor creep ductility particularly at lower temperatures and is severely oxidised at 800^{0}C. The technical application of this materials is in doubt. Further studies should take a selective approach with respect to material and test conditions and the work should receive a European collaborative dimension.

iii) New improvements in high temperature materials

Superalloys and engineering ceramics are severely damaged by corrosion and mechanical stress at very high temperatures. Exploratory data have resulted in the development of corrosion resistant Si_3N_4 model type ceramics and improved MeCrAlY coated superalloys.
The relationship between properties and composition/microstructure and processing technique is well under control and will form the basis for further innovations. Because of the multi parameters involved in this R&D and the limited resources available, the research should be well focussed and be integrated into a European network.

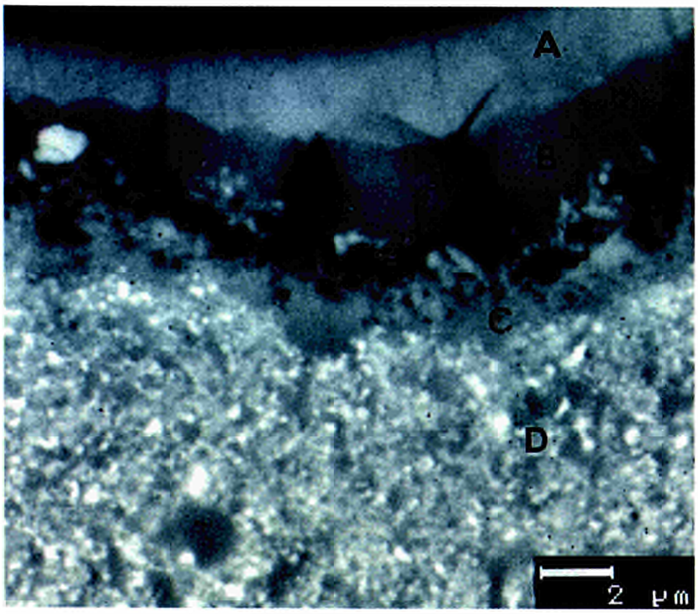
This complex materials data base forms a good basis for Research Area 2 and the modern test equipment is a useful tool for the development of test methodologies in Research Area 3.

Engineering Materials in Industrial/Emission Atmospheres



Above: *Schematic Diagram of the Petten Low Velocity Burner Rig*

Below: *Micrograph of a transverse section through Si_3N_4 exposed for 50 hours at 1100°C in the burner rig*



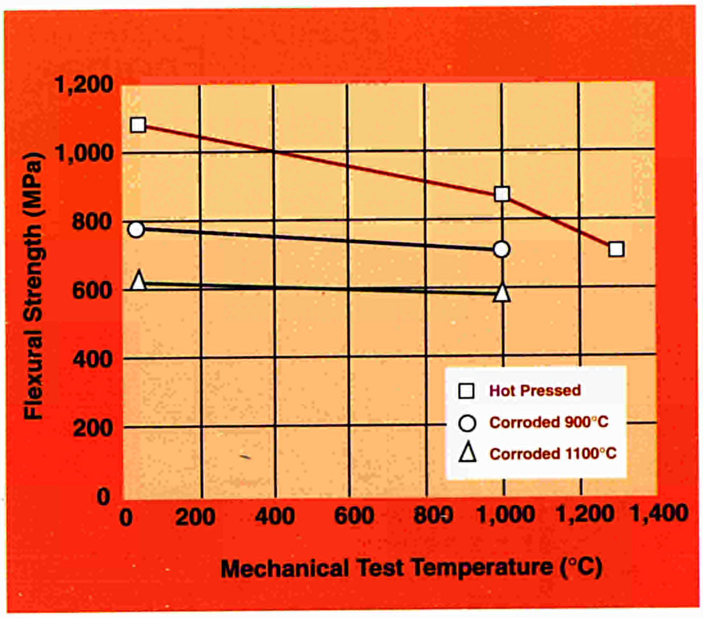
The aim of this project is to establish the kinetics and underlying mechanisms responsible for the degradation of advanced engineering alloys, ceramics and composites exposed to multi-reactant gaseous atmospheres. The synergistic influence of contaminants such as molten salts and acidic condensates is an important aspect of these studies. The project also seeks to establish the industrial applicability of such laboratory-derived data.

A new burner rig facility (Figure above) was installed and commissioned. Experimental work started on the influence of molten salts on corrosion.

The test methodology adopted for running the low-velocity burner rig was based on one for gas turbine superalloys which is currently being assessed within an international VAMAS programme. The test environment in the rig was created by burning a liquid fuel with an excess of air to produce a mixture of N_2, O_2, CO, CO_2, H_2O and SO_2/SO_3, to which salt could be added to simulate certain industrial and marine engine environments. The salt was added as an aqueous solution, made to the ASTM standard for artificial ocean water. Na_2SO_4 formed, primarily by the reaction of NaCl with SO_2/SO_3, and at temperatures below its dew point (+/-950°C under these test conditions), it deposited on material surfaces and contributed significantly to the corrosion process.

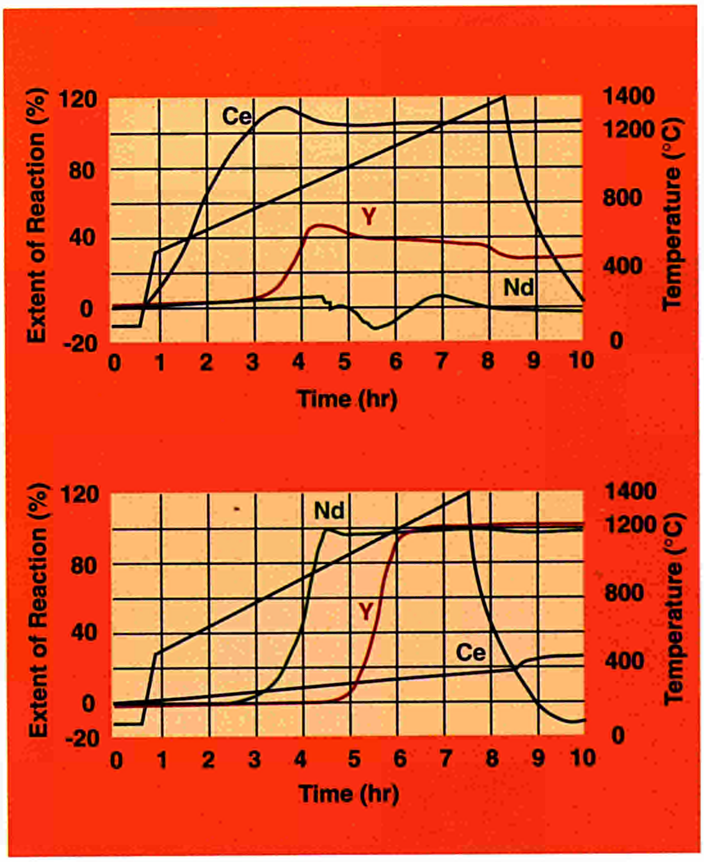
Hot-salt corrosion of Si_3N_4 was investigated experimentally using a commercial hotpressed grade containing 9% Y_2O_3 and a second high-strength grade containing 2% Al_2O_3 and 5% Y_2O_3, in collaboration with CNR-IRTEC, Faenza, Italy. On both materials, 50 hour tests in the temperature range 900-1100°C resulted in the formation of very thin Sirich surface scales and depletion of Y from the immediate sub-surface region of the base ceramic (Figure below). Even after a short-term exposure, corrosion resulted in a dramatic reduction in strength of the high-strength material (Figure on page 4 above). Higher temperature work is planned using a second thermal-cycling burner rig which was ordered in 1992.

Parallel studies of the mechanisms of corrosion of engineering ceramics at high temperatures have also concentrated on non-oxide ceramics [Si_3N_4 and SiC] with emphasis being given to gaseous corrosion in simulated or model environments using a



Above: *Graph comparing the flexural strength of Si_3N_4 in the hot-pressed and burner rig corroded conditions*

Below: *Oxidation of Ce-, Nd- and Y-H- (top) and -K-phases in air*



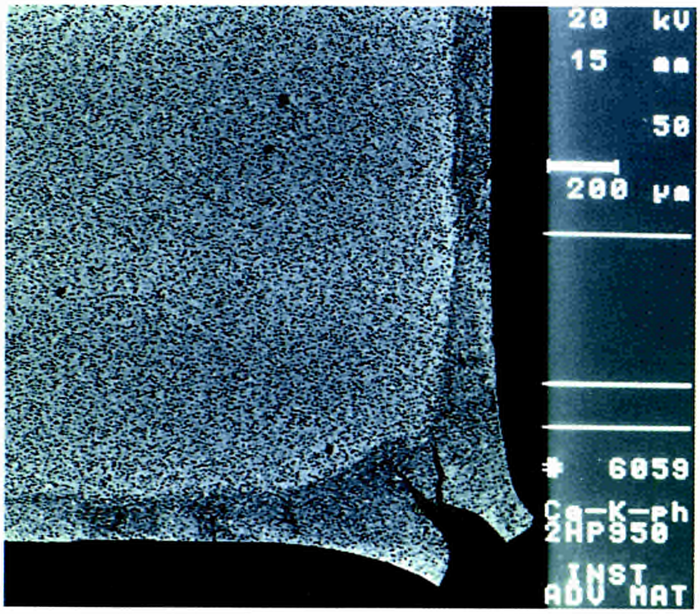
combination of methodologies, i.e., discontinuous weight change measurements, thermogravimetry and hot-stage microscopy.

The corrosion resistance of these materials is often critically dependent on the nature and amount of 'secondary' phases, usually to be found in the grain boundaries and as inclusions. This is particularly true of silicon nitrides which cannot, in practice, be fully densified without the addition of oxide sintering aids [e.g. Y_2O_3, MgO]. These oxide additives form a liquid silicate phase which, after densification, solidifies with the formation of one or more high melting point silicates. Yttrium has been one of the most important additive elements but the resulting secondary phases oxidize preferentially at temperatures below 1000^{0}C where the oxidation of silicon nitride itself is negligible. The resulting disruption of the microstructure can severely limit the use of the material. Alternatives to Y, e.g. Ce, Nd and other rare-earth elements, have been proposed and materials fabricated.

Little is known, however, about the behaviour of the secondary phases themselves, their corrosion products, rates of formation and corresponding morphological changes.
In M-Si-O-N systems, two of the most common secondary phases are the so-called H-phase [with Apatite structure] and K-phase [with α-wollastonite structure].
These materials were prepared for M = Y, Ce and Nd by hot-pressing, and their corrosion properties are being studied.

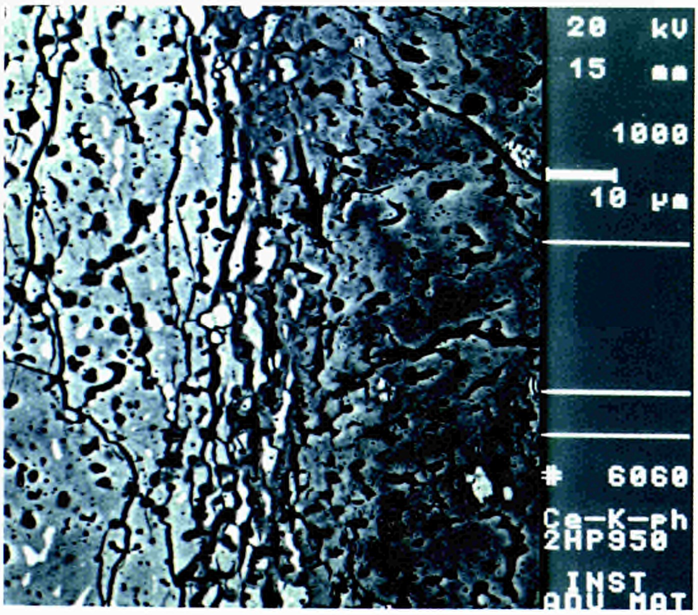
Figure below compares the oxidation behaviour of these H and K phase materials in air obtained by continuous thermogravimetry using a 2^{0}C.min^{-1} ramp to 1400^{0}C after rapid heating to 500^{0}C.
In general, all materials show significant oxidation at temperatures <1000^{0}C; both Nd-K- and Ce-H-phases are completely oxidized before this temperature is reached.
The Nd-H-phase showed considerable spallation from 900^{0}C caused by rapid internal oxidation, as illustrated by the broken part of the curve. Oxidation was complete at 1400^{0}C.

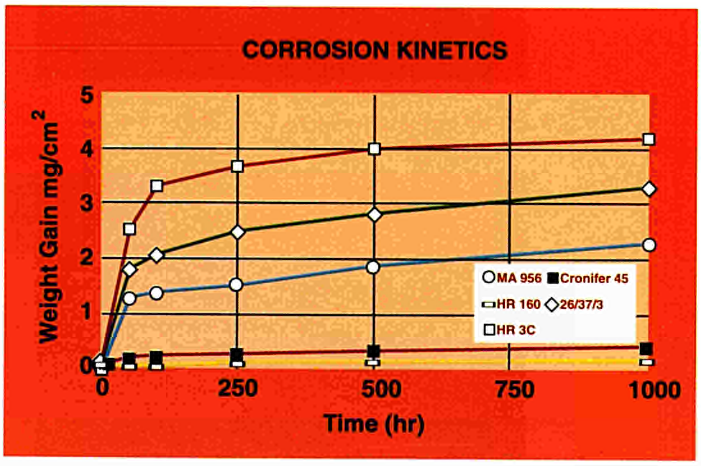
Figure on page 5 illustrates a typical SEM micrograph in cross-section of Ce-K-phase after 2h isothermal oxidation at 950^{0}C. Internal cracking is clearly visible as well as cracking of the scale.



The application of laboratory data to industrial situations is being considered from 2 points of view; firstly by a comparison of laboratory tests in both equilibrated and nonequilibrated gas mixtures, the latter being more typical of commercial processes and, secondly, by comparing these data with results from in-plant exposures.

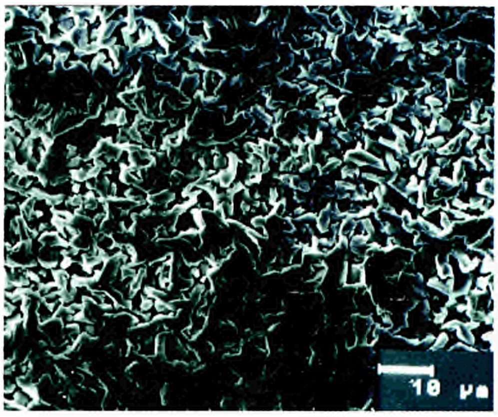
Corrosion experiments using a CO-based nonequilibrated gas mixture representative of the atmosphere found in a dry-feed entrained slagging gasifier started in collaboration with the Electric Power Research Institute in the USA. The performances of 5 candidate alloys, Table on page 7, are being studied and compared with results from similar tests carried out in an equivalent equilibrated gas mixture at 600°C. Exposures of 1000 hours were completed and the corrosion kinetics measured intermittently by mass-change measurements as shown in Figure below.



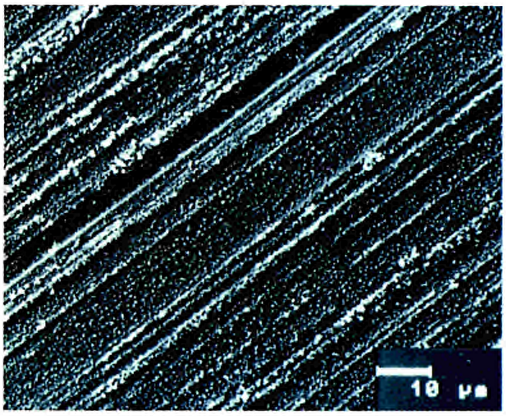


Above: *SEM micrographs of a section through Ce-K-phases after 2h at 950°C*

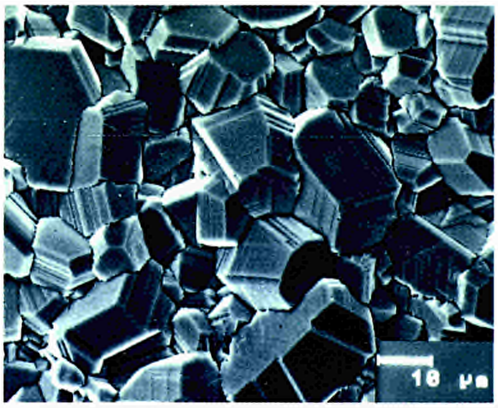
Below: *Corrosion kinetics for the alloys exposed in 0.2 H_2S - 64 CO - 3.8 CO_2 - H_2 bal, at 600°C*



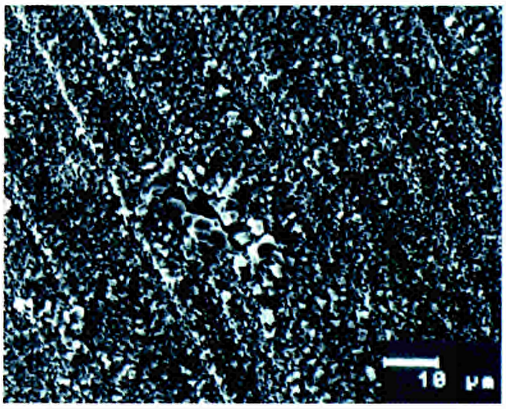
a. MA 956



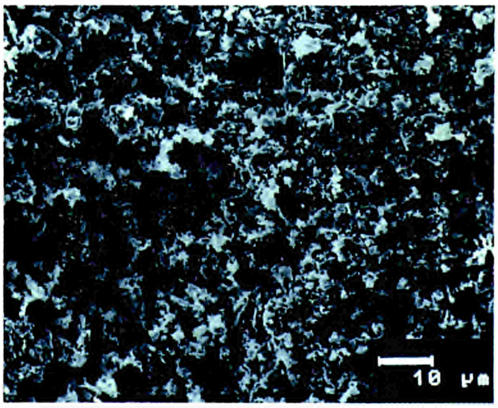
b. HR 160



c. HR 3C



d. Cronifer 45



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Some surface and cross-sectional microscopic examinations were made and the initial results of the surface SEM study are summarized in Figures above.

Comparisons with samples exposed in a pilot coal gasifier atmosphere were completed and reported in a COST 501/II action.

Above: *Scanning electron microscopy micrographs showing in plan the corrosion products formed on the alloys after exposure to 0.2% H_2S - 64% CO - 3.8% CO_2 - H_2 bal at 600°C for 100h.*

Table below: *Alloy Composition (wt%).*

Element	Alloy				
	MA 956	HR 160	HR 3C	Cronifer 45	26/37/3
Fe	bal	bal	bal	bal	bal
Cr	20	28	20	27	26
Ni	--	--	20	45	37
Co	--	29	--	--	--
Al	4.5	--	--	--	--
Si	--	2.75	--	3	--
Y	0.5	--	--	--	--
V	--	--	--	--	3
Nb	--	--	trace	--	--
N	--	--	trace	--	--

High Temperature Corrosion Resistance of Alloys and Coatings

The performance of high temperature corrosion resistant coatings on superalloys depends on their chemical composition. At temperatures encountered in the gas inlets in gas turbines, the Al content of MCrAlY coating is depleted by oxidation and by interaction with the base material.
The aim of this study was to improve the lifetime of MCrAlY coatings by increasing the Al content. The vacuum plasma spray technique was used to deposit an Al film on the surface of an existing CoNiCrAlY coating. Aluminides were then produced by thermal diffusion under vacuum or in a controlled atmosphere.
Chemical and physical analysis of the aluminized MCrAlY coatings enabled the thickness of the Al film to be correlated with the temperature and time of thermal diffusion treatment, and enabled characterization of the structure and composition of the new phases produced.

The beneficial effect of an Al addition on the oxidation resistance was confirmed by high temperature oxidation tests. The effect of the Al enrichment of the MCrAlY plasma sprayed coatings was to:

1) increase the amount of Al, the sacrificial element, on the external surface of the coatings and;
2) increase the density of the protective layers, thus sealing the existing pores by means of liquid Al diffusion.

The coating chosen for this work was the NiCoCrAlY alloy, Amdry 995. The base materials employed were the superalloys UD 520, IN 739 and Nimonic 80A whose compositions are given in Table on page 8. Air plasma spray (APS) and vacuum plasma spray (VPS) techniques were employed to deposit the films on NiCoCrAlY and Al. These techniques are economically feasible for gas turbine inlet applications.

Table below: *Chemical composition of base materials and coatings.*

Material	Amount of element (wt. %)									
	Ni	Co	Cr	Al	Ti	Mo	W	Ta	C	Y_2O_3
UD 520	balance	12	19	2.3	3.2	6	1	-	0.65	-
IN 738	balance	8.5	16	3.4	3.4	1.75	2.5	1.75	0.17	-
Nimonic 80A	balance	-	20	1.4	2.4	-	-	-	0.08	-
Amdry 995	balance	39	21	8	-	-	-	-	0.8	0.6

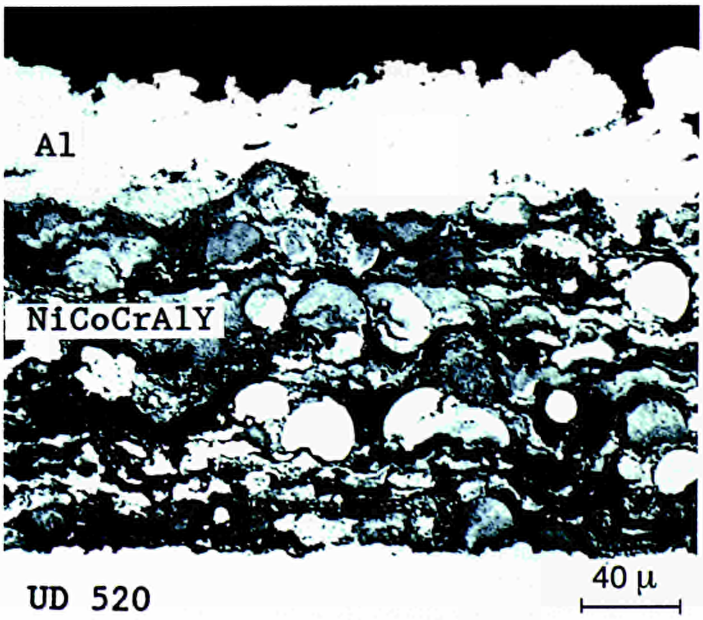
Oxidation Testing in Air.

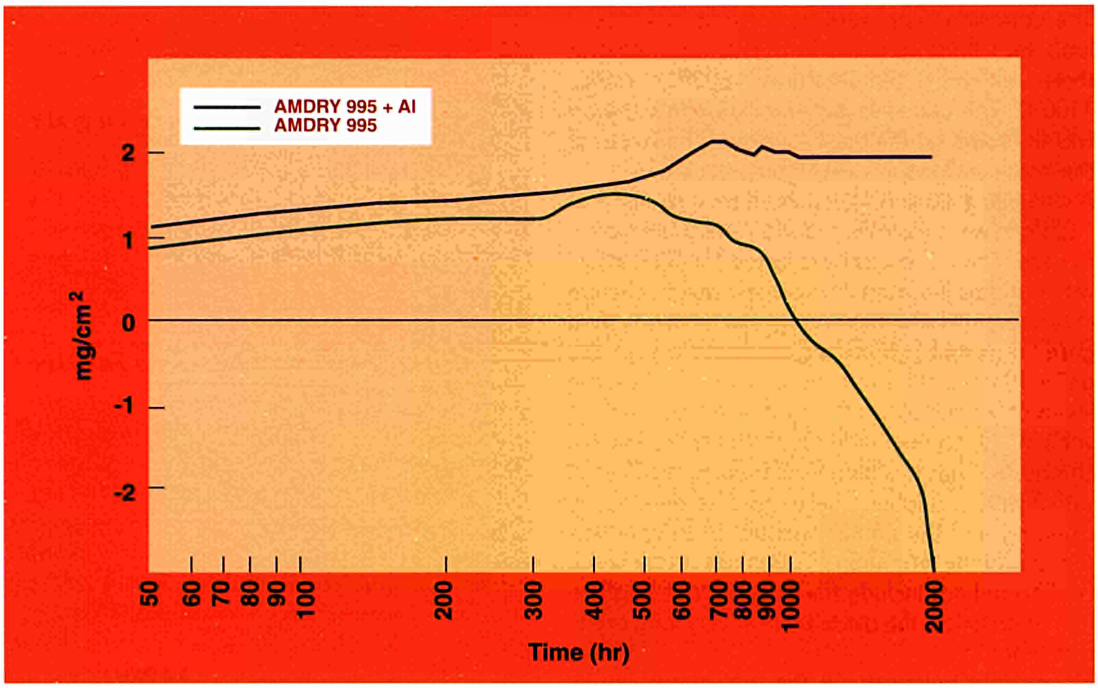
Cylindrical samples of 10mm diameter and 50mm height with rounded heads were completely coated with Amdry 995 using a VPS process. The samples were vacuum heat treated at 1080⁰C for 1 hour. Following this, the samples were covered with a homogeneous layer of Al. The surface alloying of Al on the substrate NiCoCrAlY was carried out under vacuum at 920⁰C for 3 hours. The corrosion tests were performed in air at temperatures of 1100⁰C and 1300⁰C. The variation in weight of the samples was checked every twelve hours. The specimens were taken from the furnace (air quenched), allowed to cool, weighed and then returned to the furnace immediately.

Results and conclusions

The results of this study can be summarized as follows:
- the surface roughness parameter was reduced by Al alloying,
- due to the Al diffusion process, pores in the NiCoCrAlY layer were sealed, i.e. the porosity level was reduced,
- the Al deposit must not exceed 33% of the NiCoCrAlY layer in order to avoid the formation of brittle, intermetallic compounds,
- there appeared to be no difference between the results for Al deposition obtained by APS and by VPS,
- thermal treatment for bonding contributed to a low porosity level and hence resulted in increased oxidation resistance,
- Al additions were very effective in reducing the oxidation rates of the porous MCrAlY coatings deposited by APS,
- at temperatures above 1100⁰C, the coating efficiency decreased rapidly.

Below: *Micrographic section of a coating consisting of Amdry 995 (APS) and Al (VPS) before being heat treated*





Above: *Cumulative weight changes (mg/cm²) vs time (hours) produced by air oxidation tests at 1100°C for NiCoCrAlY coatings and NiCoCrAlY with Al film VPS deposited.*

Protective effect of coatings on the corrosion behaviour of Ni based superalloys in gas turbine atmospheres

The corrosion behaviour on Ni based superalloys was studied in highly oxidizing atmospheres containing low amounts of SO_2. This atmosphere simulated the composition of combustion gases of turbines at high temperature in the absence of condensed phases. These gases were highly oxidizing and were expected to promote the formation of protective oxides on the surface.

However, they contained some pollutants such as sulphur that were expected to enhance corrosion problems. The Ni superalloys tested are intended for turbine blade applications in advanced gas turbines. To avoid or reduce the corrosion phenomena, overlay coatings have been proposed.

Most high temperature coatings rely on the formation of a protective oxide scale by interaction with the environment. The Ni based superalloys tested were: IN 738, IN 792, UD 720 and MA 6000 (ODS alloy). Corrosion tests were carried out on coated and uncoated samples. A protective coating of the NCrAlY type was used with the following composition (wt%): Ni 32, Cr 21, Al 8, Y 0.5, Co bal.

The same coating was applied, by vacuum plasma spray, to all the tested alloys. A coating thickness of about 250 µm was used.

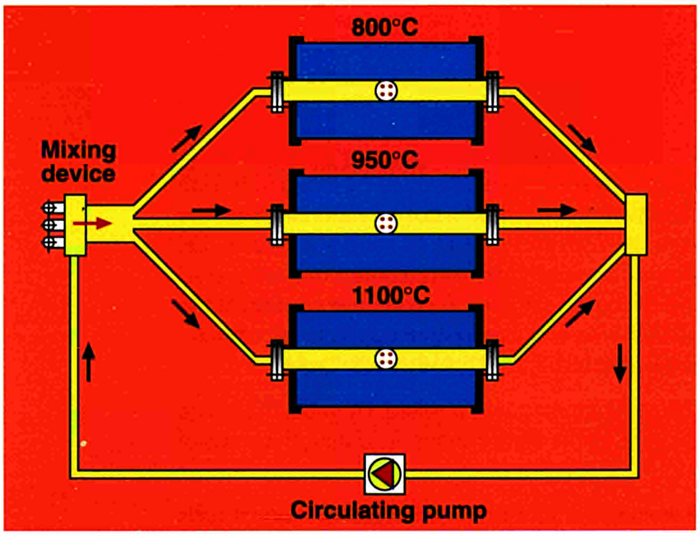
The samples to be coated were cylindrical rods with a diameter of 10 mm and length of 50 mm.

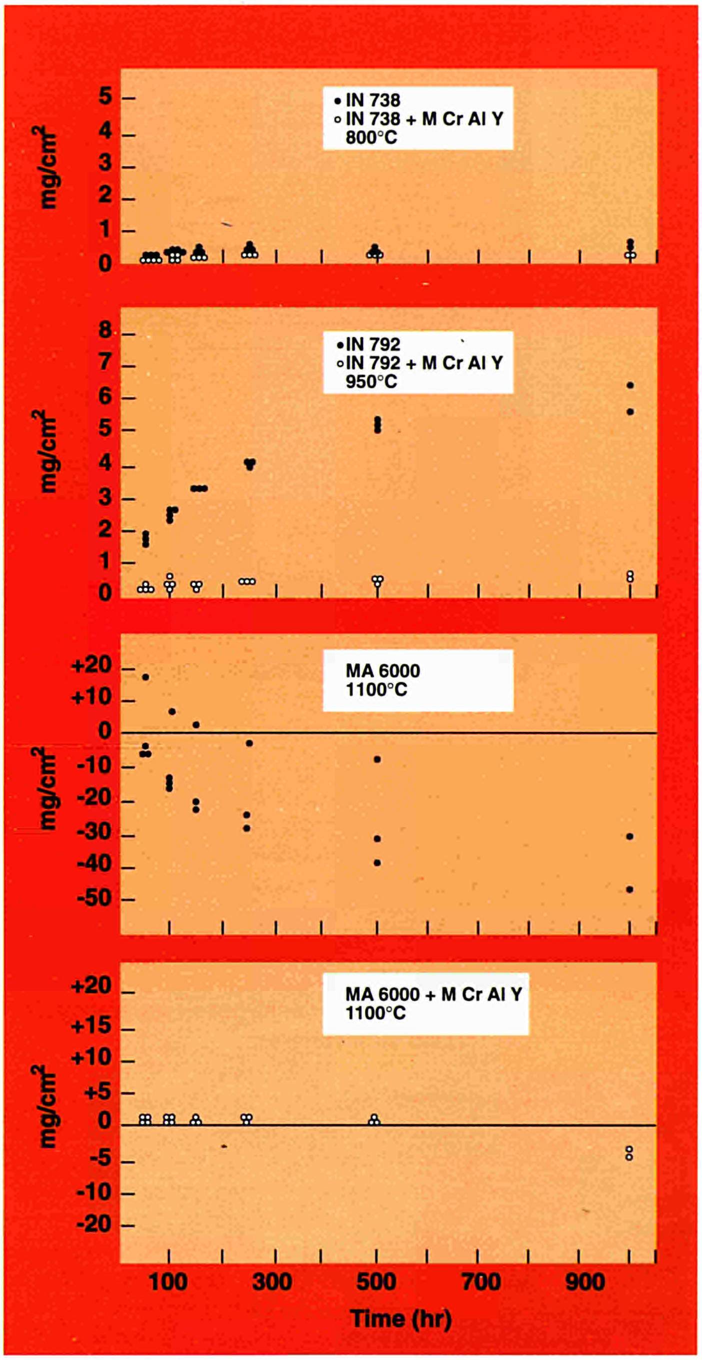
The ends of the rods were rounded to increase the adherence of the coating. The samples of the base materials were smaller rods with a diameter of 5 mm and length of 20 mm.

