ANNUAL · REPORT · 93

Institute FOR Advanced Materials

JOINT RESEARCH CENTRE

COMMISSION OF THE EUROPEAN COMMUNITIES

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I Introduction

Introduction

Here we report on progress during the middle year of our current 3 year multiannual research programme. The activities of this programme have matured sufficiently for us to assess their progress and to reflect on the future directions for research within the Institute. The Institute must remain responsive to the needs of European industry and we should ensure that our contribution complements rather than competes with the activities of other European research centres the principle of subsidiarity. In research, responsiveness means anticipating developing trends rather than just reacting to the existing situation, and this requires a high degree of awareness of current thinking in each research area.

In this context we welcomed the contributions of the two Visiting Groups of acknowledged experts in the fields of materials science and nuclear engineering who reported their assessments of our current work and their recommendations for the future. These recommendations confirmed and reinforced many of the ideas that we were already picking up through our contacts in research and industry on scientific developments and, in addition, also provided valuable input on how the Institute should best respond to these challenges. We have drawn on these ideas in revising our document "Strategy for the Institute for Advanced Materials" to reflect the latest thinking on the future role of the Institute. As detailed below, the theme of materials for high temperatures with an emphasis on the role of interfaces in determining materials properties is being strengthened and the European dimension of the work receives more emphasis, as does the application to environmentally clean technologies and recycling.

Visiting group reports

The visiting groups commented favourably on the management and execution of the current research programme. The scientific work was judged to be well targeted and of international standard; the quality of service given to HFR customers was excellent. For the future, the Institute should concentrate on its core activities in the field of advanced high temperature materials superalloys, ceramics, composites, intermetallics and interfaces in materials should be a major scientific theme. The Institute should move towards a "core + competitive" funding model in which about 20% should be obtained through competition in Framework Programme schemes such as BRITE EURAM. In addition, contract research should expand further over the next few years.

The Institute should widen its European role through measures such as the organisation, management and participation in networks and the exchange of staff.

The HFR is facing a rapidly changing situation which requires a vigorous reorientation towards a new customer base following the rapid decline in demand from traditional customers in the fission community. Production of radio isotopes for European industry and medical purposes on a commercial basis is seen as an opportunity, with the fusion programme, BNCT cancer therapy and Community based fission programmes providing additional activities.

Institute budget and manpower resources

The budget and manpower available to implement the programme in 1993 were:

Programme	Scientific Research Staff	Research Budget (Kecu)
1. Specific Programme		
Advanced Materials	108	2200
Fusion Materials	16	280
2. Support to the Commission	18	960
3. Exploratory research	6	290
4. High Flux Reactor:		
Complementary	39	200
Common	1	30
Totals	188	3960

Notes

1. The research budget for the HFR excludes the reactor running costs.
2. The above scientific activities were supported by 29 technical and 39 administrative personnel.

In addition to the above resources, 12 research staff were engaged in contract work for third parties. Contracts to a value of more than 7 MECU were signed in 1993 for execution in 1993 and later years. The level of work for third parties, currently running at about 8% of the total budget, continued to progress on schedule towards our target of 10%. The most significant single achievement was the signing of a contract for 2.6 MECU with an Italian enterprise.

Whereas the overall budget, and especially that for the specific programme, was significantly lower than in 1992, the Human Capital and Mobility programme enabled us to increase significantly the number of research workers in the Institute.
By the end of 1993 there were about 40 grant holders, most of them post doctoral research fellows.

Transfer of NDE to Petten

The movement of the non destructive evaluation group from Ispra to Petten began in 1993 with the transfer of 10 staff and the installation of a high energy X-ray source in a purpose built shielded bunker. This coincides with the development in Petten of several European wide Networks involving materials and component integrity and where the Institute will take the coordinating and management role.

Scientific achievements

Four areas in which significant progress was made illustrate various aspects of the importance of interfaces in materials:

- carbon fibre reinforced silicon nitride ceramics

Encouraging progress was made in the synthesis of silicon nitride ceramic matrix composites reinforced with pitch based carbon fibre in which the strength of the fibre matrix interface was engineered by coating the fibre.
Techniques for infiltrating fibre weaves and hot pressing were perfected to the extent that the influence of the fibre coating could be clearly demonstrated and excellent mechanical properties were obtained in roomtemperature tests due to the high degree of fibre pull out that was achieved.