The corrosion tests were carried out in a closed loop with three furnaces in parallel operating at three different temperatures: 800°C, 950°C, 1100°C. The corrosive gas circulating in the loop was the same for the three furnaces. The layout of the installation is shown in Figure left below.

A circulating pump maintained a total gas flow of ~25l/min which corresponded to ~8l/min for each furnace. The corrosion tests lasted for 1000 hours with periodic interruptions to allow weight change measurements and microscope examinations. Two gaseous mixtures were used based on Ar as carrier gas + 10 vol% of O_2 with an addition of SO_2 at the levels of 0,1 and 0.3 vol%. The concentration of 0.1 vol% of SO_2 corresponded to the level present in a combustion gas resulting from burning residual oil with 2 wt% of sulphur. These mixtures were highly oxidizing and the partial pressure of sulphur was too low for the formation of sulphides on the surface. This did not include the possibility of sulphide formation below the oxide scale at very long exposure times.

Figure right below shows the gravimetric results obtained on specimens exposed in a single experiment.





Left below: *Schematic layout of the loop used for the corrosion experiments*

Right below: *Weight change measurements for alloys and coatings exposed to Ar, 10% O_2, 0.3% SO_2 gaseous mixture*

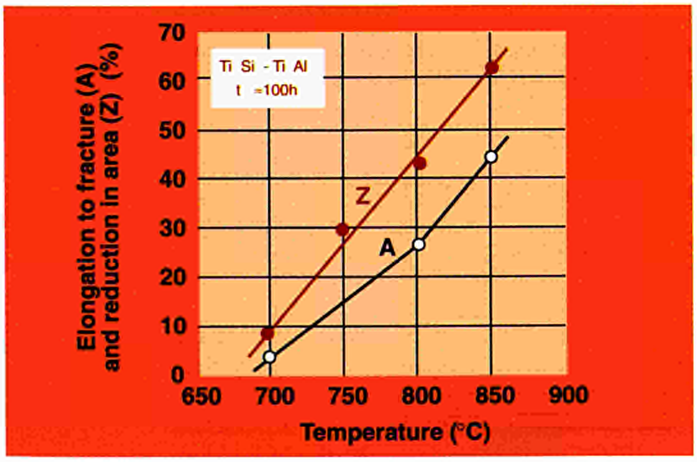
The corrosion undergone by the alloys tested was an oxidation process with the formation of friable mixed oxides on the surface which gave rise to spalling:

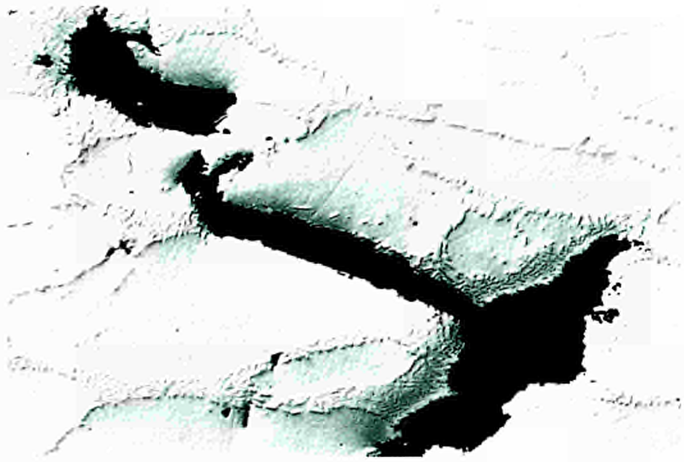
- The Ni superalloys IN 738, UD 720, and IN 792 showed the formation of a non-protective scale with precipitation of oxides in the alloy substrate. The corrosive attack increased with the temperature.
- The ODS alloy MA 6000 at 1100ºC formed a corrosion scale of mixed oxides during the initial period of the test. Subsequently a thin Al_2O_3 layer appeared on the surface.
- The MCrAlY anticorrosive coating applied on the surface of the alloys showed good corrosion resistance in all the conditions tested, apparently due to the formation of a thin layer of Al_2O_3 on the surface.

Characterization of Intermetallics and Refractory Alloys

The creep and creep damage behaviour of the intermetallic alloy Ti_5Si_3/Ti_3 Al has been investigated in the temperature range of 600-900ºC.
The material had been developed in the frame of a German high temperature material programme by the Max Planck Institute, Düsseldorf, who carried out a collaborative investigation into the RT properties and the microstructure. Although the material seems to be very promising concerning the excellent creep strength, serious problems arise from its poor creep ductility, which strongly decreases at lower temperatures. This is illustrated in Figure left below which shows the dependence of creep ductility on the test temperature for lifetimes of about 100 h.

The reason for the rather low ductility of intermetallics, which is generally observed below about 650ºC, is not yet understood and there are certainly various mechanisms to be considered. In the present case, a rather brittle behaviour of the eutectic phase has been observed as illustrated in Figure right below which shows that creep cracks propagated preferentially through the eutectic areas.
An additional problem for Ti_5Si_3/Ti_3 Al arises from the fact that above about 800ºC, severe oxidation starts due to the preferential oxidation of Ti. Thus, in spite of the good creep strength, the technical application of this material will probably be impeded by its inadequate ductility and oxidation resistance.



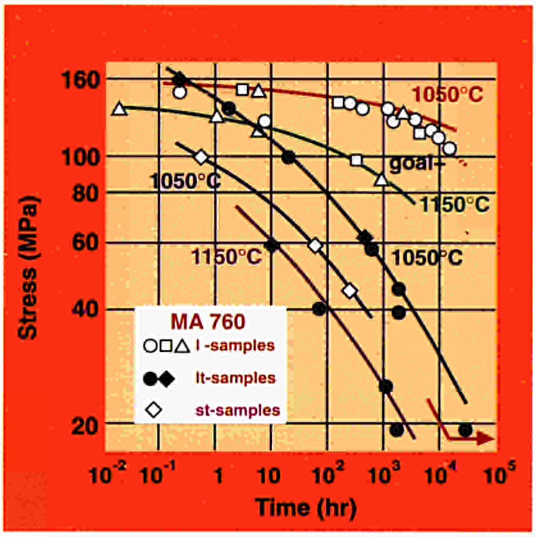


Below left: *Creep ductility versus temperature for Ti_5Si_3/Ti_3 Al. For all tests the lifetime was about 100h.*

Below right: *Creep damage mode for Ti_5Si_3/Ti_3 Al*

Characterization of H.T. Alloys

Investigation of the creep and creep damage behaviour of the newly developed nickelbase ODS superalloy MA 760 was completed except for a few long term tests. Excellent creep strength has been observed at 1050°C and 1150°C for samples oriented with the long grain axis parallel to the stress axis. Material with this orientation clearly satisfies the industrial requirement for a life of 10.000 h at 100 MPa and 1050°C which would enable it to be used for gas turbine blading applications. The anisotropic behaviour however results in a much lower creep resistance in the transverse direction as shown in Figure above. This is due mainly to a much lower creep resistance but partially also because of a lower creep ductility which, for the transverse direction, falls below 1% elongation to fracture. Moreover, investigations carried out at 1150°C have revealed an inadequate oxidation resistance. Thus the highest possible application temperature of MA 760 seems to lie around 1050°C. The inferior mechanical properties in the transverse direction need to be improved to enable it to be used for blading in large gas turbines.



Above: *Lifetime versus stress for MA 760 for different sample orientations. l-, lt- and st- denote the longitudinal, long- and short transverse direction.*

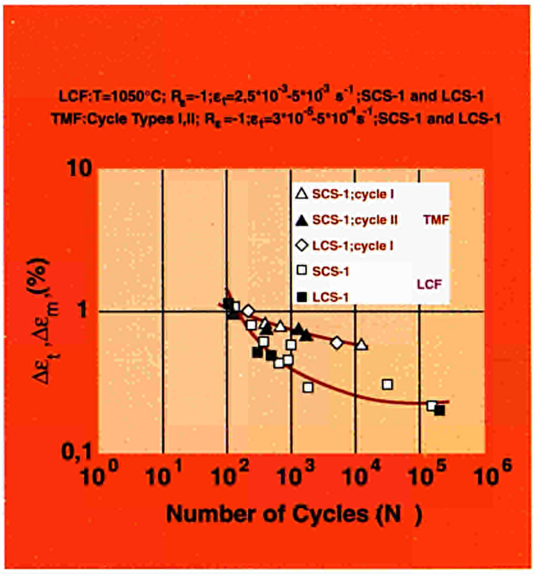
Investigation of the corrosion resistance of the ironbase ODS alloy MA 956 under deformation in a S-O-C bearing gas was completed for pre-oxidized samples. At 600°C the critical strain rate for oxide failure was about 0.6% total elongation independent of the strain rate. Detailed structural analysis revealed a clear picture of the damage mode and the corrosion resulting from scale fracture. Testing of the creep strength properties/corrosion interaction on untreated MA 956 was started.

Performance Improvement of Ceramics, Composites and Alloys

Alloys-Fatigue

Low Cycle Fatigue and Thermo-Mechanical Fatigue testing programmes on MA 760 were completed. These programmes were part of working Packages 1 and 5 of COST 501/2. Material of different extrusion size was compared, the fatigue specimens always being machined in the direction of extrusion. TMF tests with different minimum cycle temperatures, which mimic the actual service conditions of uncooled gas turbine vanes, showed no effect of T_{min} on the fatigue life when plotted in terms of the strain range. The fatigue life of TMF tested material (T_{min}=300°C and 550°C, T_{max}=1050°C) was compared with the LCF life at 1050°C as a function of the applied strain range as shown in Figure on page 13.

In the low strain range regime, typical of service conditions, the TMF life largely exceeded the LCF



Above: *Comparison of LCF (at 1050°C) and TMF lives of MA 760*

life, with much higher stress ranges being sustained in TMF.

The development of advanced mechanical testing equipment continued in the reporting year,focussing in particular on the commissioning of a multipurpose universal machine for the tensile, creep and fatigue testing of alloys and ceramics in the uniaxial mode under C-O-S type corrosive environments at high temperatures. The implementation of computerized testing continued with the development of software for the control of testing machines by means of dedicated computers.

The management of Working Package 5 (Prediction of life under simulated service loads) of COST 501/2, which involves 25 Universities and Research Institutes in Europe, continued during 1992.

Mechanical properties of monolithic ceramics and ceramic matrix composites.

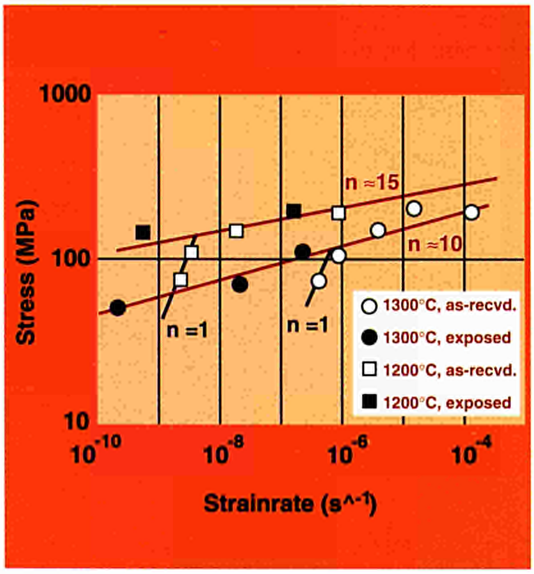
Engineering Ceramics

Two classes of material with an application potential as high temperature structural materials were studied: monolithic ceramics and continuous fibre reinforced ceramic matrix composites. Research concentrated on characterizing the mechanical properties and on studying the mechanisms of deformation, of damage and of failure, and of their relationship to the microstructure.

The monolithic ceramics investigated were a hot-pressed and a hipped silicon nitride. Long term uniaxial tests were made on the hot-pressed material. The residual strength, the toughness and the subcritical crack growth behaviour after exposure to a simulated (sulphidizing) industrial coal gasification environment at 1200°C and at 1300°C were characterized earlier by flexure testing under an inert atmosphere. In contrast to the flexure tests, the uniaxial tests on the as-received material (Figure below) showed a clear transition from a subcritical crack growth failure regime to a diffusion creep-dominated regime with decreasing stresses and longer lifetimes for both the test temperatures.

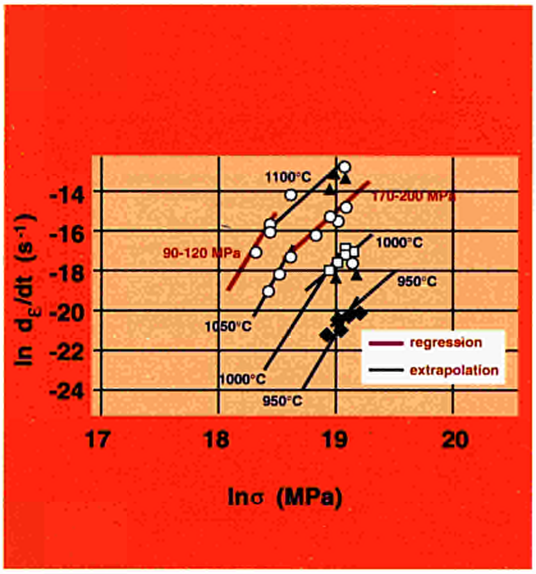
At 1200°C the transition occurred later than at 1300°C. For the exposed material the transition between the two mechanisms could not be observed

Below: *Comparison of the uniaxial steady state creep rates in as-received and pre-exposed hot-pressed silicon nitride*



in the experimentally covered range of stresses and strain rates. Microstructural investigations indicated that the observed change in behaviour from the as-received to the pre-exposed state was governed by crystallization of the amorphous glassy phase at the grain boundaries. The crystallization depended on the time and temperature history during pre-exposure.

Flexural creep tests in air and tensile creep tests in vacuum were performed on hipped silicon nitride at 1350⁰C and 1400⁰C with the aim of correlating the creep rate measured in the uniaxial tests with the deflection rate from the bend tests.



Above: *Minimum creep rate versus applied stress for a bidirectionally reinforced ceramic matrix composite tested in the uniaxial mode*

Ceramic Matrix Composites

The ceramic matrix composite consisted of a bidirectional reinforcement of cross woven alumina fibres in a silicon carbide matrix, obtained by chemical vapour infiltration.
The aim was to study the intrinsic, long term creep behaviour of the composite in the uniaxial tensile mode. For this purpose testing was performed under high vacuum with an oxygen partial pressure below 1.E-9 mbar.

Tests were made at temperatures in the range 950⁰C to 1100⁰C at stress levels above the first matrix cracking stress. All creep curves exhibited primary, secondary and tertiary creep stages.
A double stress and temperature dependence was observed for the stationary creep rate (Figure above). At high stresses these dependencies were identical to those measured on individual fibres.

At the lower stresses, the stress redistribution between the fibres and matrix, which occurred before saturation of the matrix cracking is attained, masked the true stress and temperature dependence. The extent of the tertiary stage, as well as of the rupture strain increased with decreasing stress. This was attributed to the statistical distribution of the in-situ fibre strength.

Research Area:
Reliability and Life Extension

Assessment of Research Area Progress and Achievement: Reliability and Life Extension

Modern installations use materials under very demanding conditions. It is therefore necessary to develop a good understanding of the behaviour of materials and structures under these conditions. Existing installations which have been in service for long times are frequently considered for an extension of their service life.
In both cases it is necessary to assess how these materials can fail, how the damage accumulated can be measured, and how their residual lifetime can be determined.
Four actions have been identified as the means of further understanding the problem:

- Experimental installations in which the operation conditions can be simulated, such installations may be complicated, especially if it is necessary to test components,
- Methods to measure and to quantify the damage, which develops during operation,
- Modelling of the defects generated by the operation and the influence of the defects on the mechanical properties,
- Evaluation of the models and the comparison of the prediction of the models with the results of the experiments.

The research area "Reliability and Life Extension" includes activities in each of these activities.

The project has concentrated on a few materials, some of which are relevant in other research areas of the programme. Such crosslinks allow a greater depth of understanding, and are of considerable benefit.
At this stage simple geometries have been selected in order to avoid introducing fabrication technology as a parameter, which would add complexity to the work. The facilities necessary for the simulation of service conditions, for example high temperature corrosion, creep, fatigue, thermomechanical cycling and cycling of fiber reinforced ceramics are now available. The development of methods to determine damage are being actively persued.

Optical and electrical methods for the determination of the crack growth in metals have been tested. High frequency ultra sound methods for the detection of cracks in ceramics are under development. Ultrasonic velocity measurements have been successfully applied to the determination of creep damage in steels. For a reliable prediction of life time it will be necessary to combine the results of several methods. First attempts have been made and they have delivered promising results. These are valid only for certain materials and have to be adopted for each new case. The work on modelling is at an early stage and will be developed next year.

Structural Performance of Advanced Materials

The overall aim of the project is to predict the mechanical performance of advanced materials for specific structural applications by means of experiments, modelling and validation testing. Two classes of materials are considered i.e. single crystal nickel based alloys for aero gasturbine applications, and continuous fibre reinforced ceramic matrix composites (CMC's) for aerospace applications. The project also involves the conception, design, construction and commissioning of new, advanced testing equipment.

T(hermo)-M(echanical) F(atigue) tests are applied to mimic the in-service fatigue behaviour of various bare and coated, single crystal nickel based alloys in the framework of various third party contracts. The associated modelling activity, which started in 1992 in the framework of the specific programme, attempts to predict the TMF stress response and the concurrent cyclic life. Existing knowledge in the field of the viscoplastic constitutive modelling of anisotropic materials has been reviewed and critically evaluated.

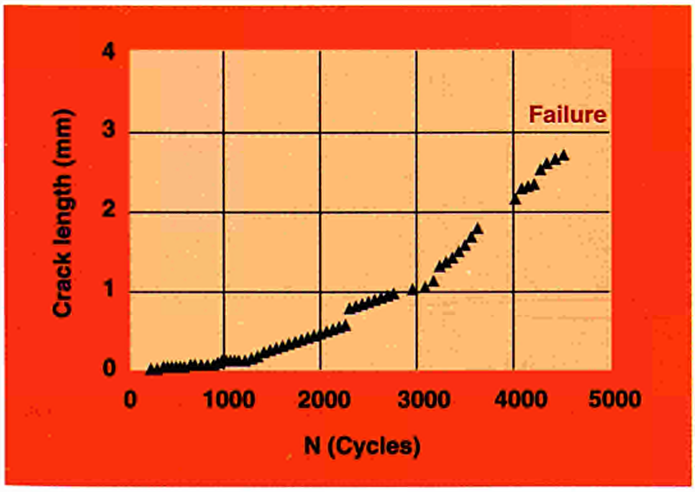
An FE code including a viscoplastic model for anisotropic single crystal materials has been acquired, and a collaboration with the Ecole des Mines de Paris, which developed the model, has been initiated with the aim of extending and improving it. The information required for this extension and for the lifetime modelling will be derived from three experimental projects defined in 1992 for execution in the framework of the specific programme.

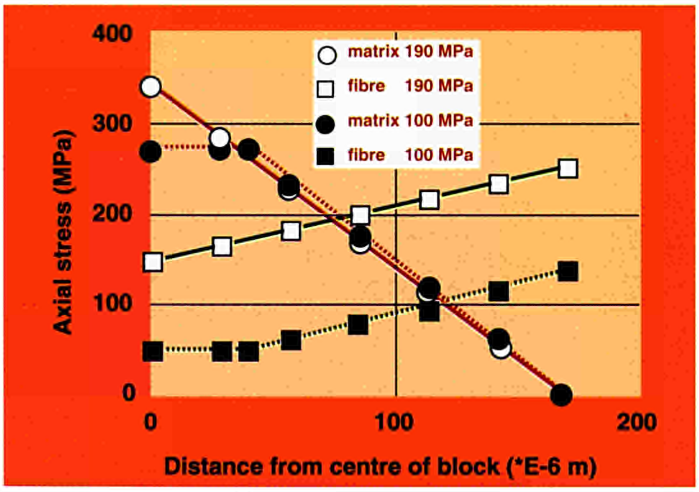
The projects relate to the evolution of the dislocation microstructure during TMF cycling, to determining the residual stresses in the sub-surface layers caused by the coatings, and to the evolution of damage developing in the form of microcracks, respectively.
Figure below left illustrates one level of information on the microcrack initiation and growth process which is accessible experimentally by means of the computer vision system used for the in-situ observation of the sample surface during TMF testing.

The modelling activity in the area of CMC's aims at rationalizing the results from creep tests and at providing a basis for creep life prediction.
A micromechanistic model for creep of 2D CMC's is under development, the starting point being an existing creep model for unidirectionally reinforced metallic matrix composites. The 1D MMC model is extended by taking account of the creep of the fibres, of the matrix cracking and of the ensuing load redistribution between the fibres and the matrix.

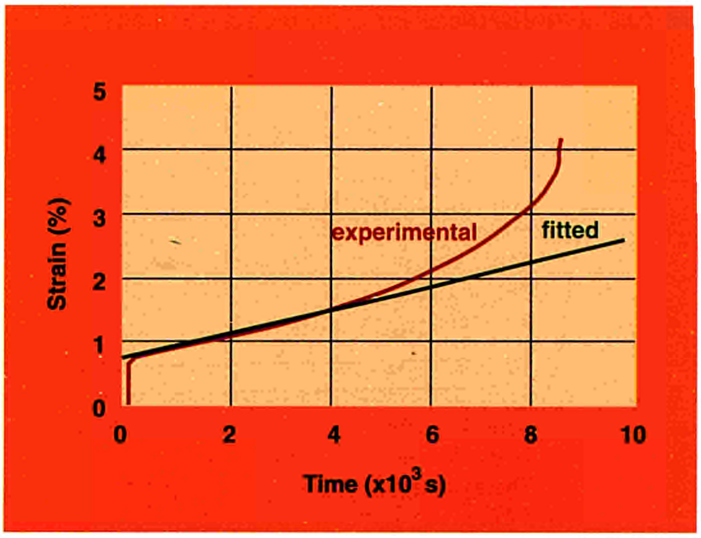
Below left: *Evolution of the length of the major crack measured in-situ on the surface of a coated, single crystal nickel-based alloy during TMF testing*

Below right: *Axial stresses in matrix block at 1100ºC and applied stresses of 190 and 100 MPa*





The stress profiles along the fibres and in the matrix are calculated using an improved shear lag model which considers the existence of three different regimes along the interface between matrix and fibre i.e. total detachment, slip, and total attachment. The model enables calculation of the axial stress distribution in both constituents of the composite for different nominal applied stress levels. An example is shown in Figure on page 17 below right for two applied stress levels. The results of the shear lag model are used as input to the uniaxial creep model of the cracked composite, yielding correct predictions of the primary and secondary creep rates, see Figure above. Work to extend the model to include the tertiary creep regime, and to creep in 2D CMC's continues.
A rig for the tension/compression testing of CMC's under vacuum was commissioned. A multiaxial tension/compression-torsion machine was installed and was used to explore the possibility of measuring crack growth along the interface of coated 4-point bending specimens.

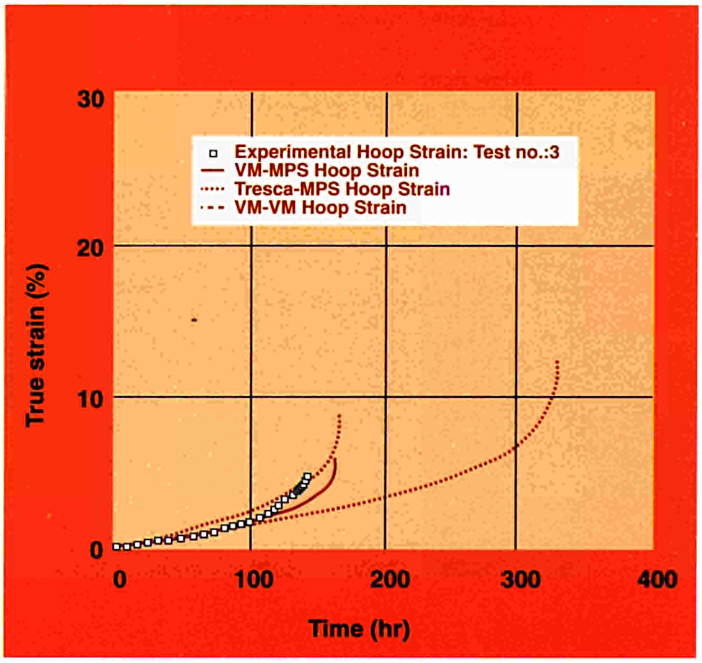


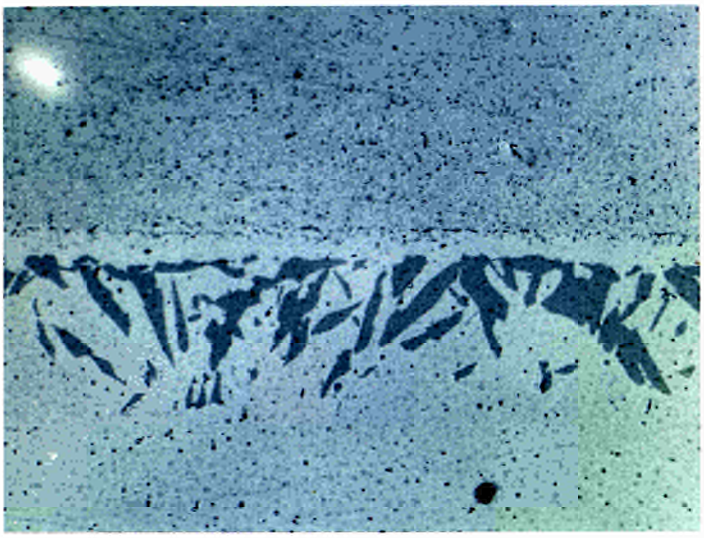
Above: *Creep curves at 1100°C and 190 MPa*

Component Integrity Testing and Evaluation

The activities associated with modelling the multiaxial creep behaviour of 2.25Cr1Mo ferritic steam pipe material (COST 501-II WP5) were successfully concluded. A continuum damage mechanics (CDM) model incorporating an element of primary creep was employed, which required the development of software routines to extract optimised material constants from a series of either constant stress or constant load uniaxial creep tests. The model was extended to cover multiaxial loading by considering the continuity of the damage parameter and by allowing the independent manipulation of the stress functions controlling creep deformation and rupture. The best correlation with results from the pressurised tubular component experiments was found for the case of a von Mises deformation and an MPS rupture failure criterion (Figure below).

Below: *Internal pressure tube result and model predictions*





Above: *Diffusion bond between MA956 tube and Nimonic 105 end cap*

In the study of the creep crack growth behaviour of 2.25Cr1Mo, a number of internally and externally notched tubular component creep tests were performed at temperatures of 550 and 600⁰C. The analysis of the creep crack growth behaviour was based on the limit load reference stress solution and the dependence of crack growth rate on the C* integral was evaluated.

Comparison of these crack growth results, and also of the stress rupture behaviour, with conventional compact tension (CT) specimen data showed good agreement in general, but has highlighted some potential limitations of the analysis. Work was started to investigate and overcome these limitations.
Work on the creep crack growth behaviour of 2.25Cr1Mo ferritic steel in high pressure hydrogen environments was delayed due to problems with the pressurising system. A major upgrade of the experimental facility to handle explosive gases at pressures up to 300 bar was almost completed.

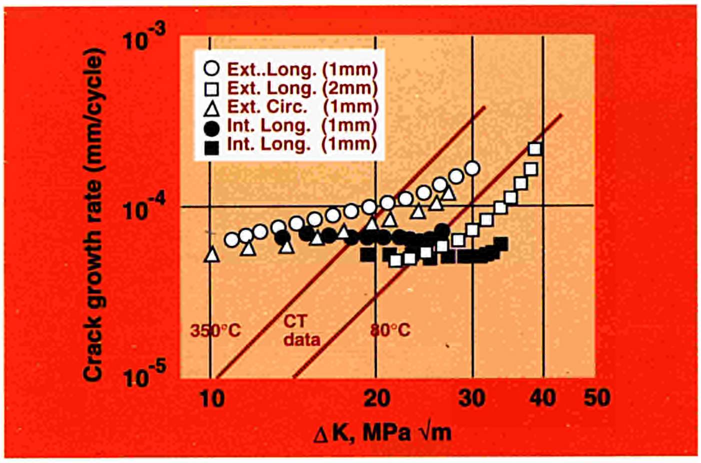
A diffusion bonding technique for joining high temperature alloy end caps to MA956 ODS alloy tubular sections was developed (Figure above). This techniques is important for the COST 501-II WP4 project where joints are required for the construction of a prototype heat exchanger and also to enable the creep properties of the ODS tube to be determined under multiaxial loading conditions in the laboratory. Initial pressure tests on these tubular test pieces indicated a sharp reduction in creep rupture lifetime compared to the manufacturer's published data for this material.
A Round Robin verification testing programme for the Internal Pressure Testing Code of Practice, covering some 12 laboratories, was initiated and preparations for the preliminary pressure testing phase were begun. The IAM part of this programme forsees multiaxial creep tests on 9Cr1Mo tubular material, which is the prime candidate material for the next generation of steam superheater components in the power generation industry.

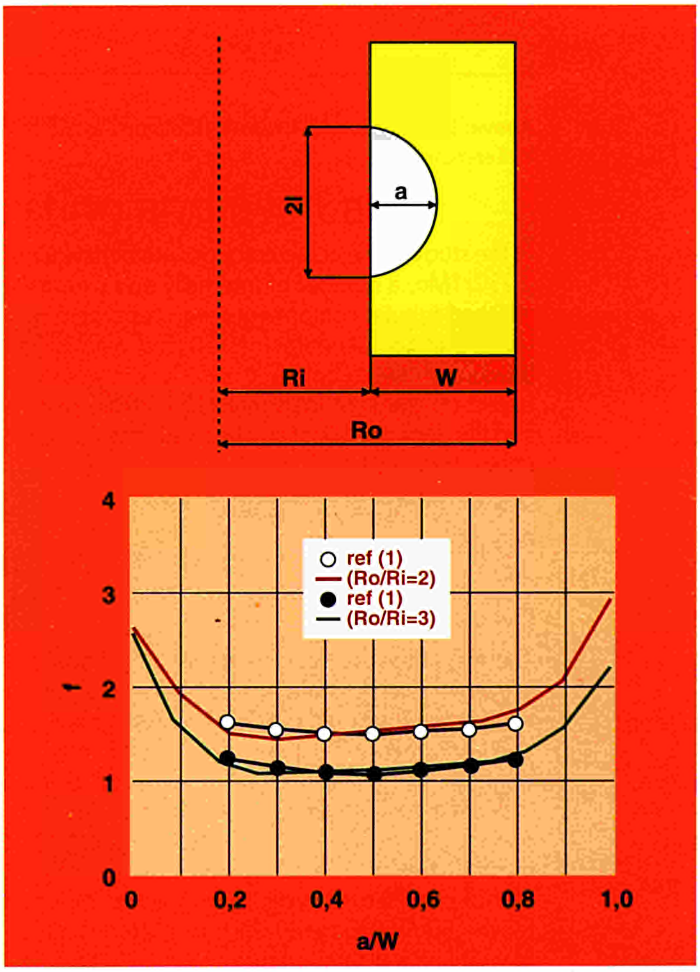
Thermal Fatigue of Components

The aim of the project is to numerically model the growth of a crack to failure in components exposed to cyclic thermal gradient fields and to experimentally verify this model for the case of the conditions seen by the first wall of a first generation Tokamak fusion reactor. The project therefore includes an in-pile rig in the HFR for the in-situ measurement of crack growth.
Out-of-pile thermal cyclic testing was completed for the 80⁰/350⁰C cycle which simulated the NET reactor first wall temperature cycle.

Under these conditions, stresses at the inside and outside walls reached similar levels at each half cycle, and it was found that the crack growth rates were apparently independent of the defect position (internal/external) or geometry (longitudinal/circumferential). For comparison of the thermal fatigue data with mechanical fatigue crack growth rates observed for conventional compact tension specimens, the stress intensity factors K were calculated using the superposition method applied to the finite element determined stress distributions.

The thermal fatigue crack grows into a decreasing stress field for most of the component life, but the crack growth rate dependence on ΔK was close to the compact tension data band for the temperature range investigated as shown in Figure above. Numerical modelling was performed with finite element (FE) and analytic methods. The PREDI-N code was developed to analytically evaluate the stress intensity factors of semi-elliptical surface cracks in cylinders. It uses a combination of the line spring model and the weight function method. In comparison to the FE method, PREDI-N offers flexibility and short calculation times. However, due to the assumptions and approximations made, its accuracy had to be verified by comparing its results with those from other sources. In Figure below the normalized stress intensity factors for an internal semi-elliptical surface crack in a pressurized cylinder were compared with the analyses by Tan and Fenner [1] using the boundary integral equation method. The PREDI-N results were within 10% and, over part of the range, even within 5% of the full analysis. These results were well within the scatter band of the result of FE calculations. Because the calculation was made for a thick walled cylinder, and given the observation that even for large crack depths a good agreement is obtained, the performance of the PREDI-N code was judged to be very satisfactory. Progress with respect to the irradiation aspect of the project is slow because of manpower shortage. The effort in 1992 concentrated on testing. The AC potential drop technique was selected for measuring of crack growth rate under irradiation. Testing revealed problems with respect to the shape of the spring loaded contacts of the crack growth rate measuring probe. These problems were tackled and solved. Additionally a computer program to calculate the theoretical relationships between crack depth and potential drop values was implemented.





Above: *Comparing the growth rates of various cracks (see legend) in a tube subjected to thermal-fatigue cycling with the crack growth rates measured isothermally on CT specimens*

Below: *Comparison of the normalized stress intensity factor f rs crack depth $^{a}/_{w}$, as calculated by means of the computer code PREDI-N, with literature data (see text)*

[1] Int. Journal of Fracture, 16 June 1980, pp.233-45

Flaw Sizing in Advanced Materials by Ultrasonics

The objective of the Ultrasonic Development Laboratory is to study and develop special ultrasonic techniques and procedures and to improve conventional ones. Three specific cases of flaw characterization, are reported.

* A high-frequency C-scan system working at up to 100 MHz was developed for the detection and sizing of small surface-breaking cracks in silicon nitride by means of leaky Rayleigh waves. Previously the maximum frequency that could be used was only 30 MHz. The depth of the investigated surface cracks, resulting from Vickers indentations, varied between 40 and 270 µm. Two focusing PVDF probes of 30 and 75 MHz were used. The C-scan images were complicated by the presence of the Vickers indentation but could be interpreted satisfactorily (Figure below left). The best results with respect to imaging and crack length measurement (see Table on page 23) were obtained with the 75 MHz probe whereas for crack depth estimation the best results were obtained with the 30 MHz probe (Figure below right).

Below left: *Typical C-scan of a surface-breaking crack, resulting from a Vickers indentation, obtained by means of leaky Rayleigh waves (length of white line along Y-axis is crack length)*

Below right: *Crack echo amplitude A normalized with respect to corner echo amplitude A1 as a function of crack depth a normalized with respect to wave length λR*

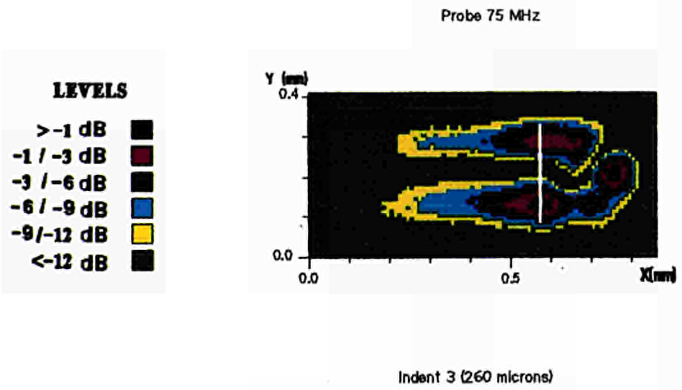
Further increasing the frequency may improve the performance of the technique with respect to imaging and crack length estimation even more.

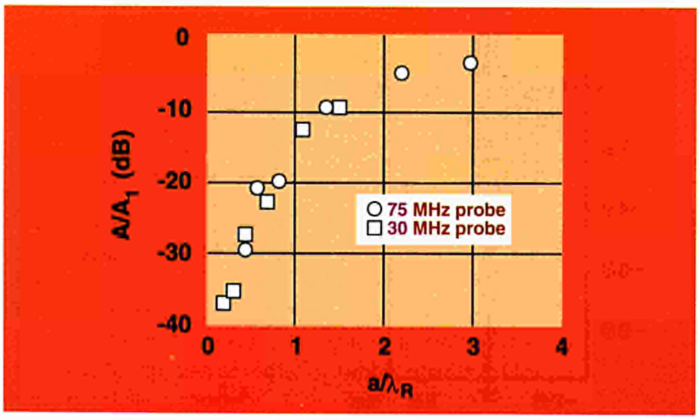
This would require, however, very good surface finish as the signals due to surface irregularities increase significantly with frequency.

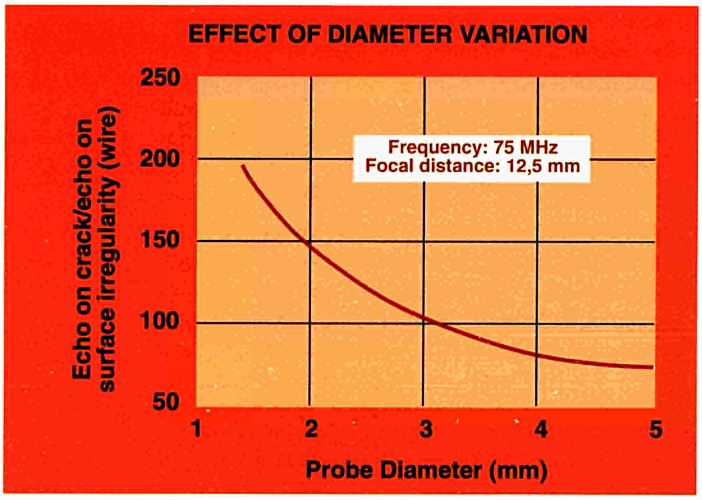
Work was started, using a model developed in our laboratory, to optimize the design of the probe in order to increase the ultrasonic responds to surface cracks compared with that to surface irregularities (Figure on page 22 above left).

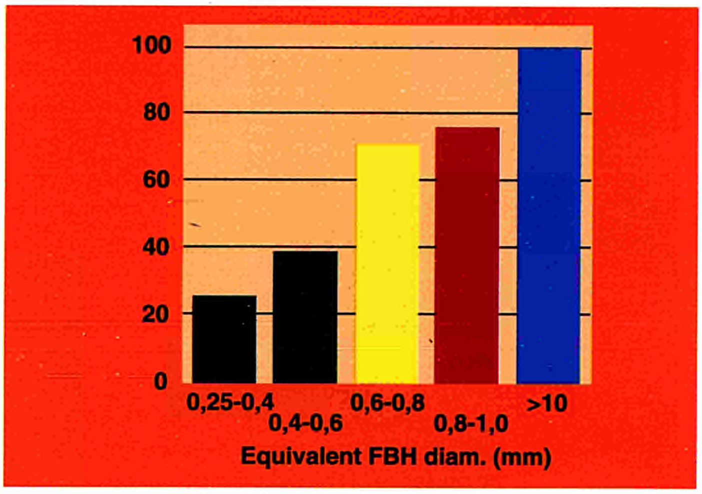
* One of the biggest problems in the field of non-destructive testing of large forgings is the difficulty of resolving clusters of small flaws.
We showed that both the sensitivity and the resolving power of focusing probes are significantly better than classical contact probes (Figure on page 22 above right). Focusing probes allowed us to detect flaws with an equivalent flat bottom hole diameter of less than 0.3mm at a depth of up to 100 mm in a large forging.

* The problem of detecting defects caused by impacts on laminates of carbon fibre epoxy resin composites and on sandwich structures was investigated. Figure on page 22 below left shows a C-scan image of the impact damage for different layers within a laminate whereas Figure on page 22 below right shows a C-scan image of impact damage in a sandwich structure. The resolution obtained in the C-scan images was 1 mm, which can be considered to be very good for this type of material.







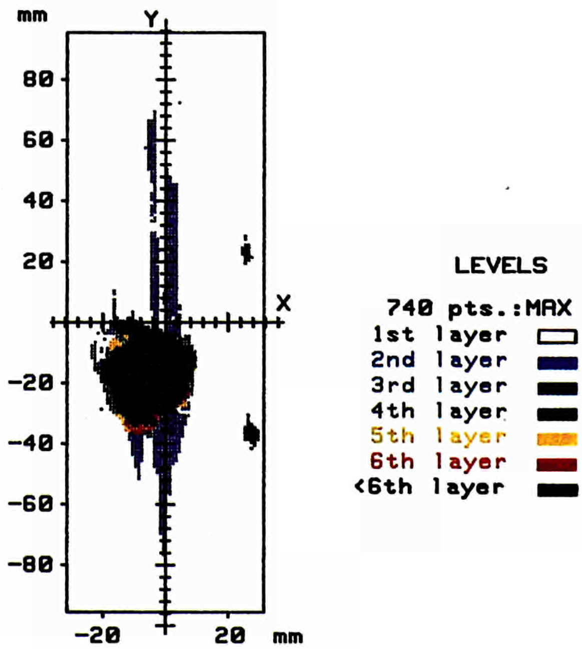


Above left: *Effect of probe diameter on ratio echo crack/echo surface irregularity (obtained by modelling)*

Above right: *Number of flaw indications obtained with classical contact probes with respect to total number obtained with focusing probes in a large forging (%).*

Below left: *C-scan of impact damage within a laminate of carbon fibre - epoxy resin composite.*

Below right: *C-scan image of impact damage in a sandwich structure.*



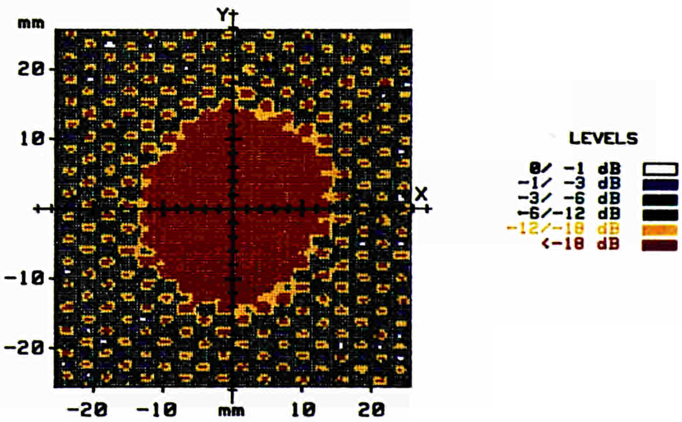


Table below: *Ulrasonically determined crack lengths compared with real crack lenghts*

Crack	Crack length measured by SEM (µm)	Crack length determined using 30 MHz leaky Rayleigh waves (µm)	Crack length determined using 60 MHz leaky Rayleigh waves (µm)
1	540	470	530
2	400	400	400
3	260	280	270
4	150	280	180
5	110	400	140
6	80	320	130

Performance Improvement of Composite Materials

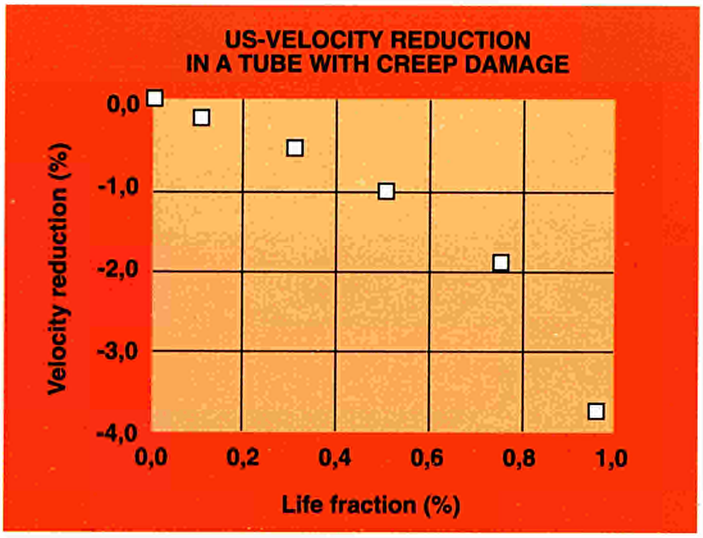
This project aims to develop methods of identifying and describing the lifetime determining defect state in materials with a designed microstructure. Microstructural damage was evaluated using metallography, the lifetimes of materials under creep were predicted using material models, the creep damage behaviour of the ODS-alloy MA 6000 was investigated, and the fatigue properties of austenitic stainless steel AISI 316L were improved by laser surface treatment.

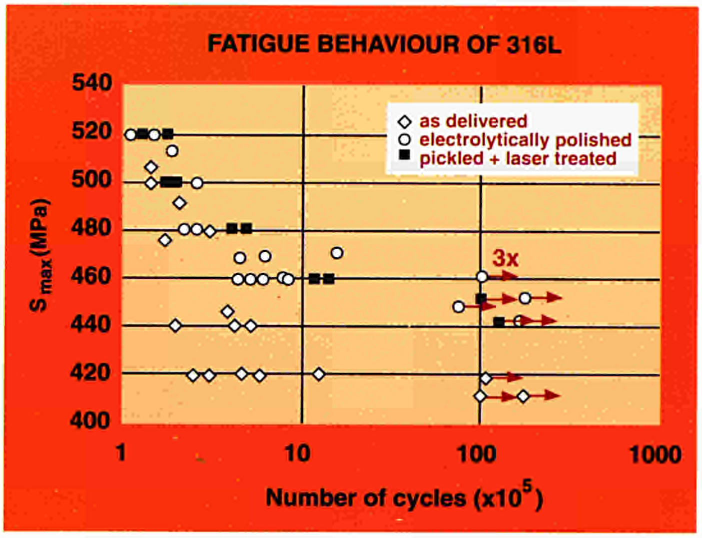
DAMAGE EVALUATION AND LIFTIME PREDICTION

Quantitative Metallography

The metallographic evaluation of microstructural damage (e.g. by the replica technique) for the remanent life assessment of components is widely accepted by the licensing authorities. However, quantitative results are required in order to use this method in combination with material models for lifetime prediction. By considering damage mechanisms in different materials, distributions of intergranular microcracks turn out to be of particular importance. We therefore investigated how mechanically meaningful damage quantities can be derived from metallographically defined parameters such as the 'A'-parameter. The 'A'-parameter is defined as the number fraction of cracked grain boundary facets intersecting a straight line parallel to the applied stress. By mathematical modelling based on stereological considerations, a relationship between statistical measures of the microcrack distribution and the mechanical damage parameter were derived. This relationship indicated which statistical parameters are relevant. In general, these parameters must be determined by image analysis. However, these results may serve as a starting point for developing and justifying simplified approaches which are less time consuming.

The results were applied to creep damage in AMCR 0033 and to low alloy ferritic steels in the framework of COST 501-II WP5C in order to provide experimental evidence for the relationship between different methods for damage determination and for the use of metallographic results in lifetime prediction models.





Above left: *Change of the ultrasonic wave velocity for a transverse wave travelling across the tube wall made of AMCR-0033, internal pressure: 43.2 MPa, inner radius 12.5 mm, wall thickness 4.5 mm*

Above right: *Fatigue behaviour of laser surface treated 316L stainless steel. Smax: maximum stress, R=Smin/Smax = 0.01*

Models for Lifetime Prediction

Creep testing and damage measurements in AMCR 0033 have produced a body of data suitable for modelling by viscoplastic constitutive equations. By including a Kachanov type damage variable which can be related to the microstructural damage state (a low concentration of intergranular microcracks), the uniaxial creep behaviour could be accurately modelled. Using a multiaxial version of the constitutive quations, the possible damage evolution in components could be calculated. With this it was possible, in addition, to simulate damage measurements: i.e. to determine theoretically the ultrasonic velocity change for a signal traversing the wall of a tube, for example, see Figure above left. This enables the effect of damage on NDE-measurements to be assessed for idealised conditions for a screening of possible areas of application. It also illustrates how to use microstructurally based damage variables in constitutive equations for lifetime predictions.

ODS Alloy MA 6000

Creep tests on MA 6000 were performed at 750°C. The machines were upgraded to testing temperatures of up to 1000°C. In collaboration with CNR-ITM creep tests at 1050°C were initiated. Post-test inspection by metallography, ultrasonic measurements and hardness measurements were begun. The stress to rupture curves obtained so far do not show much scatter.
It was observed that ultrasonic techniques such as C-scanning and time of flight measurements were strongly influenced by microstructural inhomogeneities in the grain structure.
This indicates that simple correlations such as are found in steels between ultrasonic velocity measurements and the damage state are not possible for MA 6000.

FATIGUE OF SURFACE TREATED MATERIALS

The surface modification by laser remelting is a technique with potential to improve the high cycle fatigue properties in regions of components where stress concentrations occur.
The fatigue behaviour of 316L stainless steel with and without laser surface treatment was investigated in order to optimize the surface modification process by determining the principal process parameters.

The specimens were cut from a laminated, 2mm thick steel plate. Details of the subsequent laser surface treatment (CO_2-laser, wavelength 10.6μm) and of the microstructural aspects of the surface changes are described in the frame work of the surface modification project.
The fatigue tests were conducted under load control on a servohydraulic testing machine with a frequency of 30Hz. Specimens that did not break after 107 cycles were below the fatigue limit.
The results were compared with those obtained from a set of electrolytically polished reference specimens.

It was apparent that the pretreatment of the specimen surface in order to obtain a sufficiently low reflectivity in the region of the laser wavelength had a large influence on the fatigue properties. Sandblasting was used to produce a homogeneous surface that absorbed the laser energy efficiently. By the retention of a small number of corundum particles and subsequent remelting, however, nucleation sites for fatigue cracks were created resulting in fatigue limits comparable to those of the as delivered state (Figure on page 24 above right).

A pretreatment by pickling and laser remelting using slightly modified process parameters yielded fatigue properties corresponding to those of the electrolytically polished specimens (Figure on page 24 above right).

The results so far suggest that changing the laser processing parameters, such as the beam power, the length of pulses, overlapping of melting paths, may lead to a further improvement in the fatigue properties.

Research Area: Measurement and Validation Methodologies

Assessment of Research Area Progress and Achievement: Measurement and Validation Methodologies

The development of new and improved measurement and validation methodologies for Advanced Materials is one of the principal tasks of the Advanced Materials programme. This research area combines activities which address a number of novel measurement, testing and detection methods needed to characterize and quantify the features of advanced materials such as metallic alloys, monolithic ceramics, composites, coated materials and joints. The objectives of validation imply the need to develop quality and reference characteristics of these methods, the establishment of which is an important step towards standardization. The research area indeed has a number of links with standardization activities, so that it can be considered as essentially pre-normative in character.

Pre-normative research is an objective for the Institute which is planned to increase in importance and scope. The forthcoming adoption of the 4th Framework Programme for Community R&D is expected to define significant pre-normative tasks for the JRC, and the IAM is preparing to act as the focal point for the materials field which may have a pilot function within the JRC. This research area "Measurement and Validation Methodologies" is one of the lines from which the future pre-normative research activities of the Institute should grow.

Its assessment is particularly relevant with regard to this involvement which has already reached a significant level. The characterization of these activities as pre-normative necessarily implies that the closeness to standardization can be variable.

This is also anticipated for the future scope of work which will be based in the Institute on a horizontal concept in order to make all available competences and facilities useful for this objective.

Near-standardization work for the present programme is particularly represented by the PISC and PISC successor activities which include supporting and advisory functions to codes and standard bodies at national and international levels and which have achieved the validation of procedures and models of world recognition (ASME) in international cooperation. Pre-normative research on thermal cycling and thermo-mechanical fatigue on the other hand addresses test methods too complex to standardize, where a world-wide (VAMAS, ASTM) pragmatic agreement exists to use the non-standard methods in a quasi-standard manner, proving a high interest in the presently unachievable standardization. A third group of methodologies, X-ray spectroscopy, reflectometry and fluorescence methods for residual stress and film thickness non-destructive measurement, neutron scattering for residual stress measurement and positron annihilation spectroscopy for thickness and defect determination, are at very early stages of pre-normative research likely to require much more time to standardization than the first two.

Despite the variable pre-normative status of these activities, their "research push" on standardization remains significant and has a strong effect on the research-standards interaction which is a current concern of the Community.

Materials Ageing and Degradation Monitoring, Completion of PISC

Introduction

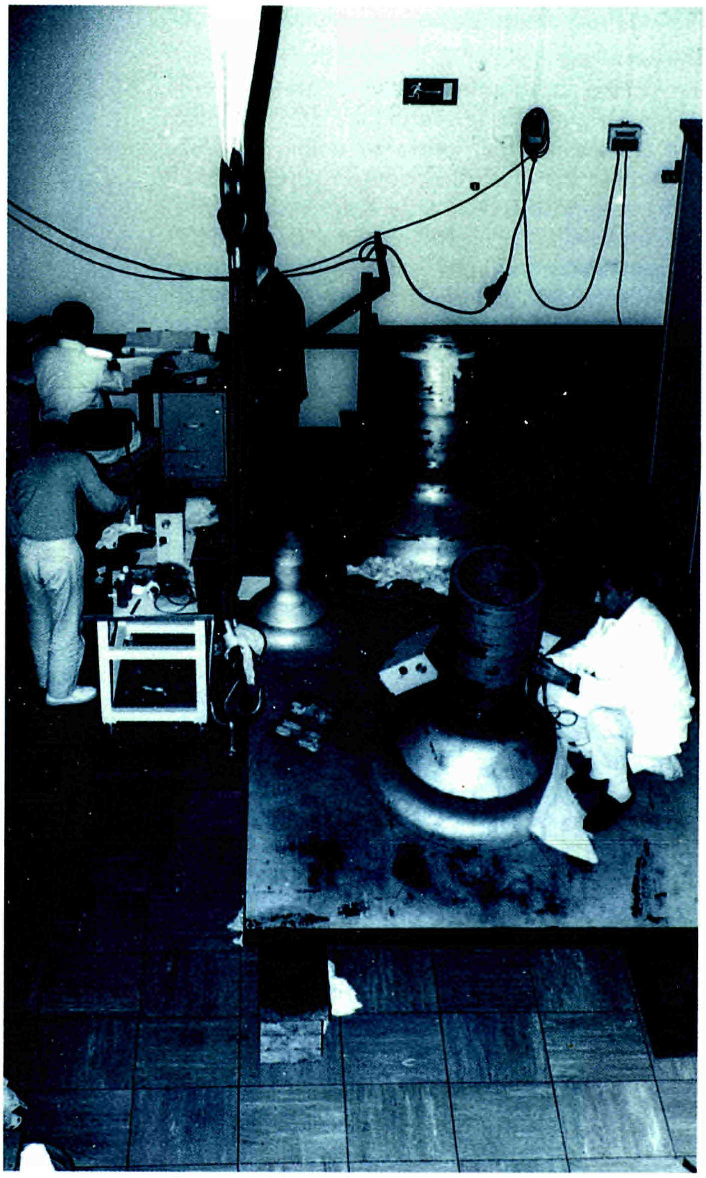
The PISC Programme (Programme for the Inspection of Steel Components) has the general objective of assessing procedures and techniques in use for the inspection of pressure components such as vessels and piping using Non Destructive Examination (NDE) techniques. The series of projects for the Inspection of Steel Components carried out since 1974 under the auspices of the CEC/JRC and the OECD/NEA is a major international effort to better assess the capability and reliability of Non-Destructive Inspection procedures for structural components. Three projects are centred on the Joint Research Centre which, in its roles of Operating Agent and Reference Laboratory, manages the programme and provides with the participants of EC countries approximately 60% of the programme funding; the other 40% comes via contributions in kind from the non-EC participating countries. OECD/NEA provides the Secretariat of the PISC Managing Board, consisting of representatives of 14 countries (8 EC and 6 non-EC countries). The programme is now at the end of its third phase (PISC III). The experience and expertise developed in the PISC exercises contribute significantly to the development of inspection qualification concepts and in particular to performance demonstration standards.

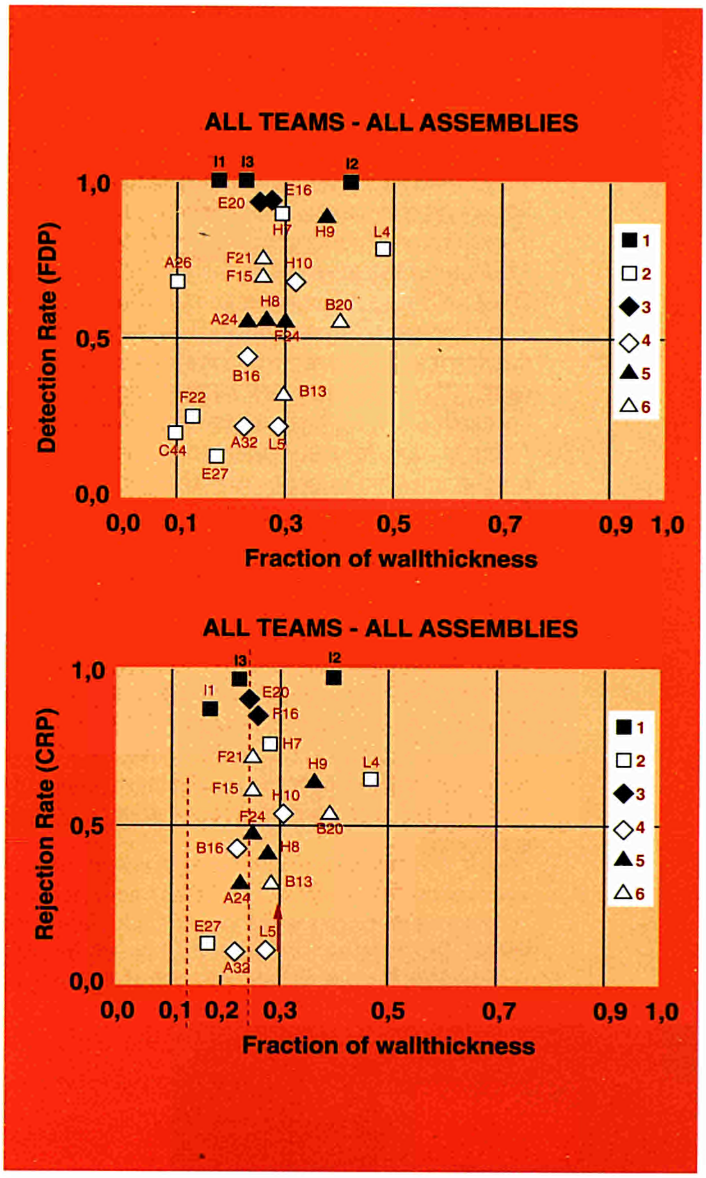
Below: *Trepanned sections extracted from the full scale vessel at MPA Stuttgart for full certification of the flaws*

PISC III Programme Status

The present stage of PISC III work is that Round Robin Testing activities have been, or are presently being, performed in several of the PISC III Actions. The uncertainties that prevailed during 1986/87 concerning the level of budgetary support have partly been resolved by accepting delays for the AST (Austenitic Steel Testing) and for the SGT (Steam Generators Tubes Testing) actions. Resources from the CEC and from participating organizations (participation to inspection, and contribution in kind) were made available to carry out the essential elements of most of the planned actions. The PISC III programme will close in December 1993. The status of work and the first trends shown by results of the major actions are given here under.

Action No. 2 (Full Scale Vessel Tests) validates the results obtained by procedures in the PISC II exercise but using more realistic inspection conditions: the installation available at the Staatliche Materialprüfungsanstalt (MPA), Universität Stuttgart, Germany was used. Phase 1 results, concerning the sizing of selected defects in the full scale vessel components are being evaluated; sizing capability exists on simple planar flaws such as fatigue cracks. Eight organizations participated in Phase 2. This phase was aimed at the validation of ASME type procedures by an international team using an ISI automatic scanner offered by RWE and MAN to PISC for the period of the exercise (Sept. '89 to April '90). Destructive examination is performed by trepanning flawed areas of the vessel (Fig. below).





Left above: *Nozzle and Safe-end of a BWR pressure vessel as used in PISC for the Round Robin Test on Safe-ends inspection. Inspection by a Japanese team at the JRC Ispra Establishment*

Right above: *Inspection performance as a function of flaw size and flaw family (four safe-ends areas)*

LEGEND

1. Branched artificial cracks in the ferritic steel.
2. Flaws in the weld material.
3. Flaws in the wrought material.
4. Flaws (cracks) in the transition: weld-wrought austenitic steel.
5. Flaws (cracks) in the transition: weld (or buttering) - ferritic steel (or primary piping).
6. Flaws (cracks) in the buttering or clad material.