- gas sensors

Semiconductor sensors based on tin oxide have been developed with a sensitivity sufficient for monitoring environmental levels of nitrogen dioxide and carbon monoxide. These sensors are intended for the mapping of urban areas where a large number of small, low cost sensors are required. These sensors have been developed to the point where they are ready for practical application.

- plasma ion nitriding

Plasma ion nitriding has been applied to improving the lifetime of cermet coated tool steel drilling heads for use in off shore drilling for oil. Nitriding can confer additional stiffness and fatigue resistance to the steel surface prior to thermal spraying with the cermet, but this is usually achieved at the expense of degradation of the tempered microstructure of the bulk material. In the plasma nitriding process, however, the usual thermally activated dissociation of nitrogen gas is replaced by glow discharge ionisation which has the advantage of lowering the treatment temperature and thereby avoiding microstructural degradation of the bulk. In service testing of a drill head is now in progress.

- cubic boron nitride films

Cubic boron nitride approaches diamond in hardness and is therefore of great interest as a hard coating material. However, hexagonal boron nitride, which has inferior properties, is more stable and will be formed unless energy is supplied to the growing film in excess of that supplied by the thermochemical processes involved. A dual ion beam deposition facility has been developed for this purpose and was optimised to the extent that films containing up to 75% cubic phase can be produced. The momentum transfer per depositing atom was identified as the most important parameter for determining the formation of the cubic phase.

- Workshop on "Materials for Coal Gasification"
A workshop on materials for coal gasification was held at Petten in June 1993 and was attended by more than 60 delegates form Europe, North America and Japan. This event, which brought together materials scientists, plant operators and process designers, coincided with the start up of the first commercial plant in the Netherlands.

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

Existing materials operate at the limit of their capabilities. Further efforts are necessary to develop new materials for reliable use in demanding operating conditions for example in aero-engines, 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 two 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,

The high temperature (up to 1400°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 materials testing laboratories have been significantly expanded during 1993, experiments can now be carried out on ceramic materials under dynamic burner rig conditions at temperatures up to 1400°C in a thermal cycling mode. Testing equipment for corrosion deformation interaction studies in a non-equilibrium atmosphere has been commissioned. A reversed bending jig was constructed which allows through zero flexural fatigue tests at high temperatures.

ii) Study of Materials degradation Mechanisms

An in-depth mechanistic understanding of H.T. steels degradation in an industrially relevant, non-equilibrated, gaseous corrosive atmosphere has been developed thereby enabling the applicability of laboratory testing in model (equilibrated) environments to be assessed.

The potential of the ODS alloy MA 760 for application in uncooled vanes of stationary gas/turbines was explored. Studies on the assessment of the damage and failure modes in ZGF and TMF have been rounded off. This work was a useful contribution to the prediction of the lifetime under simulated service loads, and carried out in the context of COST 501.

The ordered intermetallic alloy $Ti_5(Si\ Al)_3$ - $Ti_3(Al\ Si)$ has been studied. The material shows promising high creep strength but severe problems related to ductility have still to be overcome.
This new research activity will be integrated in the European network on Intermetallics.
New coating systems, for super alloys (IN738 and UD520) used in power plants based on the composite NiCoCrAlY/ Al_2O_3, have been developed with improved oxidation, corrosion and thermo-mechanical properties at high temperatures.

Monolithic phases of the types found at the grain boundaries of Si_3N_4, have been shown to exhibit superior corrosion resistance compared with the behaviour of bulk Si_3N_4 during exposure in low-oxygen atmospheres; this is in marked contrast to earlier findings in highly oxidising atmospheres.
Pre-H.T. Corrosion exposure of a hot pressed silicon nitride in coal gasification environments, results in a considerable improvement of the long term creep properties under uniaxial loading. These results tend to differ from those being obtained in real plant conditions where chemical attack and mechanical load operate simultaneously.
An in-depth literature review has been made on cyclic mechanical fatigue effects in monolithic ceramics. An international workshop on "Materials for Coal Gasification Power Plant" was held in June 1993. This event was co-organised with EPRI (USA) and attracted participants from all the world's industrial nations. The proceedings have been published.