The large data base assembled during the exercise is being evaluated. A large amount of support was given by Germany to enable the work on Action 2.

Action No. 3 (Nozzles and Dissimilar Metal Welds) completed in March 1991 the round robin tests of 4 safe- ends representing some of the most difficult technical aspects of In-Service-Inspection. In this work a Japanese- Italian BWR assembly of nozzle plus safe-end; an American BWR assembly with two nozzles and safe-ends and a Spanish PWR safe-end were inspected in 13 countries (Figure left above on page 30).

The results evaluated show that capability exists for safe inspection of the configurations considered. The major conclusions on the inspection of the safe ends are summarised below:

- In general, the performance in detection and in correct sentencing of flaws in these safe-end inspections was somewhat disappointing overall, however it was possible to identify some procedures that did show the required levels of effectiveness (Figure right above on page 30).
- There is a general difficulty in detecting and evaluating flaws in the fused material of the weld or buttering (Figure above).
- For effective results recording must be made at a low recording level.
- Mechanised inspection at high sensitivity and low recording levels appears to give the most effective results under industrial conditions.
- Detection is uncertain for flaws less than 5% of the thickness.
- The good performance of the immersion focusing probes was noted.

Action No. 4 (Austenitic Steel Testing) applies the PISC II methodology to the primary circuit piping of LWRs. Round robin tests for the capability assessment and parametric studies started in 1990. Twenty five teams have participated in one or more phases that will extend up to mid 1993.

The wrought-to-wrought pipe sections are presently being destructively examined in Japan. The circulation of Assembly 51 (wrought-to-cast, Figure on page 32 above) is well advanced as is the cast-to-cast RRT (Assemblies 41, 42 and 43, Figure on page 32 below).

INSPECTION PERFORMANCE BY PROCEDURE FAMILY
all assemblies

CODE	MFCRD	MFCRR	MFDF	MCRF	PROCEDURES
A	0.27	0.15	0.57	0.56	Manual Scan
B	0.39	0.33	0.57	0.70	Automatic Scan
C	0.30	0.23	0.64	0.73	Recording at Noise level
D	0.44	0.34	0.59	0.63	Recording at 10 to 25% DAC
E	0.17	0.03	0.37	0.34	Recording at 50% DAC
F	0.06	0.00	0.69	0.75	Manual and Noise level
G	0.43	0.36	0.61	0.72	Automatic and Noise level
H	0.20	0.00	0.89	0.71	Focussing Probes (high sensitivity and Scanning from inside)

Above: *Average correct rejection rate of each procedure family as a function of the average false call rate in rejection*

Above: *PISC III Assembly No. 51: full scale mock-up of a PWR primary circuit section*

Below: *Modular assemblies 41 and 42 used for the RRT on austenitic primary piping inspection*

A reliability exercise has been organized in the USA and will be repeated in Europe in 1993 on pipe sections coming from nuclear plants. Parametric study blocks have been made for study of the influence of metallurgical structure and of flaw characteristics whilst the studies on the influence of the counterbore has been concluded.

Action No. 5 (Steam Generator Tubes Testing) involves, in its present phase, round robin tests of individual tubes of steam generators containing realistic and artificial defects. Thirty teams from 11 countries participate in the RRT planned to last until June 1993.

Action No. 6 (Mathematical Modelling on NDE) has the objective to validate experimental mathematical models and perform parametric studies in order to assess the importance of defect characteristics. The results of the first validation exercise on three models have been published and further models are now being studied.
A new call for participation is aimed at the prediction and explanation of the Safe-End inspection results (Action 3 here above).

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. A first assessment of the results has been presented which showed that these tests could identify and demonstrate various aspects of unreliability such as the importance of the calibration stage, the personality of the operator, the duration of his involvement in a particular series of trials, and the ambient conditions.

Action No. 8 (Support to Code and Standard (C/S) Organizations) proposed by the Management Board and approved by OECD-NEA and CEC-JRC gives direct support through the PISC group of ex-

perts to C/S organizations. Such a proposal has been supported by all PISC members and involves three aspects:

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

Conclusions

The PISC III programme is proceeding as planned, until its conclusion in December 1993. The final PISC Symposium is planned to be organized at the end of March 1994.

Along with the PISC cooperative exercise example, proposals of new networks were made by IAM. They address the three traditional components of structural integrity: Materials properties, Fracture Mechanics and NDT. The first one, dealing with aspects of aged materials expertises and studies was called AMES:

AMES

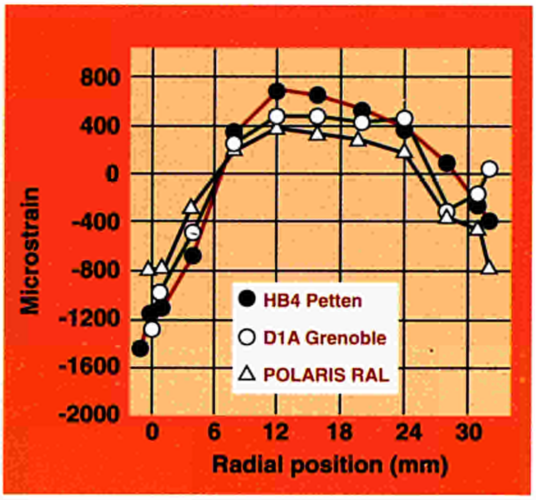
Although great progress has been made in understanding irradiation and thermal degradation of Reactor Pressure Vessel (RPV) steels, many aspects are not yet fully understood.
In particular the question of the qualification of remedial measures such as annealing and re-welding requires further work.

The international effort of the IAEA Working Group on Nuclear Plant Life Management provides contacts between institutions working in the field. Also the IGRDM (International Working Group on Radiation Damage Mechanisms for Pressure Vessel Steel) enables the exchange of information and collaboration for fundamental studies in this area. There remains, however, the problem of developing and maintaining a set of complementary capabilities in Europe.
There is in Europe also the need to create a focus for interaction with organizations in the former Soviet Union in the field of RPV materials condition assessment and annealing.
A network has been created to bring together the organizations in Europe that have the main capabilities on RPV materials assessment and research.
It has the following objectives:

. Validation and establishment of safe limits for mitigation and amelioration measures (annealing, etc.); . Formulation of a microstructurally based model capable of predicting the effect of annealing and re-irradiation;
. Validation of the applications of novel techniques, including reconstructing specimens, miniature and in-situ mechanical test procedures and also advanced microstructural techniques for RPV condition assessment for use in the longer term.
. Maintenance of a European expertise and resources for RPV condition assessment and remedial action;
. Participation in collaborative programmes with organizations of the former Soviet-Union and Eastern Europe;
. Advice to regulatory bodies and provision of a basis for the development of common European Standards.

Application of Neutrons for CMC and Coatings Characterization

The project aims at exploring the potential of neutron scattering techniques for investigating localized residual stress fields within relatively thin surface and interface layers in advanced materials, such as joints between dissimilar materials, coated materials and ceramic matrix composites. A brief, preliminary experimental campaign was conducted in 1992 in close collaboration with Dr. A. Ezeillo and Prof. G. Webster of Imperial College (London), in order to verify the feasibility of the HB4 neutron scattering set-up at the HFR for the purpose of residual stress measurements in components. Three different components were investigated to determine stresses induced by autofrettage in a ferritic steel ring by shot peening in a nickel base alloy plate and by fabrication of a composite component. Several encouraging conclusions are drawn from these preliminary measurements. The hoop strain measurements on the autofrettaged ring (7RDMA) are in very good agreement with similar results obtained using instrument D1A at the ILL Grenoble and at Polaris at the Rutherford Appleton Laboratory as illustrated by Figure above. The measurements on the shot peened plate show that the facility provides the necessary instrumentation characteristics to allow for accurate and reliable near surface work. Due to the local near surface nature of the residual stress field associated with shot peening, it is necessary for the sampling volume to be only partially immersed in the sample in order to obtain near surface data.



Above: *Measurements of Hoop Strains on 7RDMA using different Diffractometers*

A close agreement of the measured strains was established with similar data obtained with instrument D1A at ILL Grenoble. Measurements performed on the composite component revealed that although stresses could not be resolved across a single fibre or in between fibres, it is possible to measure bulk strains in both matrix and fibres using long count times.

Physical Properties of Coatings

Much effort was spent during 1992 on the further development of the capabilities of the glancing-angle X-ray spectroscopy laboratory, as well as on a continuation of the work on the improvement of the corrosion resistance of industrial alloys through the detailed study of the initial stages of high temperature oxidation. Many thin film samples from other research projects were also examined for phase, crystallite size and microstrain determination.

The X-ray reflectometer was uprated by the addition of both input and analysing monochromators, allowing a comparison of the geometry with those applying more stringent experimental conditions. It was found that in many cases one or both monochromators could be left out without causing any great deterioration in the quality of the results. This allows the simultaneous monitoring of both K-alpha and K-beta radiation (thereby eliminating zero point errors), as well as a simpler alignment procedure. The final version of the X-ray reflectivity analysis program (ReX) was also completed, and has been accepted for publication. The technique of monitoring X-ray fluorescence in order to deduce the thickness of thin films was also investigated during the past year.

A versatile computer code (FluX) has been written to simulate the intensity of fluorescence as a function of incident angle for any film/substrate combination, taking into account secondary enhancement effects in both film and substrate. This will be further developed in 1993, and will provide a method of non-destructively mapping the film thickness over extended areas. Depending on the composition, the technique will be capable of assessing films from a few to several hundred microns in thickness.
The details of the surface phase changes and oxide growth rates of Yttrium ion implanted Ni-20Cr, in comparison to unimplanted or Krypton ion implanted material, and also to the oxide dispersoid strengthened material MA 754, were elaborated. The surfaces were examined in detail by both in-plane and out-of-plane glancing angle X- ray diffraction, and by ReflEXAFS at Daresbury SRS at the Yttrium and Chromium K-edges. The results, to be published soon, shed new light on the oxidation mechanisms of these materials.
Work was also performed on the possibility of measurement of residual stresses, using X-ray methods, in computer memory chips ("Gazzelle" type) for IBM, in collaboration with the University of Brescia, in order to identify the best resin for encapsulation.

Characterization of Ceramic Surfaces by Positron Annihilation

Methods of Positron Annihilation Doppler Spectroscopy have been developed further and applied to studies of thin coatings and surface modification, in particular, defects in TiN coatings and treated steel substrates. Non-destructive methods based on PADS, for the thickness measurements of thin films on a base material have been undertaken.
Measurements and evaluations have provided a preliminary data base of the Doppler spectroscopy parameters and of the positron transmission in different materials. A first version of this method, tested in typical cases such as Al on Aluminia and Al on Cu, proved to be reliable for thickness measurements in the range of some to several tens of micrometer, while the extension to thinner films is being studied.
Favourable cases, according to the data base include combinations of various metals with ceramics (Alumina, Zirconia, Silicon Nitride, Titanium Nitride, etc.)

Pre-standard Activities

The project aims at developing and improving methods for the testing and the measurement of mechanical properties, in the broadest sense, of metallic, ceramic and composite materials, including the validation of these methods. The deliverables serve as the pre-normative input to the subsequent development, improvement or optimization of codes and standards by European Standards Institutions. The experimental activities focus on the design, the implementation and the optimization of advanced mechanical testing equipment and of the associated testing techniques, directed towards non-standardized tests on advanced materials, as well as on the determination of allowable tolerances on testing parameters. An example of the former aspect is the characterization of a newly developed radiant lamp furnace for the heating and/or rapid thermal cycling of test specimens during mechanical testing to temperatures of up to 1800-1900°C in air. The results are used to optimize the furnace in close collaboration with the manufacturer. The continuous effort to optimize the procedures of the thermo-mechanical fatigue test, which is frequently applied in our laboratory, towards smaller tolerances on some specific test parameters illustrates the latter aspect of this activity.
Other testing activities with a pre-normative impact rank under the umbrella of Round Robin testing. In the reporting period there was involvement in two Round Robin exercises. In the VAMAS round robin on the measurement of the fracture toughness of monolithic ceramics at high temperature the testing parameters have been defined, the specimen material has been received and the test samples are currently being machined. The other Round Robin is on thermo-mechanical fatigue testing of a nickel based alloy, involving several U.S. institutes and companies (NASA, G.E., MarTest, Univ.Illinois, Rockwell Science Centre, Textron Lycoming) and IAM under the coordination of ASTM.

Research Area:
Surface Modification Technology

Assessment of Research Area Progress and Achievement: Surface Modification Technology

The production, modification and characterization of surfaces and surface layers with controlled properties and functional behaviour are both scientifically fascinating and economically important.
Surface technologies are among the strong growth areas in overall industrial turnover, and the fields of application are expanding rapidly. These techniques are accessible to small and medium enterprises and provide significant environmental and resource saving potentials.

The innovative research of the IAM focuses on:
- advanced process analysis and product and performance characterization methods ultimately enabling approaches to intelligent processing technology;
- optimization of surface modification and coating processes and the comparison and combination of different techniques to optimize the product performance and quality;
- engineering the surface conditions at the interfaces of dissimilar material combinations in fibre-reinforced composites and joints and in sintering and grain growth processes;
- engineering the surfaces of materials for electrocatalytic and sensing functions.

Complementary activities, facilities and know-how have been developed at the two sites of the IAM in Petten and Ispra, and each of the two sites has installed and taken into operation a processing centre for its surface engineering R&D which has diverse links and crosslinks with the characterization and performance testing facilities of either site.

- The Advanced Coating Centre at Petten is a joint facility of the IAM and the neighbouring Dutch Energy Research Foundation ECN.
  Coating processes available are:
  * atmospheric plasma spraying,
  * high velocity flame (detonation) spraying,
  * low pressure chemical vapour deposition,
  * plasma assisted chemical vapour deposition,
  * plasma ion nitriding,
  * electron beam/laser processing equipment.

- The Surface Modification Centre at Ispra operates facilities for the following processes:
  * physical vapour deposition (sputtering),
  * ion implantation,
  * laser treatment,
  * electron beam alloying, hardening and melting,
  * low pressure (vacuum) plasma spraying.

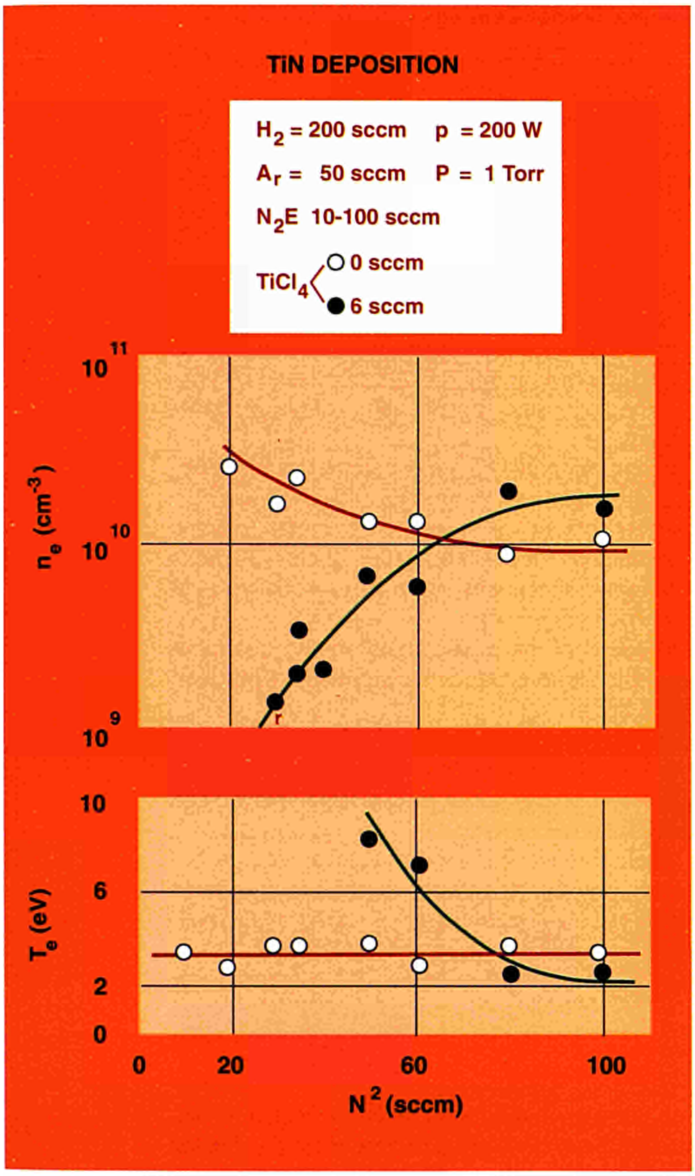
In the further development of this research area, the refinement of the existing characterization methods and the introduction of new techniques will play an important role.
The development of such techniques is in some cases the subject of separate research projects e.g. the determination of the adhesion strength of coatings by a laser pulse induced spallation test method and the application of neutron scattering to residual stress determination in coated surfaces, composites and joints.
An information base to correlate process and product characterization information from these and other techniques is being developed in the project "Coatings Databank", its goal is to provide a step towards an intelligent plasma spray processing.

The themes of the single studies are strongly influenced by technological problems.
The surface layers which are being investigated range from protection layers with a thickness of 1 mm, to layers of some nanometres on small particles and fibres.

Thermal barrier coatings have been investigated intensively. The production of zirconium-oxide coatings has found a large interest and its properties as a function of the production parameters have been studied.
This type of coating is of interest for advanced industrial applications, in particular the aerospace and gas turbine industry.
In a collaboration with an external customer such thermal barriers have undergone testing including their resistance to shock by high power laser pulses.

In the near future increasing attention will be addressed to enhancing the cross relations between the different techniques and to elaborate the improvements which can be obtained by the combination of the existing techniques.

Wear and Corrosion Resistant Coatings



Above: *Variation of electron density and energy with N_2 flow rate in PACVD - TiN gas mixtures with and without $TiCl_4$*

The installation of the major coating machinery and the associated infrastructural support of the Advanced Coating Centre was largely completed during the first half of 1992, and a series of commissioning and setting-up trials set in action for all of the facilities. The year has been devoted to the modification and tuning of machines to their specific coating tasks, to development of equipment for new applications and to trouble-shooting problems on prototype instrumentation.

For the project to develop Thermal Spraying Technology: Air Plasma Spraying (APS) and High Velocity Oxygen Flame Spraying (HVOF), proving trials on the robot and the turning table were finished in the first quarter, and the two spraying techniques were compared for the spraying of Cr_2O_3 coatings. The energy yield of HVOF was found to be marginally higher than APS (11% against 8%) but the deposition rate of HVOF was 50% lower. The Cr_2O_3 sprayed coatings have an emissivity of 0.8 to 0.95. Hardness measurements show that APS coatings are harder (HV0.2 = 1750 to 2100 (APS); 1650 to 1850 (HVOF)). The selection of optimal spraying distances has been made on the basis of coating hardness and porosity measurements. Surface temperatures of sprayed layers were measured using in-situ optical pyrometry. A new high speed pyrometer is installed, allowing 1 µs resolution times on 0.5 x 0.5 mm surface areas, which will form the basis for the investigations into process diagnostics in the next years.

The first free standing bodies in Al_2O_3 and in ZrO_2/Y_2O_3 have been made, highlighting the difficulties of fabricating angular shapes with uniform stress and thickness.

Chemical Vapour Deposition (CVD) and Plasma Assisted Chemical Vapour Deposition (PACVD) studies are focused on the development of TiN hard surfacing on steel. Commissioning of prototype mass flow control systems by the manufacturers has led to many delays, despite which, near-stoichiometric TiN coatings have been deposited both by hot-wall and cold-wall (Rydberg type) reactors.

From the hot-wall equipment, analysis of coatings showed that a low level of Cl contamination could be obtained at 600°C, but that upon reducing temperatures to 500°C, the contamination increased rapidly.

Improvements to coating microstructure by control of gas phase kinetics are in train. Cold-wall deposition showed better quality coatings but with high Cl contamination.

The influence of Cl on the formation of PACVD deposited films is the subject of further study by examination of plasma characteristics in typical precursor gas mixtures.

A radio frequency compensated plasma probe of the Langmuir type was used to monitor the density (m_e) and the energy (T_e) of the electrons from the plasma used in the PACVD of TiN coatings in $TiCl_4/N_2/H_2/Ar$ mixtures. (Figure on page 39 above).

The dependence of m_e and T_e on the N_2 gas flow was measured in 2 cases:

(i) in the absence of $TiCl_4$ in the reactive gas mixture,
(ii) when this precursor is introduced in the reaction chamber ($TiCl_4$ flow = 6 sccm) in the typical deposition conditions used in the production of TiN coatings.

The strong decrease in m_e and the corresponding increase in T_e observed at a flow rate of $TiCl_4$ flow = 6 sccm results from the capture of the low energy electrons of the plasma by highly electronegative chlorinated species.
The negative ions produced by electron attachment play an important role in the dissociative process leading to the deposition of TiN coatings.
The official status of the ACC was re-established in 1992, as a co-operative venture between the Institutes, rather than an independent foundation. The Centre is business-oriented, and considerable efforts in the last year have resulted in several proposals for contracts, with EC and Dutch national actions, including 3 COST actions, 2 BRITE actions, 1 EUREKA action and 1 NOVEM (NL) project, not including bilateral external contacts.
The Centre was formally opened in November by the Director General of the JRC and the Deputy Minister of Economic Affairs of the Netherlands.

Thermal Barrier Coatings

The objective of this study is to investigate how plasma spray conditions and, in particular, subsequent HT-ageing treatments affect the development and retention of the transformable tetragonal (t) phase in plasma-sprayed YPS ZrO_2 thermal barrier coatings and how this influences the fracture toughness and the fatigue life of coated parts.

The following actions have been successfully completed during the reporting period:
- preparation of test pieces in Alloy C-263 including deposition of a NiCrAlY-bond coat by VPS-technology and post-coating diffusion treatment,
- optimization of APS-spray parameters for deposition of relatively dense YPS ZrO_2-coatings,
- establishment of appropriate metallographic techniques for the preparation of cross sections of coated specimens, which are essentially free of any pull-out,
- selection of the post-coating ageing treatment conditions to produce transformable t-phase in YPS ZrO_2,
- implementation of a special XRD software programme for the quantitative determination of the different phases (t, t', c, m) in plasma-sprayed YPS ZrO_2,
- establishment of an experimental procedure, based upon the indentation method to determine the fracture toughness values of sprayed ZrO_2-coatings,
- design and commissioning of a test facility by which the interfacial crack growth in TBCs can be measured under dynamic loading in the 4-point bending mode.

Furthermore, in the frame of this project, a technique has been developed to determine the flexural and longitudinal elastic moduli, as well as the shear modulus of coatings. The technique is based upon the resonance vibration of freely suspended specimens using a FÖRSTER-Elastomat. It has been successfully applied to cylindrical bars and is presently being expanded to beam-type specimens.

Wear and Corrosion Resistant Coatings based on Nanodispersive Systems

The development of new ultra-high strength coatings is presently the subject of intensive R&D activities. The most promising appears to be their exploitation in systems which are far from thermodynamic equilibrium. This is the case for the so-called nanodispersive or nanophased materials characterised by an ultra-fine microstructure down to the nanometer range.

Results

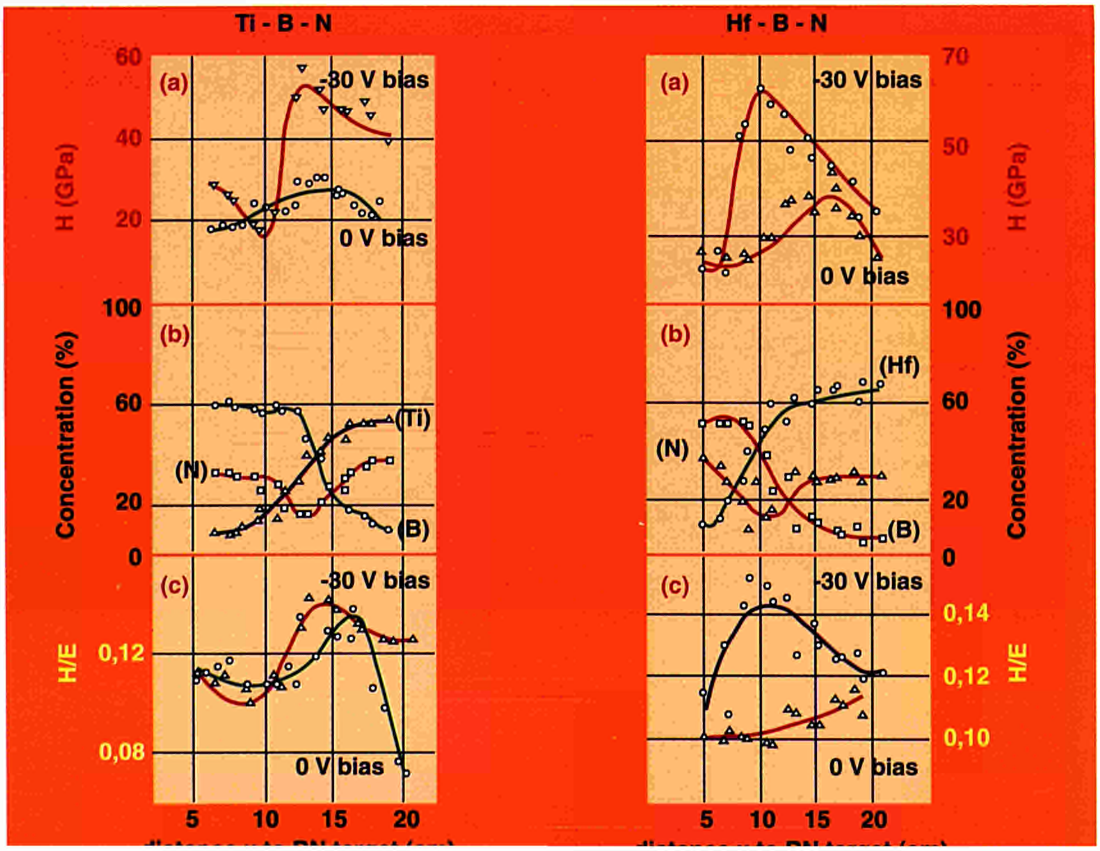
1. TiB_xN_y and HfB_xN_y films were deposited by reactive co-sputtering with and without bias voltage from a Ti and Hf target, respectively, and a BN target. As substrate material 316 steel was used. Crystallite sizes in the 10 nm range were obtained. The chemical composition of the films was varied by positioning the substrates at different locations between the Ti (Hf) and the BN target.

   The films were characterised by ESCA - Auger spectroscopy, nanoindentation and glancing angle X-ray diffractometry.

   Results are shown in Figure below as a function of the relative position x of the substrate to the metal target. As can be seen extremely high hardness values, up to 60 GPa, were reached.

2. Cubic boron nitride (c-BN) displays similar outstanding material properties to diamond but has a better high temperature resistance. It can be deposited by dual ion beam techniques, however, the reported conditions under which c-BN is formed are contradictory. We investigated the deposition conditions by a new technique: films were prepared by assisting the growing BN film with a highly focused Ar^+ and N_2^+ ion beam of constant beam voltage. In this way zones of varying phase composition were formed (large c- BN amounts in the centre of the beam decreasing continuously towards peripheral zones). Analysis was performed by FTIR spectroscopy with about 2 mm spatial resolution. The results show that the critical variable is the momentum transfer with a threshold at about 250 $(eV\ amu)^{1/2}$, below which, however, a small amount of c- BN (10-20%) is still formed. These findings can explain some discrepancies encountered in the past.

Below: *Hardness H (a), Concentration (b), and Ratio Hardness/Young's Modulus, H/E (c) of Ti-B-N and Hf-B-N films of various composition*



Surface/Bulk Structural Properties Modification

The main tools which have been used for obtaining modifications to the properties of the materials are a 5 kW CO_2 power laser and an ion beam implanter.

The ion beam implanter, produced by Dan Physik, allows the use of a gaseous ion source and a sputter source used with a thick sputter target. Originally, the existing system didn't allow the use of low melting points materials such as aluminium and magnesium due to insufficient cooling.
An improved model has been set up using the difference in the thermal expansion coefficient of copper and aluminium to ameliorate the thermal contact resistance.
Moreover, in order to reduce the implantation cost of precious metals, such as platinum, a sputter target obtained by brazing thin foil on an aluminium support has been developed.
In Table below the main applications of the ion implanter in 1992 are shown. The installation of an IBAD (Ion Beam Assisted Deposition) system is under way. An electron gun heats a metal pool which evaporates metal vapour directly on to the ion bombarded sample, vapour deposited layers enter into the target surfaces by ion beam mixing.

Surface hardening of aluminium with TiN

The surface hardness of aluminium has been improved by a factor of about 20 by covering the surface with a titanium layer of about 100 nm and by implanting nitrogen ions (N^+) of 50 KeV, at different fluences.
Surface analyses by means of the ESCA method showed that up to 30 nm from the surface of the titanium layer, carbon and oxygen were present and that their concentrations decreased with increasing distance from the surface. On the other hand those of titanium and nitrogen increased.
By means of RBS spectra the surface concentrations of titanium and nitrogen could be determined and, in some cases, the ratio N/Ti could be obtained, as well as the thickness of the remaining Ti-layers. Comparing the analysis results with the prediction of the nitrogen distribution obtained by means of the PROFILE code it appears that there were no sputtering losses in forming the titanium layer.

The hardness of the implanted layer was measured by means of a nanoindenter. It appears that when the mean projected range of N^+ in titanium is of the

General use	Ions	Energy (Kev)	Surface layer	Substrate
Ion beam mix.	Ar^+	190	Ti/BN/Ti	Ti
"	Ar^+	100	Al	Al_2O_3
"	Ar^+	195	Cr	Al_2O_3
Ion implant.	N^+	190,150,100		diamond
"	N^+	50,150	Ti	Al
"	Cr^+	180		Al_2O_3
"	Nb^+	200		$TiAl_3$
"	Pt^+	170		SnO_2
"	N^+	190		Ti6Al4V

same order as the thickness of the Ti layer a nanohardness up to 39 MPa can be obtained.

CO_2 laser

The CO_2 laser, manufactured by Rofin Sinar Laser GmbH, has been used for performing a surface treatment on the austenitic steel AISI 316, for coating stainless steel with a Hastelloy layer, and for testing the thermal shock resistance of a ceramic layer on MCrAlY.

Hastelloy coating of a stainless steel plate

Hastelloy alloys combine an excellent resistance to corrosion with elevated temperature mechanical properties. Their application, however, is limited by their high cost. It appeared interesting, therefore, to study the possibility of covering a base material with a coating of Hastelloy of appropriate thickness. An economic evaluation indicated that a cost reduction factor of 0.5 to 0.6 would be expected by the use of clad tubes rather than bulk tubes of Hastelloy. The protective layer was deposited by melting a Hastelloy C276 wire continuously fed to the surface of the stainless steel by means of a CO_2 laser. Different power levels were used in order to change the level of melting of the substrate and its degree of mixing with the deposited layer. The beam spot diameter was kept at 1.6 mm, slightly higher than the wire diameter in order to insure a good lateral mixing. The molten zone under the beam was protected from oxidation using argon as a cover gas. A coating three millimeters thick is obtained using four successive passes. The first test was conducted using the laser beam at a specific power of 3.2 kW. The micrograph of the section showed that the deposited layer appeared to be well anchored to the stainless steel substrate. No porosity was apparent. However it appeared, particularly at the interface between the base material and the coating, that some small fingers of the base material had penetrated into the deposited layer. In order to obtain a good adherence it is necessary to have a good molten pool where the two metals intermix and form a strong metallurgical bond.
However it appears that inside the molten pool convective (Marangoni) flows are formed due to gradients of surface tension. As a consequence a small stream of the molten stainless steel enters into the mass of the molten Hastelloy. The subsequent rapid cooling freezes the molten pool. Other samples were prepared using the laser beam at a lower power. It can be seen that in this case the anchoring of the deposited layer is rather poor. A certain amount of porosity is present in the layer; it is higher for the layer treated at a lower power.

Thermal shock tests

On request of the firm Aermacchi a series of thermal shock tests have been performed on samples of MCrAlY covered with a layer of stabilized ZrO_2 0.5 mm thick to act, as a thermal barrier. The samples were formed by 4 plates plus three complex pieces and in particular a winglet, a flap and a little window. The thermal shock test consisted of subjecting each of the three plates to laser pulses with a duration of 10 ms at incident power densities of 2, 4 and 6 kW/cm^2 respectively. Moreover on each sample a series of 100 shots at a power density of 2 kW/cm^2 have been performed in order to evaluate the resistance of the thermal barrier to thermal fatigue. The fourth plate has been subjected to pulses of the same duration and power density but obtained with a high voltage electron beam. The electron beam presents the advantage that the incident power is equal to the adsorbed power. The sample was treated with the electron beam at power densities of 2, 4 and 6 kW/cm^2 but no thermal fatigue tests were attempted. The three complex pieces have been subjected to a series of 100 shots at a power density of 2 kW/cm^2.
The work has been completed by a metallographic examination of the effects of the pulses on the different plates. The results obtained show that both the plain sample and the more complex one have resisted successfully the tests even at a power level (6 kW/cm^2) which exceeds the possible working load. Two main mechanisms are apparent. On one side at low power there is a coalescence of the small porosity giving rise to a more coarse pore structure and, particularly in the thermal cycling tests, to formation of large fissures. At the higher power level a recrystallization of the zirconia is added to this phenomena which increases the dimensions of the grains. No difference is apparent between the samples treated with the laser beam and those treated with the electron beam. The repeated thermal shocks on the complex pieces did not produce any spalling of the ceramic coatings.

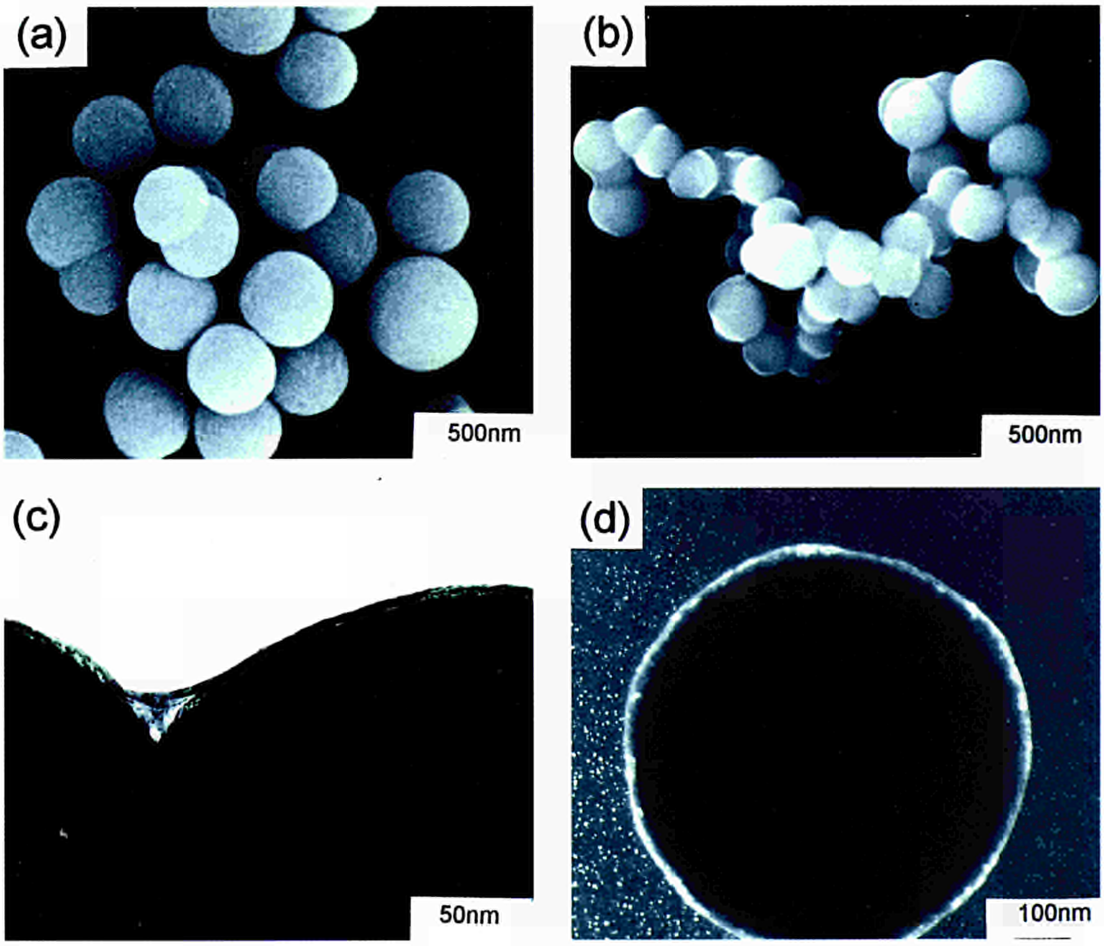
Sub-Microstructural Engineering

Progress in the development of advanced ceramic materials depends increasingly on the control of the chemical composition of ceramic powders at a sub-micron scale. If additives are used, for example, to promote sintering or control grain growth, then the ideal configuration for them is as a uniform coating on the ceramic particles. We report here on the use of a solution chemistry technique, i.e. homogeneous precipitation, to produce such coated ceramic powders.

Homogeneous precipitation is a technique in which an acidic solution of metal salt + urea (Table on page 45) is slowly heated to release ammonium and carbonate ions into the solution thereby gradually and uniformly increasing the pH until colloidal basic carbonate is precipitated.

An ageing process is then used to accrete this nanoscale precipitate. In the absence of a substrate, ageing produces spherical particles. However, if particles are already present in the solution at the start of ageing, the precipitate will accrete on them as a uniform layer.

Below: *SEM micrographs showing particle size and shape:*
(a) Zirconia stabilized with 12 mol% Ceria,
(b) Zirconia stabilized with 12 mol% Ceria coated with Yttria
TEM micrographs showing
(c) thin layer of coated material (bright field),
(d) thin layer of coated material partly crystallized in the electron beam (dark field)



	CONCENTRATION IN SOLUTION, mol/l						AGEING	
PARTICLE	$Zr(SO_4)_2$ $.4H_2O$	$Ce(NO_3)_2$ $.6H_2O$	YCl_3 $.6H_2O$	$Al_2(SO_4)_3$ $.18H_2O$	UREA	PVP** wt%	TEMP ^0C	TIME min
UNCOATED	$8.8x10^{-3}$	$1.2x10^{-3}$	-	-	2	0.3	85	180
YTTRIA COATED	UNCOATED PARTICLES*		$4x10^{-4}$	-	1	-	85	60
ALUMINA COATED	UNCOATED PARTICLES*		-	$1.45x10^{-3}$	1	-	85	120

* concentration of particles corresponded to the concentration of reactants used to make the particles.

** polyvinylpirrolidone

Table above: *Precipitation conditions*

The homogeneous precipitation technique can therefore be used both to produce spherical ceramic particles and to coat them with a layer of a different material. The precipitate must be calcined in air at about 500^{0}C to convert the amorphous basic carbonate to a crystalline oxide ceramic. In this work the core particles were Ceria-stabilized Zirconia, and the coated layers were either Yttria or Alumina.

The SEM pictures in Figure on page 44 below show that the uncoated Ceria-stabilized Zirconia particles were smooth spheres with a rather narrow size distribution in the range 400 - 700 nm. Except for a reduction in diameter, the appearance of the particles was unchanged by the calcination process. The TEM pictures of Yttria coated particles show that the whole surface of the particles was coated with a uniform layer about 10 nm thick. From the known chemistry of the system, the composition of the coating could be deduced to be Yttrium basic carbonate, $Y(OH)CO_3.H_2O$ which is known to transform to cubic Y_2O_3 on calcining at 500^{0}C. Layers of Alumina 25 nm thick were also produced by the same technique.

These results show that homogeneous precipitation can be used both to produce spherical particles of Ceria-stabilized Zirconia in the size range 400 - 700 nm, and to coat those particles uniformly with Yttria or Alumina. However, the chemical processes involved are applicable to a wider range of chemical compositions. Calcining the core particles under different conditions prior to coating creates possibilities for producing coated particles with a variety of internal structures. The homogeneous distribution of a sintering additive such as Yttria should, for example, improve microstructural homogeneity in materials derived from coated particles compared with microstructures derived from powders without such additives. These results may also be regarded as illustrative of other possibilities, e.g. the coating of ceramic fibres and thereby the improvement of the properties of the fibre/matrix interface in ceramic matrix composites.

Development and Coating of Ceramic Fibres for H.T. Composites

Interfaces in Fibre Composites

The objective of this new area of activity is to establish an understanding of the ability of entrained long ceramic fibres to confer toughness by resisting crack propagation through engineering ceramic materials, and to tailor fibre matrix interfaces to optimise the composite properties. The project builds on earlier experience gained with SiC whisker-Si_3N_4 composites, by incorporating carbon fibres in a Si_3N_4 matrix. Fibre-matrix interfaces will be systematically varied by application of surface coating to the fibres prior to composite fabrication.

The first year of the project has focused on the fabrication of a viable composite, necessitating a coordinated approach on several fronts.

Development of fibre architecture: Since early experiments demonstrated the impracticability of infiltration of pre-woven fabrics by micro-fine ceramic powder dispersions, the project has addressed the fabrication of ceramic composites from 1D and 2D fibre lay-ups i.e. laminar arrays of unidirectionally wound fibre tapes (width ca. 50 mm). The absence of commercially wound tapes of adequate uniformity or thinness has necessitated much effort to establish, in-house, the required fibre-winding quality to allow complete infiltration of the fibre lay-ups, while retaining uniform distribution of fibres throughout the ceramic-matrix and avoiding significant monolithic ceramic inclusions.

Selection of a suitable fibre: Carbon fibres fabricated from pitch precursors offer superior properties of high elastic modulus/stiffness, coupled with low chemical reactivity, but are intrinsically brittle. Evaluation of several commercial fibres during fibre-winding experiments led to the selection of one particular grade (Tonen 500 FT) for continued study in the present programme.

Rheology of silicon nitride powder dispersions: An aqueous dispersion of microfine (mean particle diameter = 0.7 µm) Si_3N_4 powder, with 5 at % each of Al_2O_3 and Y_2O_3, has been optimised for fibre wetting behaviour and slip viscosity and stability, following an extensive study of the influence of dispersants, wetting and anti-foaming agents, solids content and chemistry and processing routes.

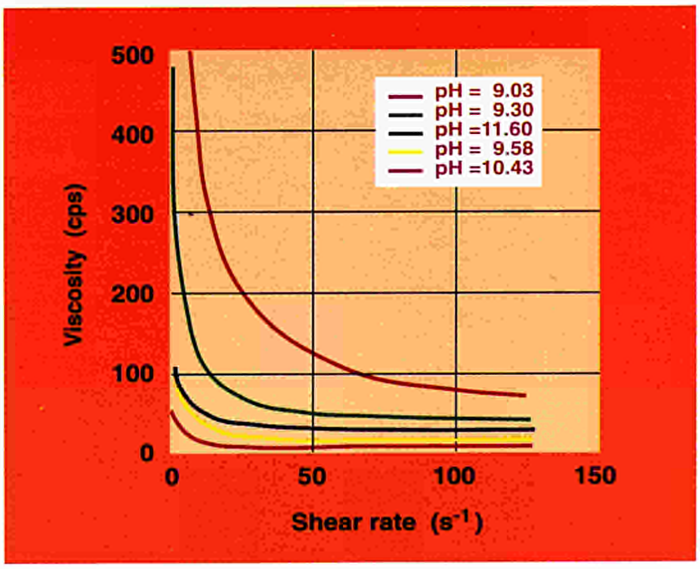
The importance of solid-liquid surface interactions is emphasized by a high sensitivity of wetting and slip stability and viscosity to dispersion pH, requiring the individual tailoring of pH for every material combination (Figure below).

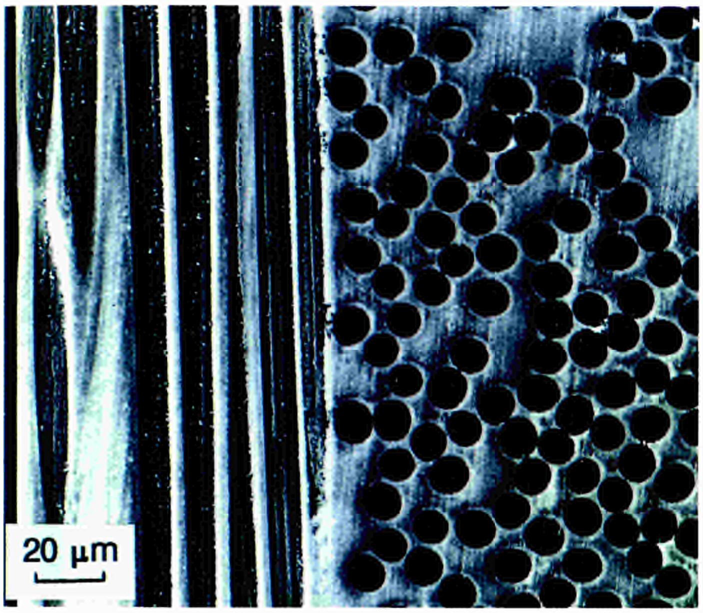
Infiltration - composite forming: Forming of composite green bodies is investigated by two principal techniques:

a) slip-infiltration of pre-wound fibre tapes, by immersion, stacking and press-drying,
b) pre-infiltration of fibre tows followed by spreading and winding to tape dimensions or directly to green-body geometry.

Composites, densified by hot-pressing at 1750°C have shown progressively well-infiltrated microstructure and uniform distribution of fibres (up to 40%) and concomitant reduction in macrodefect population (Figure on page 47).

Below: Plots of viscosity vs shear rate for suspension (65 vol.% solid content) with pH values indicated





With the near completion of optimisation of composite fabrication, the project will proceed to systematic characterisation of composites and interfaces, and to the introduction of fibres, coated with SiC, SiC-$C_{(pyr)}$ and $C_{(pyr)}$-SiC-$C_{(pyr)}$ coatings.

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

Above: *Cross-section of $0^0/90^0$-ply lay up C-fibres within a Si_3N_4 matrix*

Advances in Ceramic Joining

The activities of the ceramic joining project have progressed further towards its goal of understanding the principles that govern ceramic bonding so that ceramic joints could be reliably designed.

Joining of Silicon Nitride Ceramics

(i). Diffusion Bonding

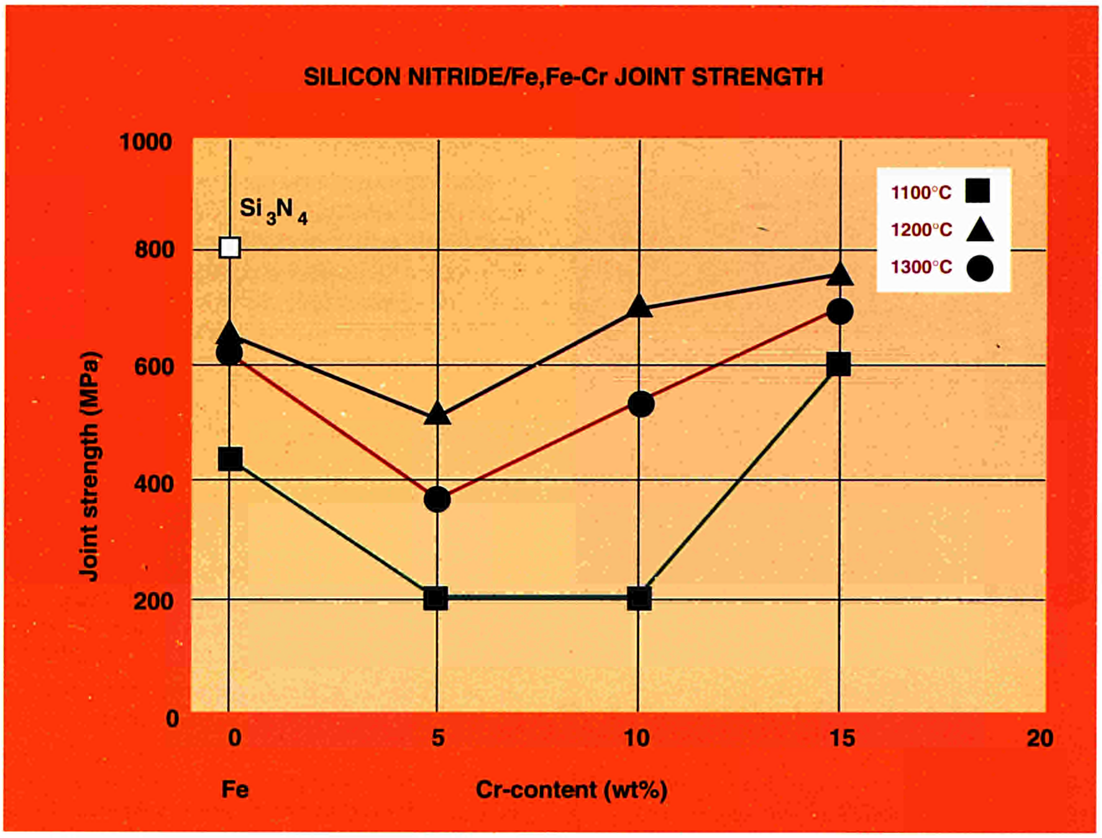
Si_3N_4 ceramics have been bonded utilizing Fe or Fe-Cr alloy interlayers as a joining agent. The joint microstructure and mechanical properties have been determined for a wide range of bonding temperatures, 1100^0-1300^0C, and interlayer compositions, Fe-(0-15wt%)Cr. Depending on the procedures and interlayer chemistry the ceramic/metal(C/M) bond can be direct or via reaction products such as Cr-nitrides, Fe-silicides in M-Si and/or -N solid solutions. The interfacial reactivity is enhanced by higher joining temperatures and Cr-contents. Joint strengths approaching 100% of the ceramic fracture strength have been achieved as shown in Figure on page 48.

Furthermore, the strength for the Si_3N_4/Fe15Cr joints at 800-900^{0}C in air is more than 50% of its room temperature value.

Given the high strength and relatively simple microstructure of the joints realized in the Si_3N_4/Fe-Cr system, work is now in progress to evaluate the interface fracture characteristics, such as interfacial fracture resistance, in order to further optimize joint performance and provide information for predicting interface failure.

(ii). Brazing

The brazing of Si_3N_4 to itself or to metallic alloys (e.g. AISI 316, MA754) has been investigated using Ni-Cr-Si, Ni-Cr-P and Ti-based active commercial brazes. The Ni-base brazes were selected for their high temperature capabilities. Although they may wet the ceramic, because of their as-cooled microstructural complexity and, in general, their brittle character they were not shown to be efficient bonding agents.



Above: *The effects of joining temperature and Cr-content in the FeCr interlayer on the strength of SI_3N_4/FeCr/Si_3N_4 joints bonded in Ar (joining pressure 50 MPa)*

New compositions are being sought that would relieve some of the problems encountered. This necessitated the experimental determination of the high temperature (900-1200⁰C) Ni-Cr-Si-(N) phase diagram (in cooperation with CTK/TU Eindhoven under partial funding from IOP ceramics).
The Ti-base active brazes, although of limited high temperature capability, are known to be able to join Si_3N_4, provided that an "appropriate" interlayer is also used in the case of Si_3N_4/metal joining. The goal of this ongoing study is to assess the effects of the mechanical and physical properties of the interlayer on the performance of Si_3N_4/steel joints. These results are related with those from computational tools as discussed below.

(iii). Modeling of Joint Stress Distribution

Testing experience has indicated that, fracture in ceramic/metal joints often originates in the ceramic near the interface rather than at the interface, and that the joint strength can vary considerably within a bonded block. These observations point out the effects on the joint integrity of thermal and elastic mismatch between the bonded materials and the metal interlayer plasticity, as manifested by post-joining residual stresses. Stress field characteristics in the joint, taking into account the metal interlayer plasticity, are calculated by finite elements using the ABAQUS code (IAM internal cooperation with the Laser Spallation project).

Chemical Sensors and Electrocatalytic Materials

The study of sensing systems for chlorine and nitrogen oxide has continued. For chlorine gas electrometric sensors the departure from the theoretical (Nernstian) values and in particular the influence of an auxiliary layer interposed between the gas electrode and the solid ß''-Alumina electrolyte has been measured and the results have been critically analyzed. Also the factors influencing the rate of response have been investigated.

For nitrogen dioxide the interpretation of the first results of the electrometric sensors appears to be much more complicated. For the environmental NO_2 pollution the use of sensing systems based on the modulation of the electrical conductivity of semiconductors looks convenient.

Microcrystalline tin oxide layers of thickness ranging from 500 to 4000 Å were deposited by sputtering on to polished alumina sheets. This was done at CSIC in Madrid. The layers were then placed in a stainless steel cell and the system brought to a temperature in the range 120 to 300°C.

The layers were characterized in our laboratories for thickness, crystallographic structure, morphology and composition by ellipsometry, Auger sputtering, ESCA and glancing angle X-ray spectroscopy. Ultrathin (60 Å) aluminium films were deposited on to the tin surface of some samples in order to catalyze the ionosorption process. The layers have grains of extremely small size, generally lower than 100 Å.

The results look excellent as some samples show a remarkable variation of conductivity at concentrations lower than 100 ppb, i.e. below the first alarm threshold (which is legally imposed in NO_2 control of towns). Such sensitivities, as far as we know, have not yet been reached elsewhere.

Sensors of an other type have been designed, developed and tested. They consist in a multilayer assembly of SnO_2/SiO_2/Si(n)/Si(p). Such heterojunctions are directly polarized by applying a linearly increasing current from 0 to 3 mA at a rate of 2.5 μA/sec. The Si(n) layer undergoes depletion, then inversion and finally the "punchthrough" and/or "avalanche multiplication" negative resistance conditions. The voltage breakdown forward (V_{BF}) depends on the bending of the conduction bands in SnO_2. The results will be presented at the Eurosensors Congress in Budapest.

Surface Scaling

Cracking and spallation of protective oxide scales and coatings due to thermal cycling is a subject of considerable concern in a wide range of technological applications. Since the understanding of the phenomena of spallation is poor and insufficient to predict the material behaviour in a reliable way, a programme to study the possibility of using Thin Layer Activation (TLA) to measure material loss during thermal cyclic oxidation was started. Due to its specific properties, TLA is expected to contribute to a better understanding of the phenomena of spallation. Feasibility activation studies were carried out on various high temperature materials, including coated alloys. One of the most important conclusions was that material loss with a sensitivity of less than 1μg is easily detectable. Multi specimen test facilities for scaling and spallation under cyclic oxidation, including the possibility for forced gas cooling were designed and partially tested.

To obtain some preliminary experience with the use of TLA for spallation studies, a simple experimental set- up was also constructed and tested. This facility became operational at the end of september 1992. It was successfully used for the testing of thermal barrier coated superalloys, supplied by ONERA, France and Cr sheet material produced by a powder metallurgical route. The collaborative action with BCR-Brussels and the Complutense University of Madrid on the production and characterisation of reference oxides in the frame of the VAMAS Surface Chemical Analysis Activity, was continued. Various surface analytical techniques were investigated to accurately characterise these layers, especially in terms of thickness and composition. Activation analysis using proton and alfa particles, nuclear reaction analysis and grazing incidence X-ray reflection were selected to be the most suitable techniques.

Research Area: Materials Information and Data Management

Assessment of Research Area Progress and Achievement: Materials Information and Data Management

During recent years much awareness has developed of the high commercial and strategic value of materials information. Industry has begun to consider it as an important resource that needs management provisions. In parallel to this development, the research sector as the principal producer of materials information has become more sensitive to the requirements of information management and allows for it in most of the new research programmes (in the past such provisions were usually neglected).

The Institute for Advanced Materials has been addressing this subject for many years and has included a specific activity on Materials Information and Data Management in its materials research programmes.
This activity has always proceeded on the basis of minimum resources and developed limited effect as long as the perception of the value of material information was low.

Within the research activities of the Institute, this area has its own importance, however it is too small to organize management functions for the information output of the large European R&D programmes which are funded, organized and supported by the Commission. Some contributions to the COST 501 project and the Fusion Technology Programme could nevertheless be effected by the HTM Databank.

The scope of activities has had an impact in a small number of relevant fields: pilot concepts such as an advanced user interface, standard data input, the association of data and models and advanced data evaluation, standardization efforts on advanced ceramics and high temperature mechanical testing. Some results of the activities are marketed for third party use. Nearly all activities have a strong standards relationship, and a major effort is addressed to the planning for a new multinational scheme for institutionalizing European pre-normative research.

Coatings Processes Data Banks

A Computerized Relational Plasma Spray Coatings Properties and Processes Data Management and Manufacturing system (CPP-DB) is under development. Its objective is to assist coatings users and manufacturers to optimally manage their data and to give support to the coatings producers in order to achieve optimum product quality and to tailor coating properties.
The system includes the following general content: coatings industries, processes/properties, publications, experimental data, manufacturing recipes, mechanical tests, post processing and powder thermo-physical-mechanical data. The properties involved include: adhesion, residual stress, porosity, electrical conductivity and other layer properties. The CPP-DB provides other facilities such as: on-line process data acquisition and storage in the data bank, process mathematical models (stress/strain, particles speed/temperature, etc.), data analysis/evaluation and graphics, powerful multi(combined)-search with assist mode as well as data export (process set-points) to coating process control devices.
The system is a multi-user network system for data management which also can run as a stand-alone process local system in order to assist the manufacturing or production system (a first step towards an Intelligent Processing System).

HTM Data Banks

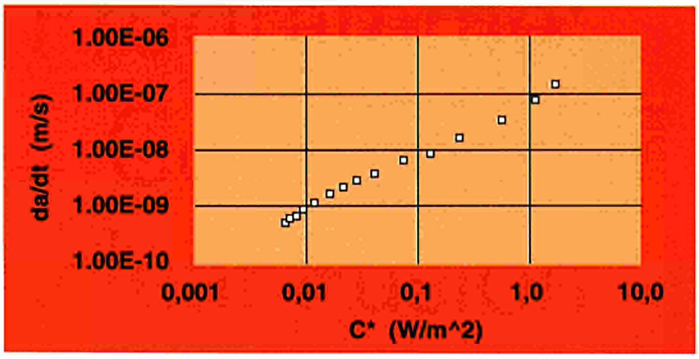
High Temperature Materials Databank

The High Temperature Materials Databank (HTM-DB) supports the Data and Information Management for Advanced Materials Projects by providing computerised information on materials properties through the storage of mechanical and physical test data in combination with a sophisticated modelling and evaluation system. It aims to cope with requirements for data management, evaluation and input for computer aided processing and information services. It further serves the dissemination of data between collaborating parties in joint projects. The databank is designed as a relational data system and is implemented with the commercially available ORACLE Database Management System (DBMS) on a workstation. The PC-based user interface is installed on the PC-side under Microsoft Windows and is therefore compatible with other Microsoft Windows software such as spreadsheets or help compilers. The user interface interacts with the workstation via a network connection. ORACLE provides facilities for Ethernet and/or asynchronous local and/or wide area network connections so that the online service to project partners (COST, BRITE, FUSION, etc.) can still be maintained.

In 1992 work has continued on:
- the transfer of the evaluation programme library from the Amdahl mainframe to the PC/MS-DOS environment and,
- the user friendly data entry and data retrieval interface,
- data entry for COST and FUSION programmes.

Help files for online help and data output options such as data listings, graphics (Figure below) and reports are installed into the user interface. A brochure which describes the new HTM-DB is designed and printed.
Emphasis is given to implement intelligent user guidance. Therefore a cooperation with the University Regensburg is initiated to improve the HTM-DB under ergonomical aspects to create a professional software which can be commercialized.

Below: *Chart of a creep crack growth data as a predefined Microsoft Excel for Windows macro*



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 1992, efforts have been directed to the following activities:

Within the frame of the Institute initiative for Standardization and Pre-normative R&D of Advanced Ceramics the following meetings were organized:

- CEN Technical Committee on Standards for Advanced Technical Ceramics (184),
- Six CEN Working Group meetings addressing Standards for ceramic classification, powders, monolithics and composites,
- VAMAS, Technical Working Area 3; Pre-normative R&D of Advanced Ceramics.

An International Symposium on Ultra High Temperature Mechanical Testing was held in September 1992, at Petten.

The Symposium was conceived in response to the increasing demands of industry for stronger, lighter structural materials to endure repeated exposure to extreme environmental conditions. The development of ceramic matrix composites and the quality control of components has created a need for new testing facilities and procedures.

The scope of the meeting was specifically focused on test methods and procedures for ultra high temperature mechanical testing above 1500^{0}C. The principal subject materials were ceramic matrix composites including C-C, intermetallics, refractory metals, and monolithic ceramics.

14 Invited papers and 10 posters were presented.

100 Experts attended the Symposium, which also included an exhibition of High Temperature Testing Equipment and Materials.

2. Contribution to the Specific Programme: Controlled Thermonuclear Fusion

Research Area: Fusion Materials

Assessment of Research Area Progress and Achievement: Fusion Materials

The study of materials for fusion reactors and their behaviour in fusion typical environments, is an established research area in the IAM. Emphasis of the work has evolved with changes in fusion rector designs and is now concentrated on two main lines:

- the development and study of low activation materials, and
- the materials behaviour in components under thermal fatigue conditions.

In addition, at the request of the European Fusion Programm, the IAM is devoting a limited effort to the characterisation of stainless steels during and after neutron irradiation in the HFR. The IAM activities are fully integrated in the EC programme and are monitored by the corresponding groups. Most of the planned activities have made good progress during 1992. Two activities, the AMCR data collection, and the disruption studies, will be concluded in the near future. The irradiation experiments in the HFR, which are requested by the NET team, require longterm planning. These studies are producing important information for the characterisation and the engineering data base of the European reference steel of the type AISI 316. Connected to the work on stainless steel is the installation of a data bank on this material.