Engineering Materials in Industrial/Emission Atmospheres

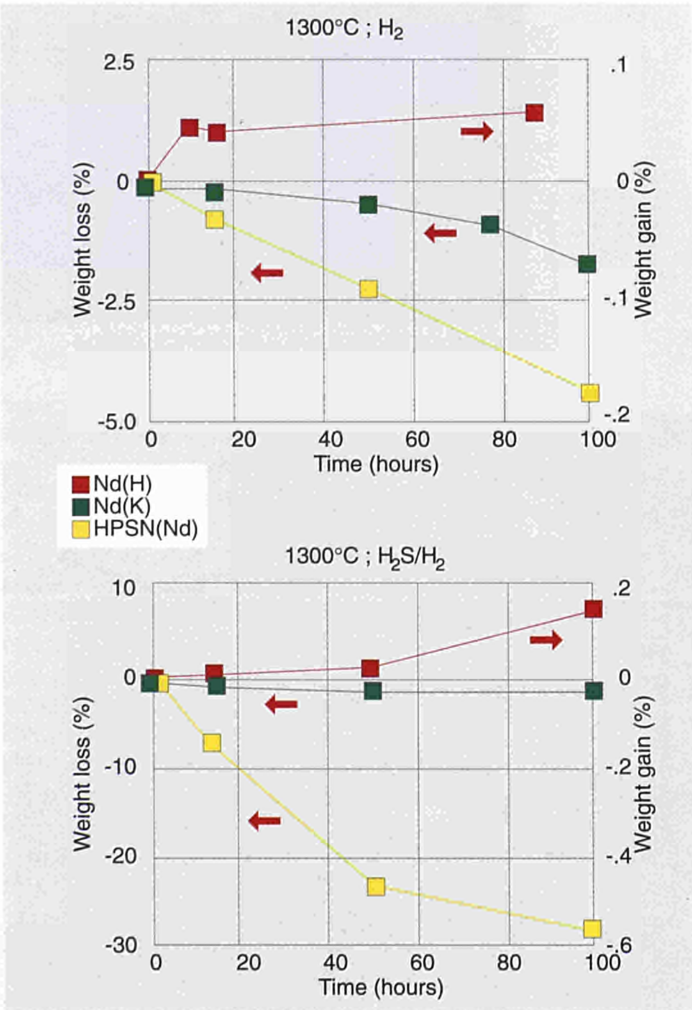
This project seeks to determine the kinetics and mechanisms of corrosive degradation in advanced engineering materials in multi-reactant gaseous atmospheres. The synergistic influence of salt contaminants and acidic condensates is also an important aspect of this study which, in addition, seeks to establish the industrial applicability of such laboratory-derived data.

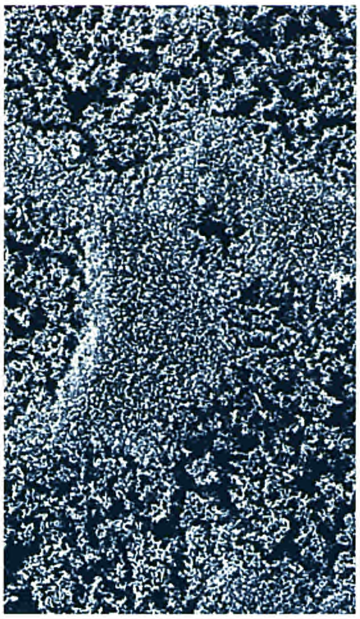
The study has focused on the effects of high temperature exposure on the physical integrity and flexural strength of 3 hot-pressed grades of Si_3N_4, i.e. a commercial 9%Y_2O_3 material; a small-grain, high strength development material (made with Japanese UBE powder) containing 2%Al_2O_3 and 5%Y_2O_3; and a grade containing 3%Al_2O_3 and 8%Y_2O_3. These exposures were made in a burner rig at 1000^{0}C to 1300^{0}C under conditions designed to simulate particular aspects of a gas turbine combustion environment. A second burner rig having a higher temperature capability of 1400^{0}C, has also been installed.

Kinetic studies and associated activation energy calculations show that the surface reaction was essentially oxidation at temperatures above the dew point of the Na_2SO_4, although Na is found throughout the surface corrosion products. The presence of Na appears to reduce the temperature and/or the rate of devitrifaction of SiO_2 but contributes significantly to silicate formation only at temperatures around 1000^{0}C. Room temperature flexural strength was reduced by exposure to the corrosive environment. Further work will establish the individual contributions to reduced strength by corrosion (production of surface flaws) and annealing of the base material (modification of the grain boundary phase).