The informatics part of the data bank is now developed and in future the efforts will be devoted to the introduction of the available data.

The studies on the fiber reinforced SiC are delivering encouraging results. The producers of the material are showing increasing interest and are collaborating by delivering the material in the desired form. The IAM is the driving laboratory in this field, and the contacts with other laboratories are strengthening.

The experimental and theoretical activities on thermal fatigue have been a central point in a world wide programme. They are part of a network under the guidance of the IAEA. The results obtained so far are valuable but demonstrate that considerable further effort will be needed.

During 1992 the IAM has maintained its recognised position in the field of fusion materials.

Low Activation Materials

Ceramic matrix composites (CMC) with continuous ceramic fibre reinforcement are under study as potential low activation structural material for fusion reactors. A cooperative research programme on this subject has been established with CEA (Commissariat à l'Energie Atomique), ENEA (Ente Nazionale Energie Alternative) and SEP (Société Européenne de Propulsion). Collaborations have also been established with a number of Universities in France, Italy and Germany for aspects like measurements of thermal conductivity and joining/machining of CMCs.

Commercial SiC/SiC composites, obtained from European manufacturers, were evaluated by doserate calculations (ANITA code and REAC-ECN 5 library). These materials, though not elementally tailored, had already the potential to fulfill all current low-activation limits of interest in Europe. At this point, the analysis of the impurities, however, was based only on normal chemical/spectroscopic methods. The study was then extended to quantitative analysis of scarse but critical trace-elements by charged-particle activation-analysis (CPAA) on different types of materials. The results have shown clearly that a higher grade of purity is obtained in material produced by Chemical Vapour Infiltration (CVI) in comparison to other batches produced by liquid infiltration and subsequent pyrolysis.
Moreover, irradiation of CVI-type material in the HFR Petten gave complementary results from neutron activation analysis (NAA).

After quantitative analysis of the content of 32 different crucial trace elements, it can be concluded that European industrial 2D reinforced SiC/SiC materials will not cause any problem in terms of low activation criteria. This is of particular importance after the work by Cierjacks [1], where the low-activation advantages of Cr- and/or V-based alloys were critically reviewed from the aspect of fusion spectrum induced sequential nuclear reactions.

These complications in calculating the neutron induced radioactivity of future fusion reactor materials are not expected to cause comparable restrictions for SiC based materials. The important sequential reactions [(p,n); (d,n); (α,n)] do not lead to critical or long-lived radioisotopes.

The results of three-point-bending experiments on unirradiated CMC suggest that mechanical testing can be performed outside of the interlaminar failure regime. Parametric studies varying the ratio of lower span length to specimen thickness produced results comparable to those in the literature on CFC (Carbon Fibre Reinforced Carbon Composites). A three-point/four-point bending stage for high temperature application and different span length positions was designed and is under construction.

The formation of He in SiC/SiC by transmutation reactions due to fast neutrons is currently being simulated by irradiations at the Ispra Cyclotron. Under helium jet cooling, 39 MeV α-particles were implanted in CMC specimens to high fluences (2500 appm). First results show no macroscopic swelling of specimens irradiated at 700°C. Analysis to determine whether the inherent residual porosity of this type of material is an advantage is underway. Ion induced radioactivity was identified by decay analysis to originate from the ^{32}P isotope via the reaction $^{30}Si(\alpha,pn)^{32}P$, $E\alpha > 14.08$ MeV. This should allow bending tests and microscopic analysis of the implanted specimens within the next months.
Meanwhile, 2D SiC/SiC specimens were sent to HFR Petten for neutron irradiation to different fluences at 700°C. Mechanical tests after irradiation are foreseen and will be facilitated by radiation protection techniques developed for the ion irradiated samples. Larger batches of SiC/SiC specimens have been ordered in order to perform thermal conductivity and permeation measurements on as-received and coated materials.

A feasibility study was performed concerning the possible application of low-activation chromium-alloys in a fusion reactor. It was found that chromium and chromium-alloys with either vanadium or iron are much more corrosion resistant than stainless steels. In addition the mechanical properties of chromium and chromium alloys are superior to those of stainless steels apart from ductility. The ductile-brittle transition temperature in well annealed pure chromium and chromium alloys which do not contain carbon in amounts larger than 100 ppm is between room temperature and 300°C. It became evident that the brittle-ductile transition temperature depends not only on the carbon content but also on the structure of the materials, namely dislocation density, grain size, surface preparation, etc.

Pure chromium and chromium with various amounts of vanadium, iron, and manganese were ordered.
A cost estimate feasibility study for the development of low ductile-brittle transition temperature chromium alloys has been asked from European industry.

[1] S. Cierjacks,
Fus. Eng. & Design, 13 (1990), 229-238.

Radiation Creep in Stainless Steels

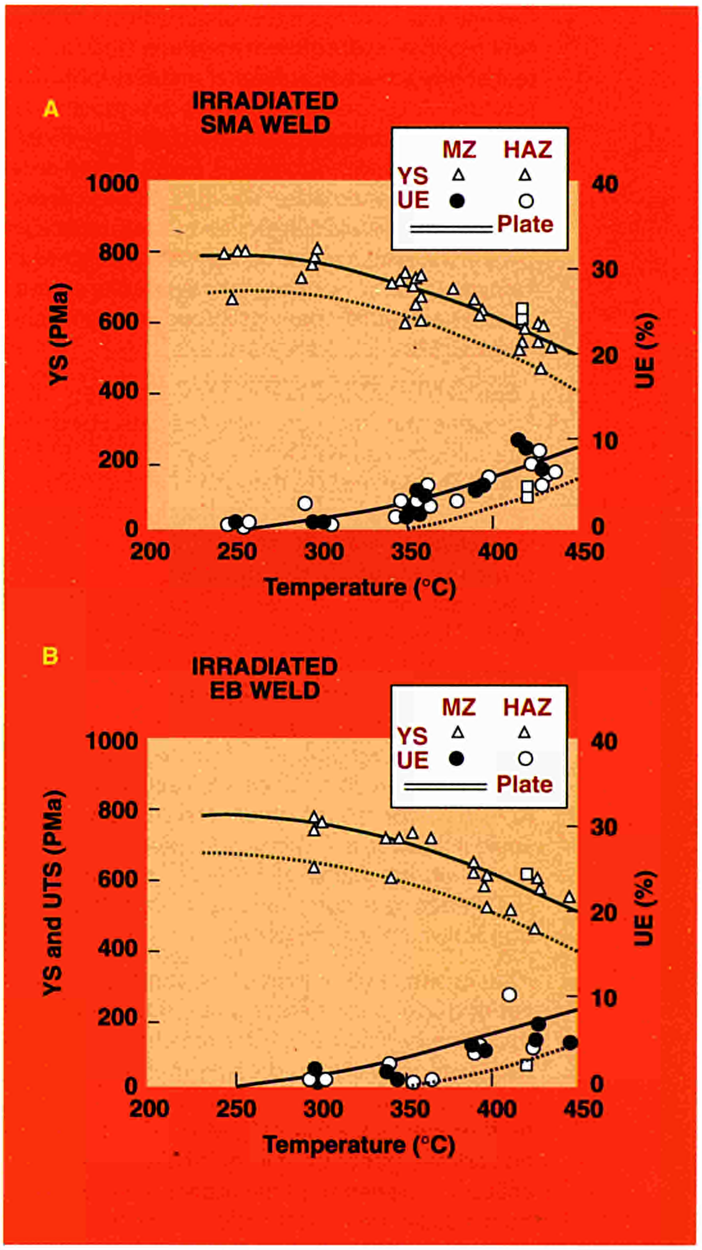
Irradiation creep elongation was measured in the HFR at Petten on AMCR steels, on type 316 EC reference steels, on US-316 and PCS-steels, varying the irradiation temperature between 300 and 500^{0}C and the stress between 25 to 300 MPa.

Depending on the alloying additions to the steel and on the thermomechanical pre-treatment a positive or negative creep elongation is found at the beginning of the irradiation depending on the irradiation temperature and the applied stress.
The phenomena of primary creep elongation is attributed to the formation of radiation-induced precipitates and compounds and not to irradiation creep.

After an irradiation of about 2 to 5 dpa secondary steady state creep is observed. It is assumed that this secondary creep phenomenon is mainly due to irradiation creep rather than volume changes from precipitate and compound formation or to alignment of previously formed precipitates or compounds under the applied stress.

The stress exponent of the secondary irradiation creep rate in annealed and cold-worked steel alloys is n = 1.0. The creep rates of cold-worked materials are almost independent of the irradiation temperature (Q_{irr} = 0.132 eV).

The results obtained in the HFR at Petten for cold-worked AMCR steels, for EC-316, and for the US steels are compared with those obtained in ORR and in EBR II.
The creep rates per unit displacement rate are equal in all three reactors except for cold-worked AMCR and US PCA steels, which are a factor of two smaller in HFR than in ORR.



Above: *Variation of irradiation hardening over heat affected zone and molten zone of the SMA weld (A) and of the EB weld (B). Square points are for Phenix irradiation. Dotted lines are for suggested design margins*

It is found that carbide formation in solution annealed steels during irradiation causes an increase in the volume of the materials; i.e. a large increase of the creep elongation is found in the primary creep stage. α-ferrite is formed on irradiating AMCR and type 316 steels below 400^{0}C causing a decrease of the specimen volume; i.e. a negative creep elongation is observed.

All the steels are unstable below T_{irr} = 400^{0}C. α-ferrite formation in AMCR type materials can cause such large volume changes that they may fragment. If the matrix of AMCR becomes slightly enriched in chromium during irradiation the very brittle α-Mn-phase is formed and the materials are no longer ductile.

Behaviour of Mechanical Properties under Irradiation

Samples of electron beam and submerged melt arc weld joints in solution annealed 316L plate were taken from the heat affected zone and molten zone.

The samples were irradiated in the HFR, at damage production rates and helium production ratio matching NET operating conditions; respectively $3.3x10^{-7}$ dpa/s and 10 appm/dpa. The irradiation dose of 15 dpa corresponds to a 14 MeV neutron fluence of 1,5 MWyr/m^2.

The extensive data on electron beam and submerged melt arc welds in 30 mm SA 316 NET reference plate material provided a valid statistical basis to determine the limits of variation of postirradiation tensile properties in heat affected and molten zones.

Empirical relations were obtained for the temperature dependence of irradiation hardening at 15 dpa in SA 316L, which are used as a reference for reduction in YS and UE to account for presence of welds, YS has a level of 810 MPa up to 300^{0}C, followed by a gradual decline to 590 MPa at 425^{0}C. Engineering UE falls below 1% at 250-300^{0}C, followed by recovery to about 7% at 425^{0}C.

There was no difference between the welds in variation of tensile properties for the heat affected and molten zone (Figure on page 58 A and B).
For both types we suggest applying a reduction of at least 110 MPa in YS and 4% in UE. Below a 380^{0}C irradiation temperature, values of UE below 1% are to be expected.

Radiation hardening is known to saturate at about 6 dpa in the 250-300^{0}C range. Although sufficient experimental data are lacking, indications are found of saturation at 425^{0}C below 15 dpa. It is suggested to extend application of the empirical relations for temperature dependence of radiation hardening and margins for welds, from 15 dpa to 6 dpa.

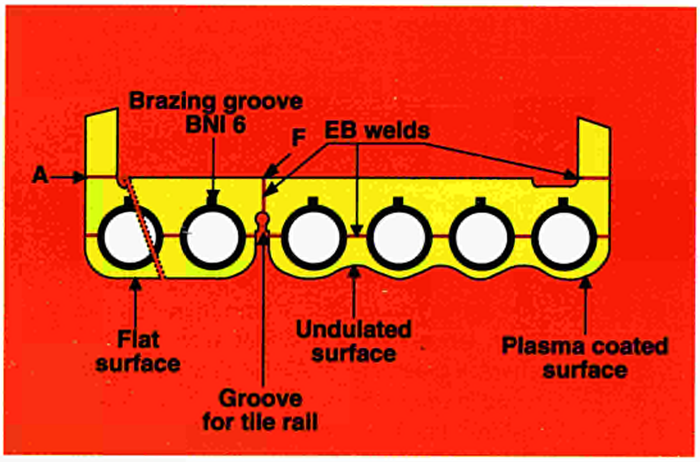
Thermal Fatigue

Thermal Fatigue of First Wall Mock-ups

The testing of two prototype first wall mock-ups was started and completed during the reference period. The mock-ups are the same width as a toroidally sub-divided blanket box but very much shorter in poloidal length. They consist of a sandwich construction of austenitic 316L stainless steel plate and tubing.

The same design, which is conceived to lead to a "double containment" of the coolant water from the plasma chamber to fulfill the stringent limitations for total water leakage into the plasma chamber of 10^{-8} g/s, is fabricated either by electron beam plate-to-plate welding and tube brazing (Framatome component), either by throughout brazing (Ansaldo component). A non-destructive testing exercise involving the mock-ups manufacturers, the NET Team and the NDT unit of our Institute has been started to determine the size of manufacturing flaws and their growth during testing. Braze flaws of greater than 1 mm diameter can be detected and accurately sized. After 5000 thermal cycles at a frequency of 18 cycles per hour, with a peak heat flux of 600 KW/m2, the testing programme was suspended for the first non-destructive evaluation. Visual observation with a video-microscope and an endoscope revealed the presence of cracks on both components. In the fully brazed mock-up the crack was initiated at one of the external corners of the plate-to-plate braze. The crack propagated through the braze towards the cooling tube and continued along the lower temperature braze around the tube itself. Eventually the crack penetrated the tube wall at a depth of about 50 mm, causing a water leak.

In the welded and brazed mock-up a longitudinal crack is clearly visible on the surface opposite the one exposed to the heat flux; it propagates along the EB weld joining the two panels each of which contains four coolant channels. The presence of a crack in the EB weld is surprising, as Low Cycle Fatigue tests carried out at ECN Petten show that the EB joint has equivalent or even better fatigue properties than the base material. The crack is more than 200 mm long and it is not clear whether it was initiated on the back surface or at the root of the groove for the tile rail. NDE is in progress to get a better insight into the component structural integrity. Figure above shows the cross section of the two components indicating the location where failure occurred. From these experimental results and from those previously obtained on specimens without metallurgical modifications the following conclusions can be drawn:



Above: *Location of the thermal fatigue failures on the Ansaldo (A) and Framatome (F) components*

1) the braze does not act as crack-arrester;
2) double containment of the cooling water is not effective;
3) extrapolation of mechanical fatigue data to thermal fatigue leads to conservative lifetime estimate for the base materials;
4) extrapolation of mechanical fatigue data to thermal fatigue leads to unconservative lifetime estimate of welded joints;
5) neither welded and brazed joints will sustain the peak heat flux anticipated for ITER first wall.

Blanket Studies

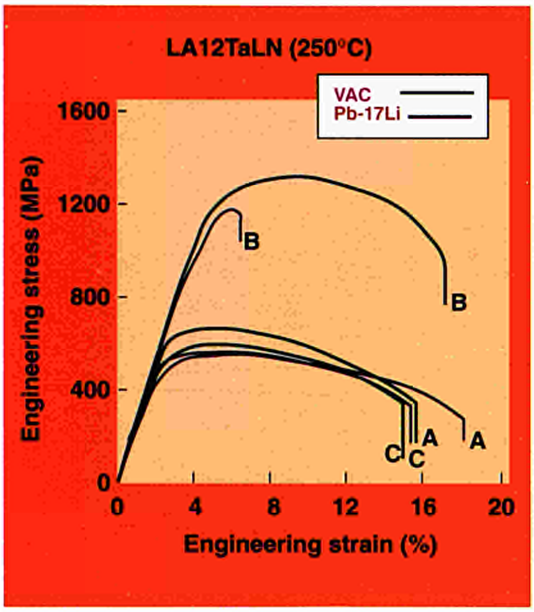
INFLUENCE OF Pb-17Li ON THE PROPERTIES OF BLANKET MATERIALS

Mechanical Properties

Tensile tests on MANET specimens under vacuum, Pb-17Li. H_{22} and H_2/Pb-17Li have been completed, Liquid Metal Embrittlement (LME) was exhibited only by untempered Heat Affected Zone (HAZ) specimens. No severe hydrogen embrittlement was observed.

Initial constant load tests on MANET have indicated a small decrease in the time to failure for tests in H_2 compared with tests in vacuum. Initial tests on welded MANET II specimens have been carried out.

Due to the late delivery of the MANET II material from KfK, an interim Liquid Metal Embrittlement study was carried out on four Low Activation martensitic steels supplied by AEA Culham. The four low activation steels exhibited similar behaviour to



Above: *Liquid metal embrittlement of Low Activation Martensitic Steels. Typical stress strain curve:*
A) Normal Heat Treatment
B) Heat Affected Zone
C) Heat Affected Zone + Post-Weld Heat Treatment

that shown by the MANET steel. In the normal heat treated state no Liquid Metal Embritlement (LME) is observed.
However, severe LME is exhibited by all the steels following heat treatment to simulate a welding operation. A post-weld heat treatment of 1h at 750°C was sufficient to avoid LME in all of the steels except LA13Ta (Figure above). In terms of susceptibility to LME the steels LA7TaLN, LA12TaLN and LA12TaLC were similar to MANET, whereas LA13T exhibited greater susceptibility.

Production of Corrosion Barriers

The production of oxide layers on 316 stainless steel appears to be a promising way to reduce the permeation of hydrogen while increasing the corrosion resistance of the steel exposed to Pb-17Li. However, due to the high temperatures used in the oxidation of the steel this process can not be used for the MANET steel (which needs a specific heat treatment to produce an optimised martensitic structure). Therefore some initial tests were made to produce an aluminide layer on the surface of MANET by a low temperature thermal treatment following the plasma spraying of pure aluminium. This has allowed to the production of aluminide layers on MANET while still retaining the mechanical properties of the steel. Hydrogen permeation tests are currently being carried out on this coating within the ETHEL laboratory.

Fusion Materials Databank

The development of a fusion candidate structural materials data base (FUSION-DB) has been incorporated in the 1992-94 European Fusion Technology Programme and it is now a contractual obligation of all Associated Laboratories participating in the NET/ITER Materials Research Programme to make available their raw experimental data, in digitized form and preferably in ASCII files,to IAM-Petten for direct input to this data base. Several Laboratories have submitted such data to us and these have been inserted into the FUSION-DB. The Associated Laboratories will have ON-LINE access to the data base early next year. This is because the HTM-DB, on which the FUSION-DB is based, has just been transferred from JRCIspra to IAM-Petten. The new data base is now operating in an ORACLE environment installed on a UNIX Workstation.

3. Supplementary Programme:

- Irradiation Experiments in the High Flux Reactor Petten
- Operation of the High Flux Reactor

Irradiation Experiments in the High Flux Reactor Petten

In recent years, as part of the programme of the Institute for Transuranium Elements of the Joint Research Centre in Karlsruhe on the development and improvement of Advanced Fast Breeder Reactor fuels, a series of experiments have been performed in the High Flux Reactor, under the programme "Optimisation of Dense Fuels". Last year, 3 experiments were prepared for irradiation.
The first two experiments NILOC 3 and 4 (consisting of 5 fresh, mixed nitride fuel pins and 1 fresh, mixed oxide fuel containing nitride breeder pellets) are extensions of the irradiation series NILOC (NItride fuel, Low in Oxygen and Carbon) first proposed in 1987. The 6 irradiation capsules were manufactured and the fuel pins encapsulated at Petten. The irradiations (2 HFR cycles for NILOC 3, and 1 HFR cycle for NILOC 4) were performed and completed before the end of the year. Maximum linear fissile powers up to 950 W/cm were achieved with no apparent detrimental effect on fuel pin performance and stability.

The fuel pin in the third experiment, POMPEI (Pellets Oxyde Mixte, PEtten Irradiation), which has the principal aim to study the behaviour of mixed nitride fuel at high burn-up arrived in Petten at the end of the year. As part of a recent programme at Karlsruhe to study transmutation of longlived radioisotopes, the POMPEI fuel pin was altered to contain 3 fuel pellets containing varying amounts of ^{99}Tc.
The irradiation is planned to begin in early 1993.

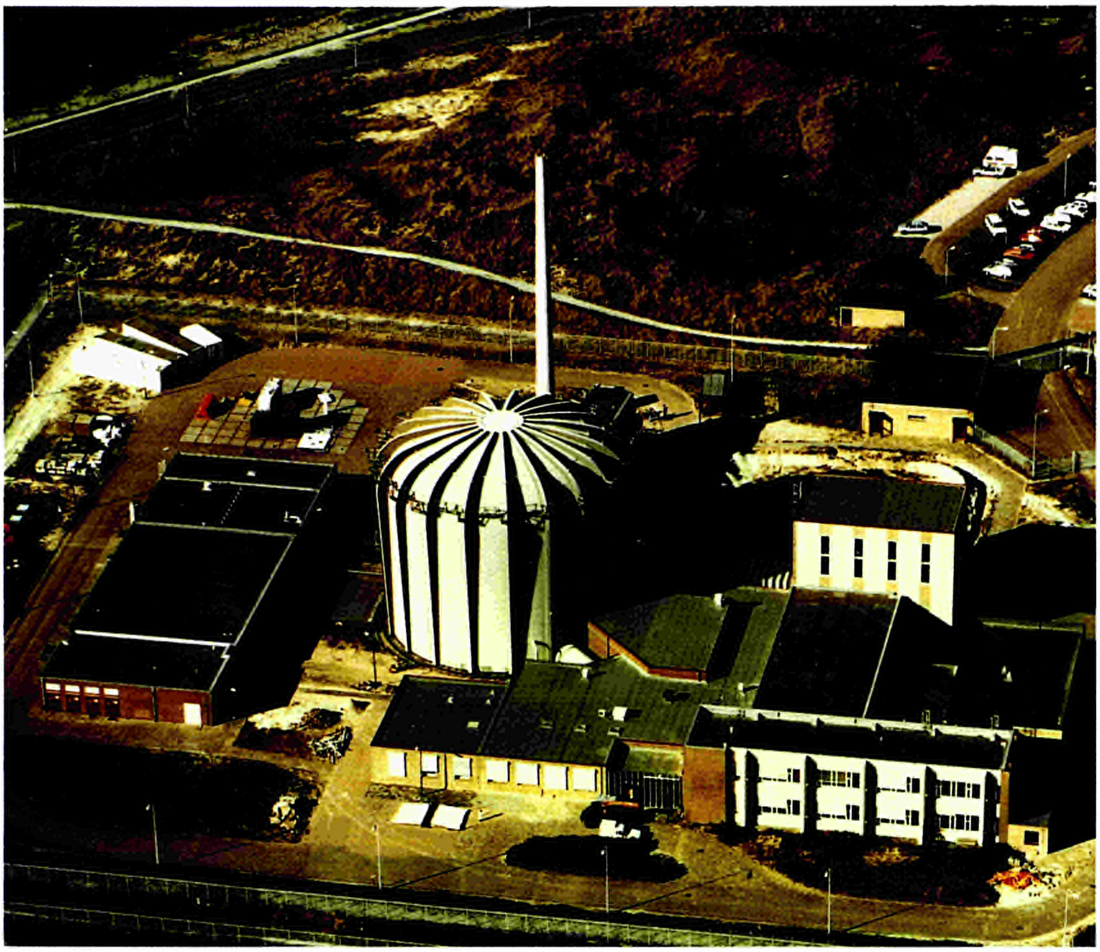
Operation of the High Flux Reactor

The High Flux Reactor (HFR) in Petten is operated and exploited under a supplementary programme which is predominantly funded by the Federal Republic of Germany and The Netherlands; a small complement of the programme budget is provided by JRC specific programmes, and an increasing share is earned by services for third-parties. In this chapter only the projects for German and Dutch Institutions are addressed, whereas the other activities are covered in other chapters of this report.

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

The irradiation programmes on light water reactor fuel were continued with power cycling tests of high burn-up PWR fuel rods which had been pre-irradiated in commercial reactors. These tests are performed in a new irradiation capsule which provides representative LWR operation conditions already at linear heat generation rates of as low as 150 W/cm. For a number of earlier irradiation experiments on PWR as well as BWR fuel rods the post-irradiation examinations were completed and the projects were finished.

A new test programme on the transient behaviour of high burn-up MOX containing PWR fuel rods is under preparation. Assistance was provided for an important upgrading project at the Petten hot cells to refabricate short test fuel rods from full length commercial fuel rods. The project on iodine solubility and degassing from a PWR fuel rod after a LOCA scenario was terminated after the 5th in-pile test. This programme confirmed earlier observations during the TMI-2 accident and hot cell investigations, that the iodine release to the biosphere is more than a factor 1000 smaller than assumed in present safety codes.
The irradiation programmes related to the high temperature gas cooled reactor, comprising graphite and fuel irradiations, are phasing out.
Ongoing projects have, however, been continued to schedule. Presently still five individual irradiations within the "fundamental properties graphite programme" are either under evaluation, being irradiated, or being ready for irradiation.
In the graphite creep programme one irradiation was performed with intermittent measurement of dimensional changes of the samples and with temperature changes during irradiation. Past irradiation experiments have been finally evaluated and reported.



Above: *The HFR Building at Petten*

The HTR fuel irradiation programme was continued with two proof tests of spherical fuel elements. In these tests the operating and safety related conditions of the "HTR-MODUL" are simulated. The objective is to confirm low coated particle failure and low free heavy metal contamination of the fuel element. Eight fuel elements are tested simultaneously. The essential information is obtained from online surveillance of the release of volatile fission products (Kr, Xe and I). The fuel elements exhibited excellent irradiation behaviour with fractional fission gas releases of $< 10^{-6}$, even during temperature transients of up to 1200°C fuel element temperature.

Fast breeder reactor fuel irradiations in support of the European fast reactor project were continued with short transient tests and long term irradiation tests for fuel and cladding axial displacement investigations. Future experiments with the ambitious objective to study the performance of high burn-up, pre-irradiated fuel pins under power cycling and power-to-melt conditions are under preparation and the necessary development work is under way.

The redesign of the alpha-tight EUROS cell for encapsulating longer fuel pins has made further progress. Although towards the end of the year diminishing support of the EFR from the UK has brought this project into jeopardy.

Fusion related investigations comprised irradiation projects for prospective constituent materials for fusion devices, in particular for first wall materials. The various experiments launched during the year have involved damage studies on metals, welded metal joints and brazings, and first wall coating materials. Irradiation, on small scale, of super-conducting materials will continue in the future.
The investigations of irradiation damage and tritium release kinetics from different ceramic lithium compounds under irradiation have been continued too. Under the German contribution to the supplementary programme, the project on "healthy tissue tolerance study" in preparation of patient treatments with Boron Neutron Capture Therapy (BNCT) was continued and extended in co-operation with the University of Bremen at the Petten-BNCT facility at beam tube 11 of the HFR. It encompasses a large animal model study supported by cell culture and phantom experiments*.

The Netherlands' contribution to the HFR supplementary programme addresses several areas, such

* *See also BNCT contribution on page 76)*

as contributions to the European Fast Reactor (EFR), fusion research programmes, use of the beam tubes for solid state and materials research, and extensive utilization of the radioisotope services for radioisotope production and neutron activation analysis. All the Netherlands projects are managed via Energieonderzoek Centrum Nederland (ECN).

For the EFR programme, radiation damage studies on structural materials are performed. The studies comprise crack propagation investigations on compact tension specimens and irradiation of samples for post-irradiation creep fatigue testing. The objective of this work is to provide data on creep-fatigue properties of irradiated stainless steel type 316 L (N) for the EFR design data base, and to verify creep fatigue interaction models.

Within the fusion programme, martensitic steels and vanadium alloys have been irradiated at different temperature and fluence levels. A new project has been launched to study the particular effects of the neutron spectrum on irradiation induced property changes of martensitic and austenitic steels including electron beam welds. Main emphasis is on fracture toughness. The programme on irradiation testing of tritium breeding ceramic blanket materials, where ECN is cooperating with a number of European research centres, has made good progress. Tritium release under irradiation is studied for different zirconates, aluminates and silicates as a function of irradiation temperature and lithium burn-up. In addition, the influence of purge gas chemistry on tritium release is studied. The sixth experiment in the EXOTIC series has been successfully terminated at a maximum lithium burn-up of 3%. The irradiation campaign EXOTIC-7 was started where irradiation damage phenomena will be investigated in a long term experiment with lithium burn-ups of up to 10%.

Five beam tubes, HB 1, 3, 4, 5, and 9 at which dedicated neutron spectrometers are installed, are in permanent use for condensed state physics and materials science applications. Another beam tube, HB 7, is applied for ECN's contribution to the BNCT programme. The equipment installed at this beam tube is successfully applied for prompt gamma determination of boron concentrations in the blood which is an important pharmacological parameter in BNCT.

Recently, ECN has started a programme on the transmutation of the long half life fission products Tc99 and I129, in which the physical and chemical aspects of the transmutation concepts are being studied and, in co-operation with IAM Petten, a facility for irradiation experiments at the HFR is being designed. The tests are aimed at investigating the transmutation efficiencies of technetium and iodine samples and to test inert matrices for future irradiation of americium containing samples in the HFR.

JRC has continued its efforts to prepare the HFR for future neutron applications in science and technology. In neutron radiography, a preparative study on the detection of corrosion in aircraft components and structures of ageing aircraft was undertaken which yielded promising results.

Furthermore, a sensitivity study on the detection capabilities on the ingress of hydrogen in zircaloy tubes was successfully performed. A feasibility and design study for a facility to perform neuron transmutation doping of silicon at large scale has been completed; however, investments into such a facility will only be made if justified by market demand. Efforts towards a device for studying thermal fatigue of fusion materials under irradiation have concentrated on testing various types of potential drop probes for measuring crack propagation. For the BNCT facility at HB 11, an extensive programme of preparatory and qualifying investigations has been performed throughout the year. The facility is exploited under the supplementary programme (see above), with additional support form the JRC exploratory research fund. The reactor itself was operating smoothly with a very high availability of 280 operation days which was in fact more than scheduled, with only very few unscheduled interruptions of regular operation.

More detailed information on the HFR programme, operation as well as utilization, is given in the Annual Report 1992: Operation of the High Flux Reactor. EUR 15219 EN.

4. S/T Support to the Services of the Commission

Standards for Advanced Ceramics

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

Standardization Activities:

The European Standard Organization CEN executes under mandate of the Commission a programme for the development of 44 standards and prestandards. The technical committee (TC) 184 "Standards for Advanced Technical Ceramics" set-up to coordinate the activities operates five working groups (WG) to which Institute Scientists actively contributed in four cases.

The standardization programme for the period 1990/92 was concluded and 44 standards (EN) and pre-standards (ENV) submitted to CEN. For the period 1993/95 continuation of the activities of the CEN TC184 is envisaged. The new standardization programme comprises items, addressing advanced technical ceramic test methodologies for powders, monolitics, fibre reinforced composites and coatings.
A Round Table to review the situation of standardization of advanced ceramics worldwide is under preparation. The requirements of producer and user industries for standards will be addressed.

Pre-Normative R&D:

- Ceramic Corrosion

Following analysis of the results of the worldwide survey of ceramic corrosion activities and attitudes towards standards, attention was focussed on two main themes:

1. Establishing the real level of practical support that could be expected from the European-based ceramics corrosion community and attempting to assemble a working group for prestandards research and development.
2. Initiating a laboratory study on methodology development and assessment, focussing in the first instance on a methodology for hot-salt corrosion.

There is large majority agreement that standards should be approached by formulating guideline methodologies, preferably based on existing, well established laboratory test techniques. Efforts to assemble a small working group have been made and the first workshop is planned to take place in 1993.

In laboratory studies, the potential of a methodology already developed for testing metallic gas turbine materials in a burner rig has been evaluated for testing engineering ceramics. Obviously, if the metals test procedure could be adopted for ceramics, substantial effort could be saved. Unfortunately, modifications to the metals test procedure need to be made, mainly in the areas of specimen preparation and assessment of the degree of corrosive degradation. The need for a mechanical test to be incorporated into the guidelines is being evaluated. When the first stage of the laboratory study is complete, draft guidelines will be issued for discussion within the working group.

- Mechanical Testing of Advanced Ceramics

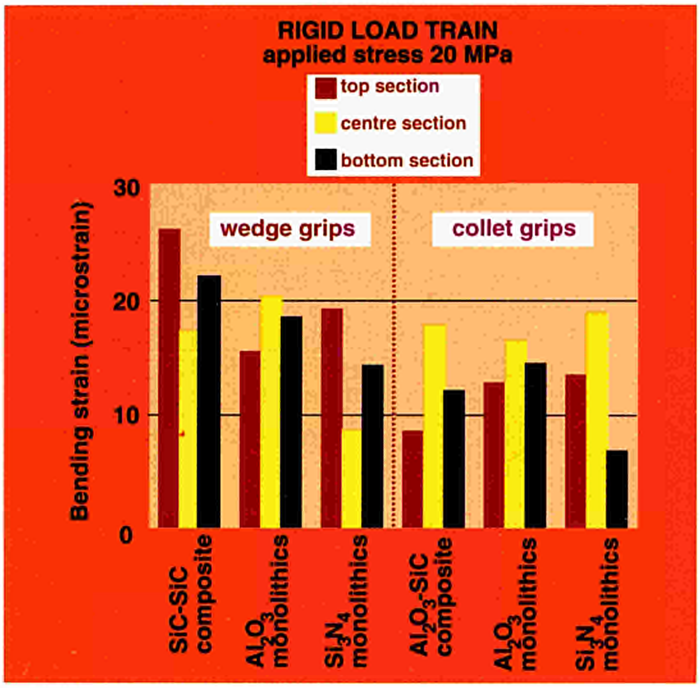
The project aims at stimulating and effectively supporting the development of European (pre)-standards in the area of mechanical testing, both through experimental research and Committee work directed at the drafting of prestandards and codes of practice.
Experimental work in the reporting period focussed on the problem of the gripping of uniaxial test specimens of ceramic materials, in particular on the associated requirement of limiting of the bending component to a sub-critical level during mechanical testing.
In that framework the performance of a novel type of hydraulic wedge clamping grip system for the uniaxial testing of ceramic composite specimens with variable thickness along their length has been explored. Findings up to now are summarized in Figure above on page 69, indicating that upon incorporation of these grips in a rigid load train the bending induced misalignment of the specimen is only slightly larger than when a hydraulic collet type clamping system for constant thickness specimens is used.
The desk activities cover the participation in a number of Committees involved in pre-normative research or in the drafting of pre-standards.

Currently IAM is representing the CEC and its smaller Member States in VAMAS Technical Working Area 3 Ceramics. The Institute also provides the Chairman and the Secretary to a Working Party created in 1992 in the framework of the High Temperature Materials Testing Committee (UK), involving members from Europe and the USA, which is set up to draft a code of practice for the measurement of alignment in mechanical testing.

- *Flaw Sizing in Advanced Ceramics by means of Ultrasonics*

1992 activities concentrated mainly on:
* Review of on-going standardization activities on NDT of ceramics,
* R&D in the field of non-destructive testing: determination of physical characteristics (elastic constants, porosity, ...) of ceramics (monolithic and composites) using ultrasonic methods.

From the review it became clear that there are relatively few standardization activities in the field of NDT of ceramics.
A feasibility study was conducted to assess the possibilities of ultrasonics to determine the physical characteristics of two ceramic composites, an Al_2O_3/SiC and a SiC/SiC material. Only ultrasonic techniques in through-transmission can be used to characterize and inspect these composite materials. The conventional ultrasonic methods used to measure absolute values of attenuation and propagation velocity yield results with an error margin of 50% for the considered specimen thickness and used frequencies. It is nevertheless possible, despite this large error margin, to deduce from these results some guidelines for the development of an ultrasonic system working in through-transmission. Relative measurements may be very useful.



Above: *Comparison of resulting bending strains on three locations along the gauge length when using two types of hydraulic grips*

We have shown the possibilities of an ultrasonic technique to assess in a qualitative way the homogeneity of these ceramic composites and give indications about the presence of material damage. It is clear that further work is needed to establish the link between these results and the intrinsical characteristics (porosity, lay-up of the fibre cloths, ...) of the composites considered.

Standards NDT for Pressure Vessels and Performance Demonstration of NDE Techniques

The achievements and results from 1992 concern both support activities, which are built on the experience gained from the execution of the PISC programme, i.e.:

The activity on "standards NDT for Pressure Vessels" is executed in support of DG III: "Internal market and Industrial Affairs". The establishment of Inspection Standards for pressure vessels is a critical requirement for process economy and industrial safety spanning many major technologies and directly influencing industrial trade.
The objective of the work is to contribute to the harmonization of NDT procedures for pressure vessels.

"Performance Demonstration of NDE Techniques" supports DG XVII: "Energy".
The objective being to harmonize in-service Inspection practices in the nuclear industry, through NDE performance demonstration procedures. Such procedures, to be accepted and established, require demonstration exercises.

During 1992, efforts of the IAM/NDE service concentrated on the development of PISC type networks with European institutions involved in technical and scientific studies related to heavy duty industrial structures subject to non-destructive inspection.

Two major directions are supported at a European level:
- Qualification of inspection (NDE) procedures of structural components;
- Structural Integrity assessment exercises involving the information provided by inspections, material testing and fracture assessment methods.

Each of these directions led to the organization of a network:
- ENIQ: European Network for Inspection Qualification,
- NESC: European Network for Evaluating Steel Components.

These networks are organized along the PISC scheme.

ENIQ: European Network for Inspection Qualification

The general objective is to set up a European Network for the coordination and management of expertise and resources for the assessment and qualification of NDE inspection techniques and procedures primarily for nuclear components.
The primary focus is on the capabilities and limitations of the NDE techniques and procedures used as well as on qualification of In Service Inspection (ISI) trough performance demonstration and other processes.
The ultimate goal will be the supporting of international codes and standards bodies by making available the results (state-of- the-art), technical tools, expertises and performance/capabilities demonstration exercises that can be sponsored and managed at a European level. As a consequence, ENIQ should help in establishing a European attitude towards inspection qualification in general.

Investigated actions are:
- management at a European level of materials to be used for qualification of inspection procedures and for training of inspection personnel,
- organization and management of qualification exercises for inspection techniques and procedures on appropriate mock-ups with the possible creation of a central bank of specimens,
- fabrication and commissioning of test assemblies,
- providing support to codes and standards organizations,
- providing support to regulatory bodies such as technical help for usual and unexpected problems that may arise in power plants, e.g. due to ageing of components,
- studying the various national attitudes in Europe with respect to inspection qualification in view of identifying possible common bases for harmonization.

NESC: European Network for Evaluating Steel Components

The assessment of integrity of a cracked structure is an interdisciplinary activity with inputs from NDE, material testing, stress and fracture analyses. Practices used in different countries to inspect and

assess the fitness for purpose of damaged structures such as pressure vessels and piping vary.
Benefit will be obtained through international exchange of information and experience on common problems.

In the past there have been several collaborative programmes designed to evaluate and exchange knowledge and capability in specific disciplines of integrity assessment; PISC and FALSIRE are recent examples in the NDE and fracture mechanics areas, respectively.

However, it is the interaction between the disciplines and the actual ways by which technical decisions and judgments are made which are crucial in determining the outcome of the total structural assessment process.

The concept of NESC is to explore international practice in the entire process of structural integrity assessment.

The major objectives of NESC will be to organize and manage an experimental programme that will:
- create an international forum in which information about the processes of structural integrity assessment can be exchanged,
- create an organization to undertake specific collaborative studies,
- work towards the use of best practice and the harmonization of international standards.

A proposal was made by HSE (Health and Safety Executive, UK) for a project on the Pressurized Thermal Shock (PTS) transient. This project will serve to initiate NESC by providing a focused problem for study and participation. The project is designed to be of interest to a wide number of organizations involved in the safety of PWR pressure vessels. The PTS transient is considered to be the event which poses the greatest challenge to the integrity of the vessel of pressurized water reactors. It has a significant influence on the safety. The PTS event is of most concern when the vessel has been aged by the effects of irradiation.

Materials Science and Technology for Aeronautics Applications

This work is a support to DG III "Internal Market and Industrial Affairs" whose policy is to improve the competitivity position of European aeronautics industry in respect to the US and Japan and other upcoming industrial nations. The IAM contribution was focussed on the role of Materials Sciences and Technologies.

During 1992 technical assistance was given in the preparation of DG III guidelines.

Studies have been made to evaluate the role of advanced materials in future modern aircraft design and the European position for Materials Research and Technology in air frames and aero-engines actualized.

Assistance was given in defining the European needs for materials technology to compete with other industrial nations.

Standardization of Radiopharmaceutics

Within the frame of this Project supporting DG XI "Environment", the Ispra Cyclotron laboratory organized, in cooperation with the Eurocourse Service and under the sponsorship of DG-XI, a three-days Course (30.6 to 2.7.1992) on: "Cyclotron Production, Quality Control and Utilization of Medical Radioisotopes". Lecturers of the course were experts form E.C. Countries as well as Cyclotron Staff. The Course, addressed Paramedical Staff and Nuclear Technicians, with the aim of providing up-to-date information on cyclotron produced radioisotopes for medical use.

The Course included three visits:

i) to the Laboratories of a Radiopharmaceutical Firm,
ii) to an Hospital using Radioisotopes Positron Emission Tomography (PET),
iii) to the Cyclotron itself (production line of I-123).

The positive resonance from the participants and the demand expressed by medical quarters for the future led us to establish on annual Summer School event, sponsored by DG XI.

Ceramic Catalyst Support

This work is carried out in collaboration with DG XI (Environment); DG III (Internal Market and Industrial Affairs) and DG VII (Transport). The purpose of this study is to evaluate the failure mechanisms of ceramic catalyst support carriers in exhaust systems in automobiles.
A "state-of-the-art" study has been finalized and resulted in pinpointing the needs for future research. The experimental work is jointly carried out with European Industries: materials manufacturers, which provide the test materials, and the automotive industry.

The results can be summarized as follows: the influence of gaseous corrosion at temperatures up to 1200°C has been investigated; no effect of importance has been noticed. First tests show that hot corrosion affects the materials performance and this is aggravated by thermal shocks. The creep and fatigue behaviour are under thorough investigations and it appears that the mechanical stresses determine the failure mechanisms. The boundaries for safe operation are being determined and the laboratory test results compared with real operation test conditions.

The Rôle of Materials in Environmental Problems arising from Power Stations

The work assists DG XI (Environment) in defining its strategy with respect to their industrial environment policy. Support was given in the preparation of guidelines for the development of modern energy efficient and environment friendly fossil fuel conversion and utilization plants.

The IAM contribution was focussed on

i) materials studies for high efficient gas turbines and
ii) materials investigations for use in power station exhaust systems (catalytic conversion or industrial high temperature filters).

Materials Databanks

Materials Data Systems and Standards

The cooperation between DG XIII "Telecommunications, Information Industries and Innovation" and JRC Petten on factual materials data systems and standards which began in 1984 was proposed to continue within the new IMPACT II Programme of DG XIII for 1992-1995 with new objectives. The design of the future activities was adapted to allow for the global changes that have taken place in this area, in particular:

- the replacement of centralized systems by distributed systems as a consequence of progress in information technology and telematics,
- the increasing interaction/integration of materials information with the CIM/CAE environment,
- the progress in the development of regional and international standards both for communications and the information content of a transmission.

An important consequence of this evolution was the need to expand the scope from pure materials database technology to its interaction with CIM/CAE and standardization.

Two 1992 activities made the relevance of these changes visible:

- the joint organization of the "CEC-CODATA Workshop on Materials Data for Computer Aided Engineering" (Feb. 1993 at Frankfurt am Main, Germany) by CODATA and JRC Petten,
- the interaction of JRC Petten with the STEP activities (STEP = Standard for the Exchange of Product Data) in ISO, CEN, BSI and VAMAS and within the Commission (ESPRIT-CIME, DG XIII).

The natural development towards priorities outside the IMPACT II principal objectives made a revision of this project desirable.

Relevant pre-normative activities addressing the interaction of materials data systems with advanced manufacturing technology (CIM/CAE) and the associated standardization continued within a new framework, while the general support to the development of materials information systems closed down at the end of 1992.

Technology Transfer and Utilization of Research Results

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

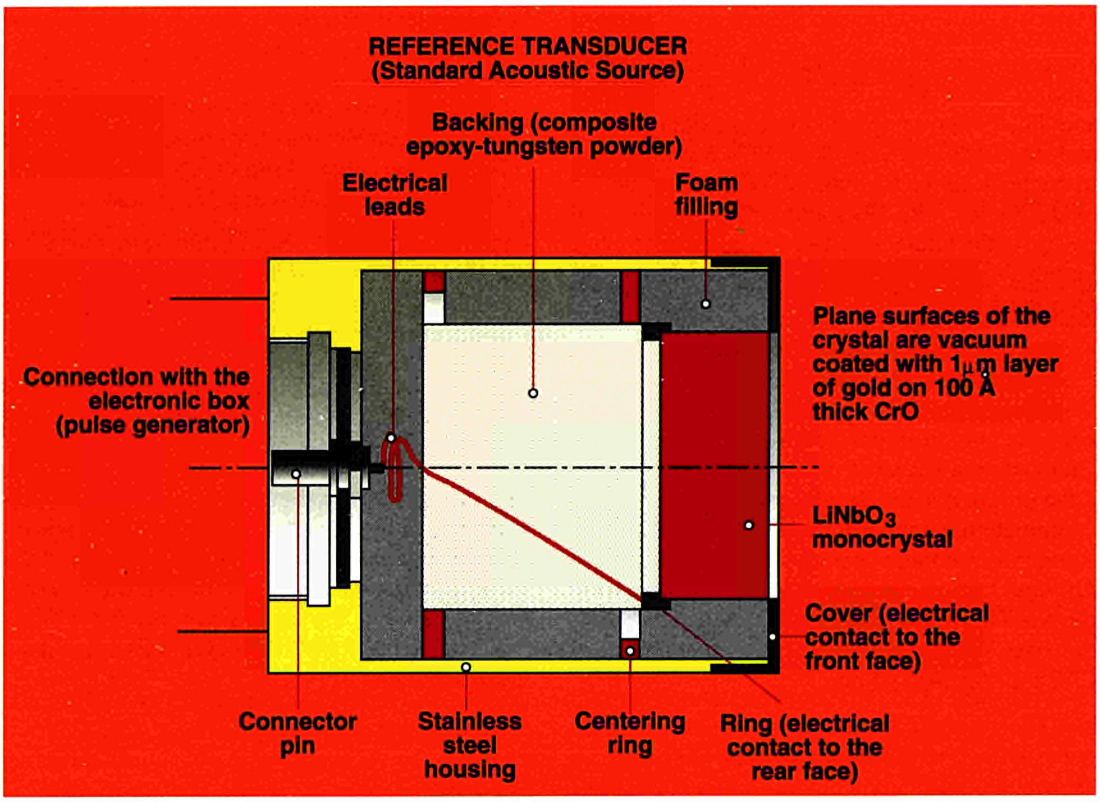
Passive Down Heat Transport

Object of the activity is to demonstrate to industry that this innovative heat transport system is reliable, efficient and safe.

Durability tests on the double-control float-valve have been continued for few months with full satisfaction. A new container for the float-valve was prepared, allowing tests with the new working fluids, including the fluid Meforex 124.

Other fluids, Meforex 123 and R142b will also be tested. One thermosiphon unit of greek construction has been modified by the implementation of our heat transport system, for doing comparison tests with an original thermosiphon unit.

The operation of the sewage treatment plant installed in a mountain refuge and heated by the innovative solar system was continuously monitored. The minimum design temperature of 25^{0}C was always exceeded, allowing a fast decomposition reaction in the anaerobic digestor. The plant, in stand-by conditions, is telemonitored during the out-of-season period. Assistance was given to a licensee for the design of a much larger size plant and for new applications.



Above: *Reference Transducer (Standard Acoustic Source)*

Ultrasonic Reference Transducers

The reference transducer was invented as a result of a collaboration with INSA (Lyon) and DASSAULT AVIATION (St. Cloud). The system generates a pressure step whose characteristics are determined by laser interferometry.

It allows the calibration of ultrasonic transducers in a wide band of frequencies. The co-inventors entrusted the firm IMASONIC from Besançon (F) with the commercialization of the Reference Transducer which changed name to Standard Acoustic Source (S.A.S).

The license agreement N^0 CEINB 234 was given in July by DG XIII. The NDE laboratory provided the licensee with the software "SASCAL" to be used on a PC for the calculation of the sensitivity of hydrophones to be calibrated.

The first two prototypes were built by IMASONIC and calibrated at the NDE laboratory. Some imperfections due to the difficulties in applying the procedure for the fabrication of the transducer head were discovered. Another defect was found in the generator of the electrical current pulse.

The various sources of errors were investigated with a prototype manufactured by the NDE laboratory in order to determine the margines of errors and the measurement accuracy for frequencies up to 20 MHz were performed.

Oxygen Sensors

Activities performed under this heading are reported in the Specific Programme section, Research Area Surface Modification Technology Project: "Chemical Sensors and Electrocatalytic Materials".

5. Exploratory Research

Boron Neutron Capture Therapy (BNCT)

During 1992, the BNCT project made significant progress towards its ultimate goal: the treatment of glioma patients at the High Flux Reactor in Petten. Throughout the year, a very extensive programme of experiments were performed. This included:

Free-beam measurements

to assess and confirm that the flux intensity and neutron energy spectrum remain constant, four measurements were performed using thermoluminescence detectors (TLDs) and sets of 12 stacked, activation foils. The results show that the beam parameters vary by just ±4 %.

Phantom measurements

to determine the thermal neutron flux distribution within the target volume, phantom irradiations were performed using tissue-equivalent or plastic, cylindrical and beagle head like phantoms containing activation foils or wires, and TLDs.
Comparison with similar measurements performed in July 1991 showed that the present thermal neutron flux is some 20% less than before, thus confirming the previously reported uncertainties on the condition and stability of the beam.

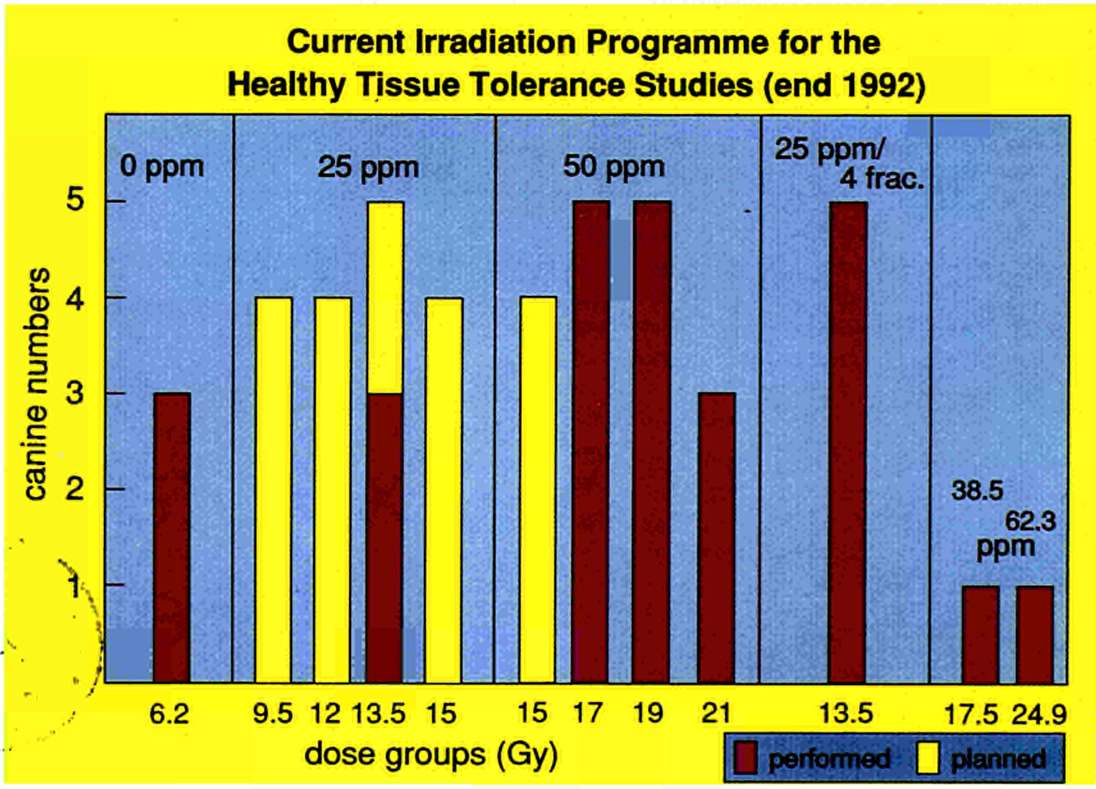
Comparison of measurements performed show variations of no more than ±4 %.

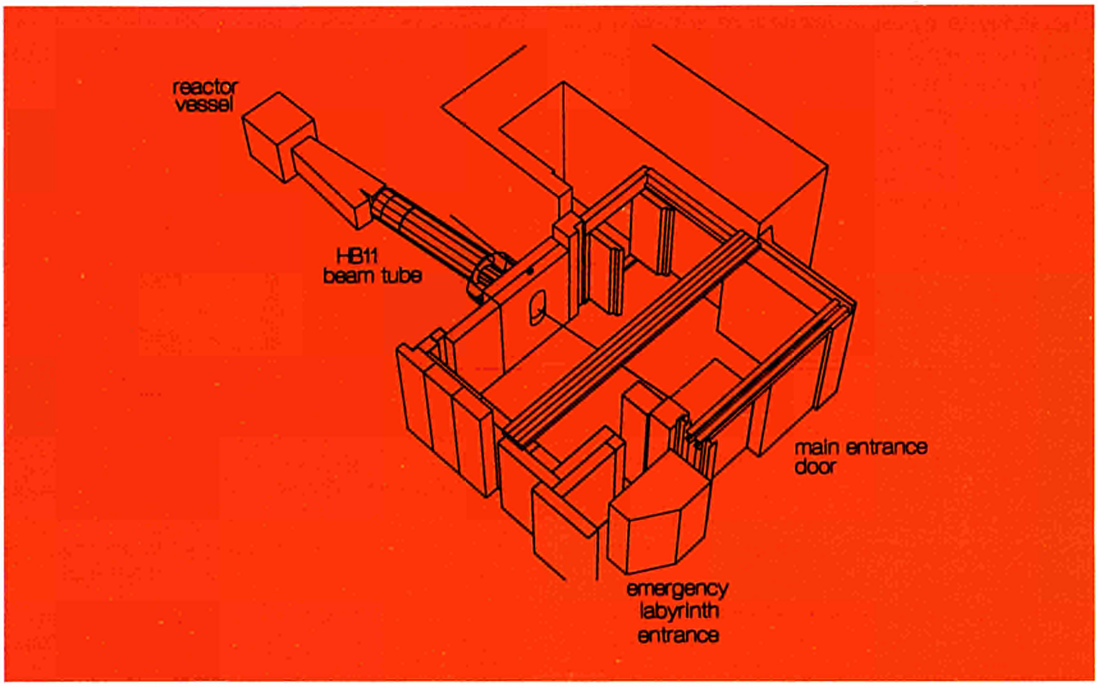
Cell culture experiments

to observe the effect of the boron capture reaction at depth inside a phantom and to determine the RBE values of the various beam components, over 100 irradiations were conducted on various cell lines at different depths in phantoms, containing different concentrations of ^{10}B. Participating groups included JRC and ECN Petten, AEA Harwell, University of Bremen and the Netherlands Cancer Institute, Amsterdam.

Healthy tissue tolerance studies

prior to patient treatment, it is mandatory that for different concentrations of boron and different radiation doses (Figure below), the tolerance limits of the healthy (brain) tissue tolerance study continued with a further 24 in vivo irradiations of the canine brain.
The study will be completed in 1993, whereby it should then be possible to specify the starting dose at which clinical trials on human glioma patients may begin.





Above: *Computer-aided drawing of the enlarged irradiation room at the HB11/BNCT facility: showing part of the structure (4 steel beams) that form part of the complete ceiling*

On-line dosimetry

to assess the beam parameters during treatment, a reliable and dependable on-line monitoring system must be available. As such, a twinned system of GM tubes and fission chambers were installed behind the gamma shutter in the beam port and is currently being tested.

Treatment planning

to determine the radiation dose distribution in the human head during BNCT, a treatment planning scheme is being developed based on the Monte Carlo code MCNP. The code has been used throughout the year to validate and compare with results from the phantom experiments.

Patient treatment room

It will be necessary to substantially enlarge the room in order to be able to treat patients.
As such, a new room has been designed using a 3D computer aided programme, see Figure above. The present set-up will be dismantled in summer 1993, with the new set-up ready towards the end of the year.

Beam optimisation studies

to improve current beam performance, ie. increase the neutron fluence rates and "soften" the neutron energy spectrum, some complementary computational studies were carried out by JRC Ispra. Whilst minor improvements were indicated, they are currently considered not to be cost effective.

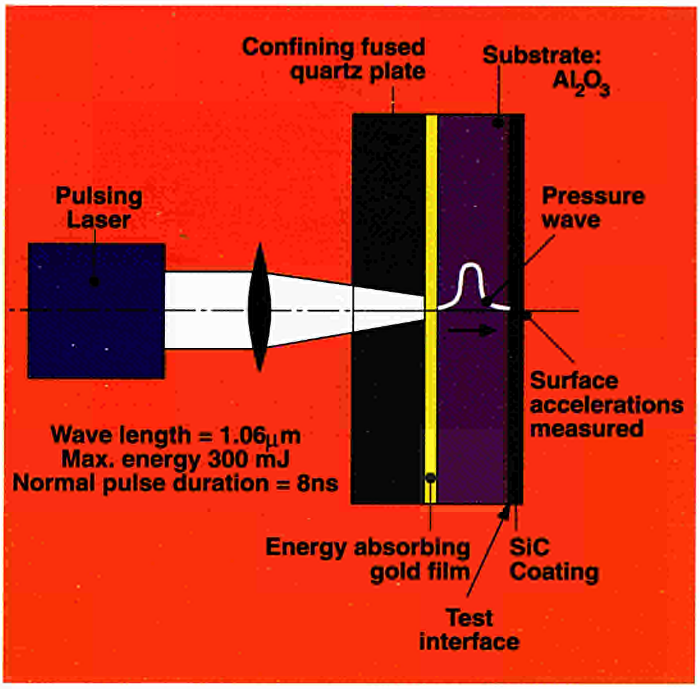
The BNCT project at Petten is entering into a critical stage, with most of the experimental programme nearing completion.
During 1993, the initial experimental work will be over and the patient treatment room ready to receive patients.

Adhesion in Film and Coatings by a Laser Pulse Induced Spallation Technique

The ability to measure strength of adhesion is a central requirement in contemporary thin film technology as well as in protective coatings technology. This project aims at the development of a generic technique for measuring the strength of adhesion of a thin film to a substrate, using a laser pulse induced spallation method and a Laser-Doppler-Interferometry device. The successful completion of this project should yield a reference test measurement technique suitable for calibrating practical techniques.

The experimental facility consists of a Nd-YAG pulsed laser with pulse duration of 8 nsec and 300 mJ energy capacity per pulse at $\lambda = 1.064$ μm and beam diameter of 6 mm. The generated flux can be adjusted by means of a beam expander and beam splitter. The generated pulses are monitored using a digital storage oscilloscope of 1 GHz resolving capacity.

Two such oscilloscopes are used to record the signals from a Laser-Doppler-Interferometer (VISAR), which measures the velocity of the vibrating specimen surface, from which the stress experienced at the interface at the moment of spallation can be calculated. As shown in Figure above the laser beam impinges on the gold film with which the substrate is coated.



Above: *Schematic of Laser Spallation Experiment*

Spallation experiments have been performed on Al_2O_3 specimens 25 mm in diameter and 1 mm thick coated with a 2 μm thick SiC film on top of a 50 nm interlayer of evaporated carbon which has been deposited in order to reduce the interface strength.

Spallation has been achieved several times on each specimen and the spall size (0.5 to 1.0 mm diameter) was found to be energy level dependent.

Upon careful examination by Optical Microscopy it was concluded that all spalls had taken place within the substrate about 30 to 40 μm below the coating outside surface.

Using the present laser pulse characteristics and the specimen physical properties, the reflected tensile stress waves that cause spallation have a width of about 80 μm and the peak stress is achieved at a distance of about 30 μm from the free surface, while the film thickness is only 2 μm.

This means that the tensile wave magnitude at the interface is at least one order of magnitude lower than that where spallation occurs.

The numerical models required for the simulation of the phenomenon associated with the experiment are rather complex due to the material (energy absorbing film) and geometric (gap between specimen and the confining crystal) non-linearities. In addition to the thermal conductivity, specific heat, coefficient of thermal expansion, mass density and modulus of elasticity dependence on temperature and phase (as the gold film undergoes melting), the material non-linearities of gold include wave length and temperature dependence of its absorptivity.

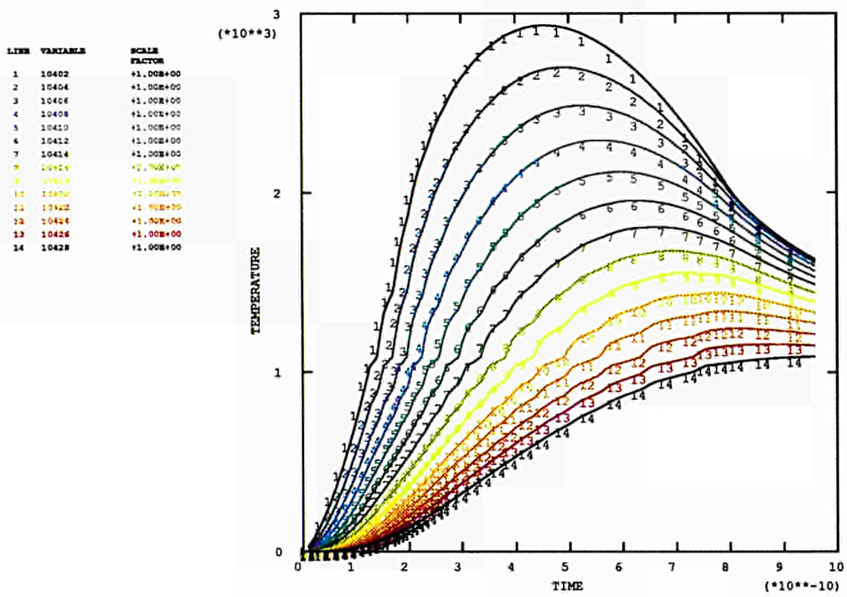
Furthermore, as the gold undergoes rapid volumetric expansion during melting, at the early stages of the experiment, the induced strain rate is extremely high and consequently strain-rate dependent material models are required. Finally the melted gold undergoes radial diffusion and the accurate estimation of the induced shock wave magnitudes depends on the adequate evaluation of its flow.

Linear and non-linear numerical simulations of the above mentioned experimental work have been performed using ABAQUS and DYNA2D. First the thermal transient problem is solved which yields the temperature time-history over the specimen domain used as an input for the stress wave propagation problem.
Figure below shows the induced temperature time histories at the outer layer of the gold film which undergoes melting.

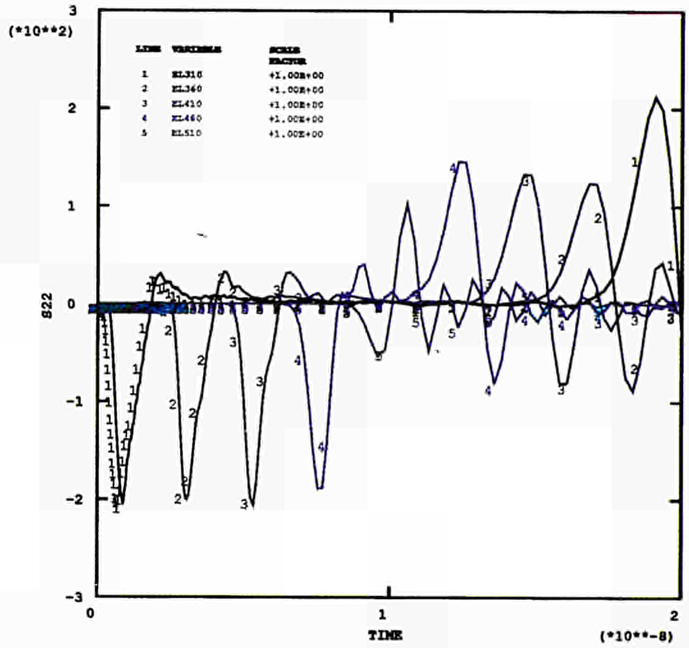
Assuming that the overall gold absorptivity is as low as 2.5% and neglecting all material nonlinearities, it was concluded that the peak value of the tensile wave is about 30 MPa, while the maximum tensile stress experienced at the interface is only 2.5 MPa. Furthermore, the peak value of the stress wave occurs within the substrate and at a distance of at least 30 μm from the interface.
This is expected as the stress wave width is the product of the material sonic velocity and the laser pulse duration (11.5 μm/sec * 8 nsec = 90 μm).

In a first effort to account for the gold non-linearities, the experiment has been further simulated again with a linear model where however the pulse duration is shorter by one order of magnitude (from 8 nsec to 0.8 nsec), thus leading to a more rapid shock. Figure on page 80 above shows the evolution of the stress wave at different locations throughout the gold and the substrate/coating domain.
It is now observed that the maximum stress experienced at the interface is about 105 MPa while the peak value of the reflected stress wave is about 210 MPa and occurs at a distance of only 2 μm from the interface.

Below: *Temperature time-histories across the outer layer of the gold film*



The development of a fully non-linear model of this experiment is one of the primary objectives of this project. On the other hand the accurate measurement of the velocity of the specimen vibrating surface by a VISAR is very important as it will lead to a direct estimate of the interface strength. Finally, based on these experimental results the fine tuning and complete calibration of the mathematical/numerical model is to be achieved.



Above: *Evolution of the stress wave propagation through gold and specimen*

The Use of NDE Techniques for the Characterization of Thin Coatings

Application of NDE to thin coatings

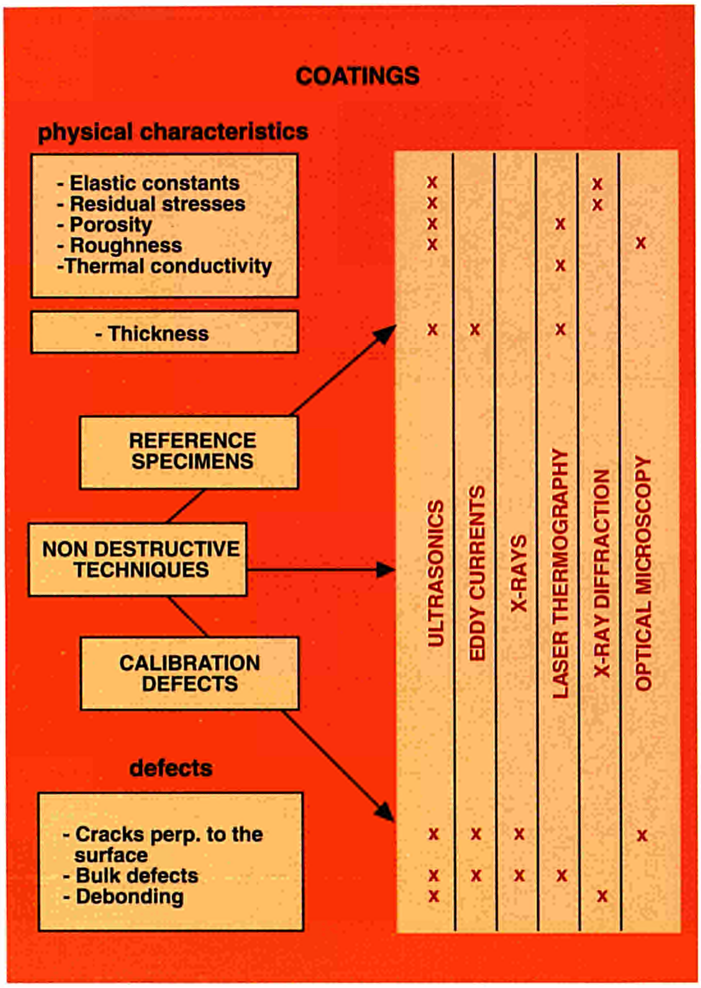
Surface engineering, and coating technology in particular, is playing an increasingly important role in the research activities of the Institute.
Full exploitation of the potential uses of coating for industrial applications requires the development of NDE techniques to characterize coating properties including porosity, homogeneity and adherence.
The work is devoted to the development of a modular acoustic microscope with adequate penetration and resolution.

Two problems have to be addressed: the measurement of the physical characteristics of the coatings and the detection and sizing of typical flaws. Taking into account the peculiar characteristics of such materials, a preliminary study was carried out in order to assess the possibilities of the various inspection techniques (Figure on page 81 above).

The thickness and physical characteristics such as elastic constants, porosity and thermal conductivity can be obtained by using non destructive techniques based upon comparative measurements for which reference samples are needed. Besides, the calibration of the equipment requires the use of artificial defects. The ultrasonic techniques have a good probability of being applied with the complementary contribution of eddy currents and laser thermography. Other techniques (also destructive) will be used to define and to select the reference samples.

For the inspection of coated samples, the specifications of a C-Scan system working in the range of 20 to 300 MHz. were developed. The system will be built by BIOSONIC and be available early 1993.

Proposed activities for 1993 include the inspection of turbine blades coated with an anticorrosion layer of MeCrAlY and a thermal barrier of zirconium oxide deposited by plasma spraying.



Above: *Potential of NDE inspection techniques for coatings*

Modelling the Erosion of Ceramic Crucibles

A well known, costly phenomenon -flux line erosion-, has for years bedevilled the pyrometallurgical and glass making industries. It consists of the scouring or erosion of the refractory crucibles in zones adjacent to the interface where the metal and the slag (or the glass and its layer of flux) meet the refractory.

A mathematical model has been developed on the assumption that the wall erosion profile can be explained by the disruption of the dissolution concentration gradients by buoyancy and Marangoni convective flows.

The physical system studied here consists essentially of a vertical crucible wall in contact with the

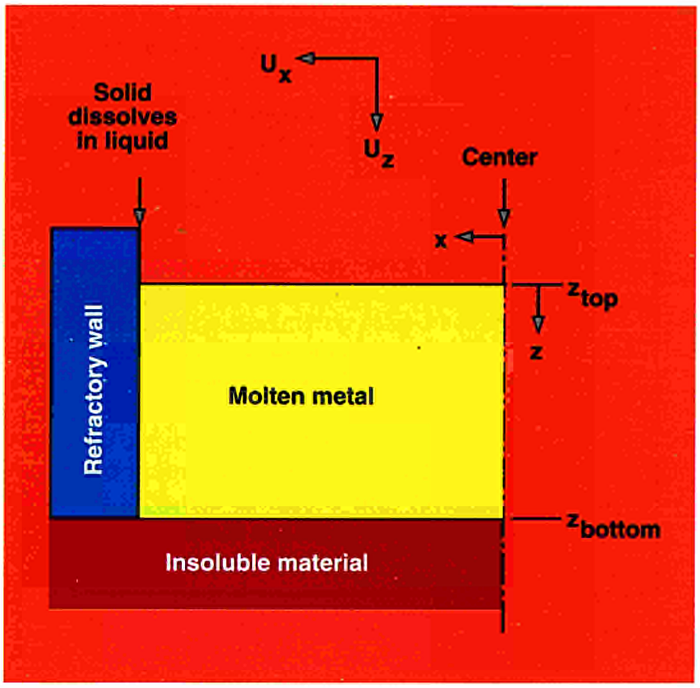
melt, shown schematically in Figure above. It is a characteristic of the furnaces used in the glass and steel making industries that heat is fed into the centre of the melt, e.g. by flame or electric arc, and that heat is lost from the melt by conduction through the walls of the crucible. This heat flux results in a temperature gradient in which the melt adjacent to the crucible wall is cooler, and therefore denser, than in the centre.
This temperature induced density gradient drives convection currents downwards at vertical crucible walls. This temperature driven buoyancy flow is the basic flow pattern common to all furnaces of this type.

Other flow patterns may be superimposed on this basic flow pattern if the crucible material is soluble in the melt. Dissolution of the crucible wall in the melt is a diffusion controlled process.
A concentration gradient of the solute in the melt is set up near the wall. The solute in the melt adjacent to the vertical wall changes the properties of the melt in two important respects and each of these changes generates a separate type of flow.

First, the melt density may either increase, in which case the basic flow pattern will be enhanced, (this case was not considered here), or the melt density may be reduced, in which case the flow pattern will be counteracted.
This is the concentration buoyancy flow.

Second, the change in the free energy of the melt due to the presence of solute will be manifest at free surface as a gradient in surface tension.
A gradient in surface tension will result in a surface flow pattern that reduces interfacial energy.
Therefore, solute which lowers the surface tension will direct fluid away from the crucible wall, and vice versa. This type of interfacial tension flow is known as Marangoni flow.

The relative magnitude and the interaction between the two types of buoyancy flow and the surface tension flow determines the rate of erosion and the shape of the erosion profile. The thermal and chemical energy which drives these flows is dissipated by the viscosity of the melt. The erosion phenomenon is therefore analyzed primarily in terms of the Grashof number (ratio of buoyancy to viscous forces) and the Marangoni number (ratio of surface tension to viscous forces).



Above: *Schematic representation of mathematical model*

In the present system the separate driving forces which are considered are shown as:

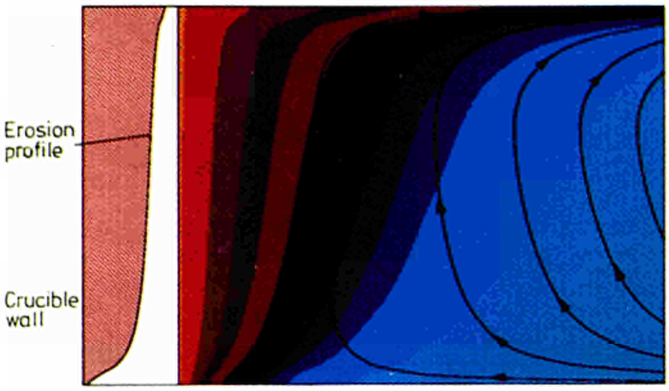
Figure a on page 83 pure buoyancy flows due to temperature gradients $Gr_T \neq 0$ and $Gr_c = Ma = 0$;

Figure b pure buoyancy due to concentration gradients $Gr_c \neq 0$ and $Gr_T = Ma = 0$;

Figure c due to positive capillary gradient, Marangoni $Ma > 0$ and $Gr_T = Gr_c = 0$;

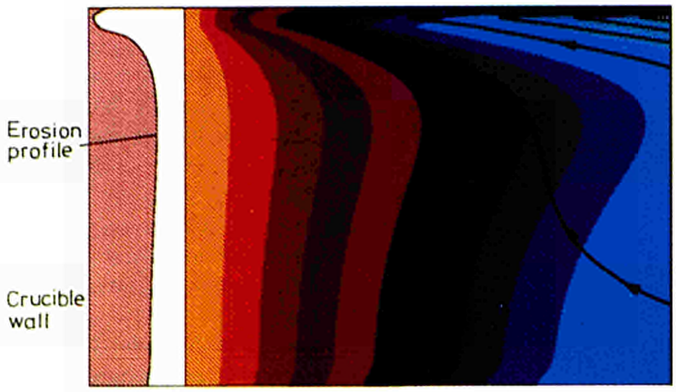
Figure d due to negative capillary gradient, Marangoni $Ma < 0$ and $Gr_T = Gr_c = 0$;

Here, we present only the contributions due to Marangoni forces.



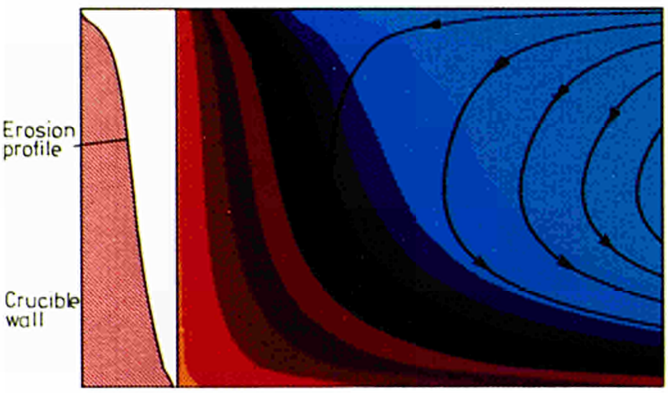
2b

A



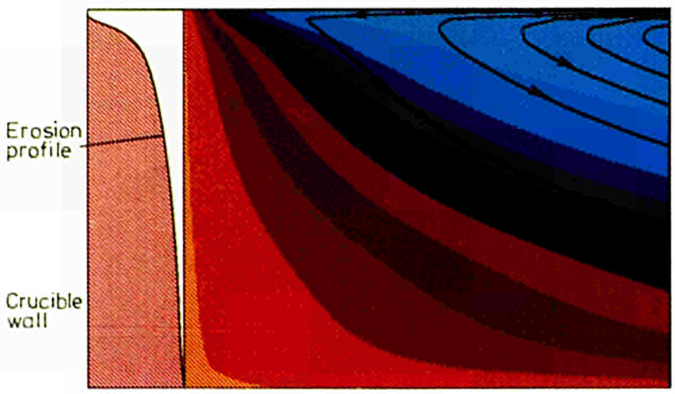
2d

B



2a

C



2c

D

Above: *Computed solute distributions and flow patterns, in the boundary layer close to the refractory wall, for different cases. Concentration ranges from that of the refractory wall (rose), to that of the bulk (blue)*

a) pure buoyancy flows due to temperature gradients;
$Gr_T = 1 \times 10^7$ *and* $Gr_c = Ma = 0$;

b) pure buoyancy due to concentration gradients;
$Gr_c = 0.5 \times 10^7$ *and* $Gr_c = Ma = 0$;

c) due to positive capillarity gradient,
$Ma = 0.25 \times 10^5$ *and* $Gr_T = Gr_c = 0$;

d) due to negative capillarity gradient,
$Ma = -0.25 \times 10^5$ *and* $Gr_T = Gr_c = 0$.

Classical Marangoni flow arises from differences in local surface tension as a result of thermal, chemical or concentration gradients (as in this case) appearing at the surface or interface of fluids, the direction of flow being from low to high surface tension. Positive Marangoni values correspond to systems where the presence of surface active species increases the interfacial tension of the medium, so that where such a species are diffusing from the wall the interfacial tension increases as it approaches the wall Figure c, where $Ma = 0.25 \times 10^5$ and $Gr_T = Gr_c = 0$.

It is seen that in the vicinity of the top surface, positive Ma values tend to drive the surface fluid towards the wall. Since Marangoni convective currents are a surface phenomenon, they create a steep velocity gradient in the liquid subsurface layers by the viscous shear effect of the fluid. The steep velocity gradients on the other hand create strong variations in the concentration gradients of the diffused wall material and since the currents sweep it away and replenish it continuously with new bulk molten material.

The erosion is enhanced at this point. In the rest of the bulk, the convection flows are negligibly small and the region is dominated by diffusion flux, creating rather homogeneous distributions, resulting in the absence of erosion profiles. This behaviour is clearly reflected in the distribution of the dissolved wall material. The erosion profile, in turn, shows relatively slow dissolution rates throughout, with the exception of a relatively narrow region in the vicinity of the top surface which erodes very rapidly. This erosion depth is a direct consequence of the surface driven flow.

Negative Marangoni values (Ma= - 0.25 x 10^5 and Gr_T=Gr_c=0) correspond to a system, where the presence of diffusing species decreases the interfacial tension of the solution.

In this case, the horizontal velocity component has the same direction as the diffusing flux. In Figure d, the corresponding flow patterns with the distribution of the diffused wall species are presented, indicating clock-wise currents at regions close to the surface.

Thus, "fresh" melt material is supplied from the bulk to the surface.

Therefore, in this case, it is observed that although the Marangoni convection is a surface effect, negative surface tension gradients create flows occupying nearly the whole volume of the molten fluid, which results in erosion profiles with maximum depths occurring just below the top surface.

However, this flow pattern creates relatively steep concentration gradients over a greater area of the refractory wall, creating rather different shapes of erosion profiles to those with Ma >0.

Ⅲ List of Publications

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Accepted for publication in Sensors and Actuators

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IV Meetings/ Conferences

Meetings/Conferences

Date	Venue	Subject Area	Type of Meeting	Co-organiser/Co-Sponsor
23/24.3	Petten	Materials for the Automotive Industry	Expert Meeting	Materials Consulting Group of the Joint Research Committee of European Car Industry
9/10.6	Biella	Impianto Solare Termico Innovativo	Seminar	DG XIII
30.6/2.7	Ispra	Utilization of Medical Radioisotopes	Eurocourse	DG XI
21/24.9	Petten	Ultra High Temperature Mechanical Testing	International Symposium	HTMT Committee, UK.
24.9	Petten	Ultra High Temperature Testing Methodologies for Materials	Expert Meeting	COST 510
25.9	Petten	Ceramics Testing	Working Group	VAMAS
28/30.9	Petten	Standards for Advanced Ceramics	Technical Committee and Working Groups	CEN TC184
30.9	Ispra	Evaluation of NDT results using Fracture Mechanics Methods	Workshop	Univ. of Florence
2.10	Ispra	Performance Demonstration for the qualification of Inspection Procedure	Workshop	
20.10	Paris	Classification of Advanced Ceramics	Working Group	CEN TC184
26/28.10	Petten	In-core Instrumentation and In-situ Measurements in Connection with Fuel Behaviour	Technical Committee	IAEA
2/3.11	Petten	Standards for CMC's	Working Group	CEN TC184
8/11.11	Rostov/Russia	Inservice Inspection Effectiveness and Reliability	Workshop	DG XI
10/13.11	Ispra	Advanced Techniques for Surface Modifications	Eurocourse	

Ⅴ Glossary

Glossary

ABAQUS	Finite Element Code
ACC	Advanced Coatings Centre
AMES	Aged Materials Expertises and Studies
APS	Atmospheric Plasma Spraying
ASME	American Society for Mechanical Engineers
AST	Austenitic Steel Testing
ASTM	American Society for Testing and Materials
BCR	Bureau Communautaire de Référence
BNCT	Boron Neutron Capture Therapy
BRITE	Basic Research in Industrial Technologies for Europe
BWR	Boiling Water Reactor
C/S	Code and Standard
CAE	Computer Aided Engineering
CDM	Continuum Damage Mechanics
CEA	Commissariat a l'Energie Atomique
CEASI	Concerted European Action on Structural Intermetallics
CEC	Commission of the European Communities
CEFIR	Ceramic Fibre Research (EUREKA project)
CEN	Comité Europeen de Normalisation
CFC	Carbon Fibre Reinforced Carbon Composites
CIM	Computer Integrated Manufacturing
C/M	Ceramic/Metal
CMC	Ceramic Matrix Composite
COST	European Cooperation in the Field of Science and Technical Research
COST 501	Advanced Materials for Power Engineering
CPP	Coatings Properties and Processes
CPAA	Charged Particle Activation Analysis
CSIC	Conseil Superior d'Investigaciones Cientifiques
CT	Compact Tension
CTK	Centrum voor Technisch Keramiek
CVI	Chemical Vapour Infiltration
DB	Data Bank
DBMS	Databank Management System
DG	Directorate General
DYNA 2D	Hydrodynamic finite element code
EB	Electron Beam
EBRII	Experimental Breeding Reactor II
EC	European Communities
ECN	Energieonderzoek Centrum Nederland
ECU	European Currency Unit
EFR	European Fast Reactor
EN	European Standard
ENEA	Ente Nazionale Energia Alternative
ENIQ	European Network for Inspection Qualification
ENV	European Pre-Standard
EPRA	European Pre-normative Research Association
ESCA	Electron Spectroscopy for Chemical Analysis
EURAM	European Research on Advanced Materials
EUREKA	European Research Coordination Agency
EUROS	European Remote encapsulation Operating System

EXOTIC	Extraction of Tritium in Ceramics
FALSIRE	Fracture Analysis of Large Scale International Reference Experiments
FE	Finite Element
FTIR	Fourier Transformed Infra-Red
GE	General Electric
GM	Geiger-Müller
HAZ	Heat Affected Zone
HCM	Human Capital and Mobility
HFR	High Flux Reactor
HSE	Health and Safety Executive
HTM	High Temperature Materials
HTR	High Temperature Reactor
HVOF	High Velocity Flame Spraying
IBAD	Ion Beam Assisted Deposition
IAM	Institute for Advanced Materials
IGRDM	International Working Group on Radiation Damage Mechanisms for Pressure Vessel Steel
ILL	Institut Laue Langevin, Grenoble
IIW	International Institute of Welding
IOD	Innovatief Onderzoeks Project
IRDAC	Industrial Research and Advisory Committee
ISI	In-Service Inspection
ISO	International Organization for Standardization
ITER	International Thermonuclear Experimental Reactor
JRC	Joint Research Centre
KECU	Kilo European Currency Units
KFA	Kernforschungsanlage Jülich
KFK	Kernforschungsanlage Karlsruhe
LCF	Low Cycle Fatigue
LME	Liquid Metal Embrittlement
LOCA	Loss of Cooling Accident
LWR	Light Water Reactor
MAN	Machienenfabriek Augsburg-Nürnberg
MCNP	Monte Carlo Neutron and Photon code
MEcu	Million European Currency Units
MOX	Mixed Oxide
MPA	Staatlich Materialprüfungsanstalt (Stuttgart)
MPS	Maximum Principal Stress
MS-DOS	Micro Soft Operating System
NAA	Neutron Activation Analysis
NDE	Non Destructive Evaluation
NDT	Non Destructive Testing
NEA	Nuclear Energy Agency
NESC	European Network for Evaluating Steel Components
NET	Next European Torus
NILOC	Nitride Fuel, Low Oxygen and Carbon
NOVEM	Nederlandse Organisatie voor Energie en Milieu
ODS	Oxide Dispersion Strengthened
OECD	Organization for Economic Cooperation and Development
ORR	Oak Ridge Research

PACVD	Plasma Assisted Chemical Vapour Deposition
PADS	Positron Annihilation Doppler Spectroscopy
PC	Personal Computer
PET	Positron Emission Tomography
PISC	Project for the Integrity of Steel Components
POMPEI	Pellets Oxide Mixte, Petten Irradiation
PTS	Pressurised Thermal Shock
PVDF	Polyvinylidene Fluoride
PWR	Pressurized Water Reactor
R&D	Research and Development
RBE	Relative Biological Effectiveness
RBS	Rutherford Backscatting
REFLEXAFS	Extended X-ray Absorption Fine Structure in Reflexion Mode
RPV	Reactor Pressure Vessel
RRT	Round Robin Test
RT	Radiographic Technicues
RWE	Rheinisch Westphälische Elektrizitätswerke
SAS	Standard Acoustic Source
SEM	Scanning Electron Microscopy
SEP	Société Européene de Propulsion
SGT	Steam Generators Tubes Testing
SOC	Sulphidizing/Oxidizing/Carburizing
STEP	Science and Technology for Environmental Protection
TC	Technical Committee
TEM	Transmission Electron Microscopy
TLA	Thin Layer Activation
TLD	Thermoluminescence Detectors
TMF	Thermo-Mechanical Fatigue
TMI	Three Mile Island
TU	Technical University
UE	Ultimate Elongation
VAMAS	Versailles Agreement on Advanced Materials and Standards
Vbf	Voltage Breakdown Forward
VPS	Vacuum Plasma Spray
WG	Working Group
WP	Work Package
YS	Yield Strength

Ⅵ List of Authors

List of Authors

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II. **Scientific - Technical Achievements**

Specific Programme: Advanced Materials

RESEARCH AREA: MATERIALS FOR EXTREME ENVIRONMENTS

Assessment of Research Area Progress and Achievement: M. v.d. Voorde

Engineering materials in industrial/emission atmospheres: J.F. Norton, D. Baxter, R.J. Fordham

H.T. Corrosion resistance of alloys and coatings: F. Brossa, F. Coen

Characterization of intermetallics and refractory alloys: J.B. Marriott, V. Guttmann

Characterization of H.T. alloys: J.B. Marriott, V. Guttmann

Performance improvement of ceramics composites and alloys: J. Bressers, M. Steen, J.N. Adami, C. Saraiva Martins, J. Timm

RESEARCH AREA: RELIABILITY AND LIFE EXTENSION

Assessment of Research Area Progress and Achievement: P. Schiller

Structural performance of advanced materials: J. Bressers, M. Steen, J. Timm, L. Vallès

Component integrity testing and evaluation: R.J. Hurst

Thermal fatigue of components: J. Bressers

Flaw sizing in advanced materials by ultrasonics: R. Denis, P. Lemaitre

Performance improvement of composite materials: H. Stamm, D. Boerman, F. Dos Santos Marques, A.M. Morrissey, R. Scholz

RESEARCH AREA: MEASUREMENT AND VALIDATION METHODOLOGIES

Assessment of Research Area Progress and Achievement: H. Kröckel

Materials aging and degradation monitoring, completion of PISC: S. Crutzen, E. Borloo, U. von Estorff

Application of neutrons for CMC and coatings characterization: J. Bressers, A.G. Youtsos

Physical properties of coatings: P.N. Gibson

Characterization of ceramic surfaces by positron annihilation: M. Forte

Pre-standards activities: J. Bressers, M. Steen

RESEARCH AREA: SURFACE MODIFICATION TECHNOLOGY

Assessment of Research Area Progress and Achievement: P. Schiller, H. Kröckel

Wear and corrosion resistant coatings: E. Bullock, E. Lang, F. Rossi

Wear and corrosion resistant coatings based on nanodispersive systems: W. Gissler, J. Haupt

Surface/bulk structural properties modification: F. Lanza

Sub-microstructural engineering: S. Pickering, B. Djurcic, D. McGarry

Development and coating of ceramic fibres for H.T. composites: E. Bullock

Advances in ceramic joining: S.D. Peteves

Chemical sensors and electrocatalytic materials: G.B. Barbi

Surface scaling: M.F. Stroosnijder

RESEARCH AREA: MATERIALS INFORMATION AND DATA MANAGEMENT

Assessment of Research Area Progress and Achievement: H. Kröckel

Coatings processes data banks: Z. Diamantidis

HTM data banks: H. Over

Information centre: M. Merz

SPECIFIC PROGRAMME: FUSION MATERIALS

Assessment of Research Area Progress and Achievement: P. Schiller

Low activation material: P. Fenici

Thermal Fatigue: R. Matera

Blanket studies: P. Fenici

Fusion materials data bank: A.G. Youtsos

SUPPLEMENTARY PROGRAMME

Irradiation Experiments in the High Flux Reactor Petten: R.L. Moss

Operation of the High Flux Reactor: J. Ahlf

S/T SUPPORT TO SERVICES OF THE COMMISSION

Standards for advanced ceramics (DG III): M. Merz, D. Baxter, J. Bressers, P. Lemaitre, M. Steen

Standards NDT for pressure vessels (DG III) and Performance demonstration of NDE techniques (DG XVII): S. Crutzen, E. Borloo, P. Lemaitre

Materials science and technology for aeronautics applications (DG III): M. v.d. Voorde

Standardisation of radiopharmaceutics (DG XI): L. Manes

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The rôle of materials in environmental problems arising from power stations (DG XI): M. v.d. Voorde

Materials databanks (DG XIII): H. Kröckel

Technology transfer and utilisation of research results (DG XIII)

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b. Ultrasonic reference transducers: R. Denis, F. Lakestani

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European Communities - Commission

EUR 15125 EN — Institute for Advanced Materials, Annual Report 1992

M. Merz, editor

Luxembourg: Office for Oficial Publications of the European Communities

1993 - 108 pages - 21.0 x 29.7 cm

Series: Physical Sciences

EN

Catalogue number: CD-NA-15125-EN-C

Abstract

The Annual Report 1992 of the Institute for Advanced Materials of the JRC highlights the Scientific Technical Achievements, resulting from the execution of

- *the specific R&D programme on "Advanced Materials",*
- *the contribution to the specific programme "Controlled Thermonuclear Fusion"*
- *S/T Support to the Services of the Commission,*
- *Exploratory 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: **M. de Groot, B. Seysener** and **F. van der Smaal**
Text Pre-processing: **Y. Den Biesen-Hey**
Lay-out and Photosetting: **J. Manten**
Printing: **Van Marken Delft Drukkers**
Cover Design: **J. Wells**

16

CD-NA-15125-EN-C

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