Parallel studies on the corrosion behaviour of Si_3N_4 and SiC in purely gaseous atmospheres continued. The corrosion properties of these materials are markedly affected by the nature and amount of the "secondary" phases. The behaviour of selected Si_3N_4 intergranular phases [Y-, Ce- or Nd-containing silicates] in monolithic form remained an important aspect of these studies, particularly in low oxygen-activity environments where severe corrosion due to "active" oxidation occurs - a mechanism which involves the formation of volatile SiO instead of the stable condensed phase, SiO_2.

Below: *Mass change as a function of time for a silicon nitride and selected secondary phases at 1300^{0}C in H_2 (above) and H_2/H_2S (below)*





HPSN (Nd) —— 100 µm

Nd-N-apatite —— 20 µm

Above: *Surface morphology of Si_3N_4 (left) and Nd-N-apatite (right) after exposure to H_2/H_2S at 1300°C*

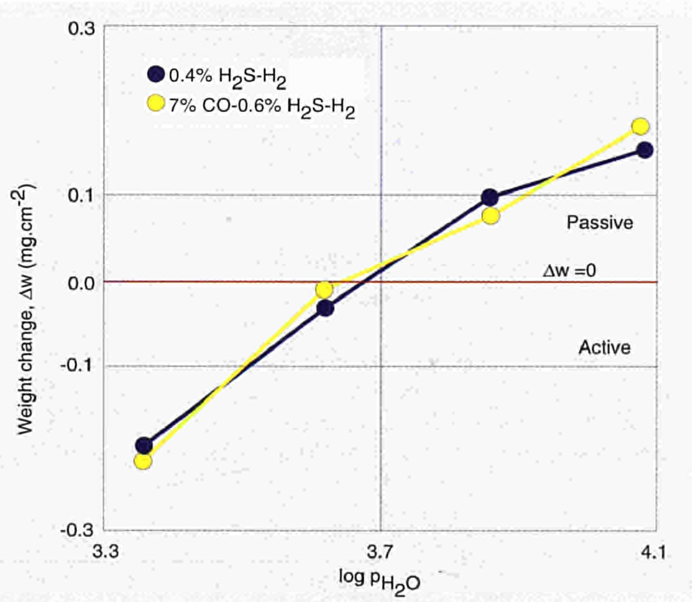
Below left: *Influence of water vapour on extent of oxidation of HIPSiC at 1300°C*

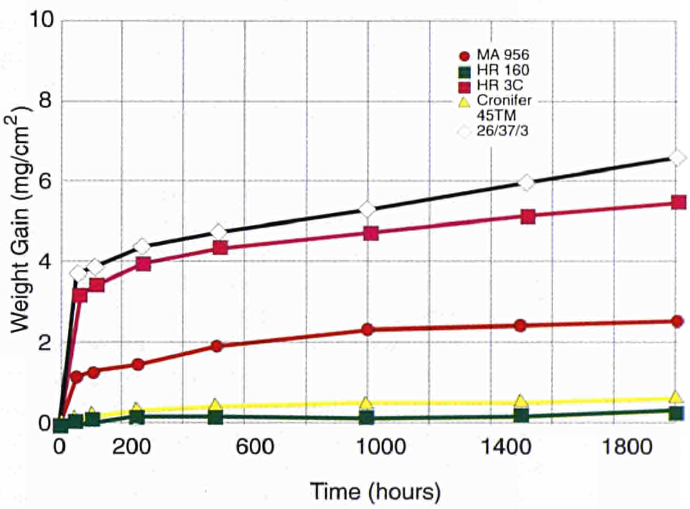
Below right: *Kinetic data for the five alloys after exposure to $0.2H_2S$-64CO-3,8CO_2-H_2 bal at 600°C for 2000 h*

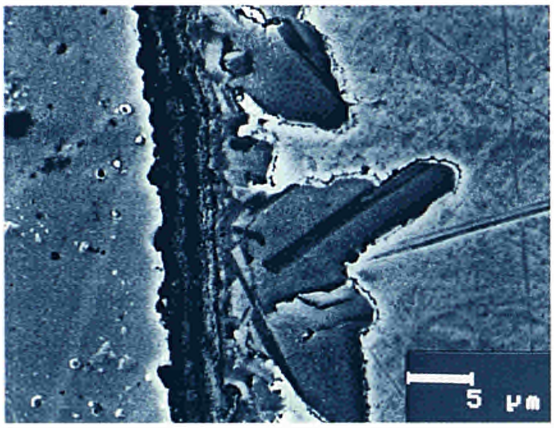
In contrast to the behaviour of the intergranular phases in oxygen-rich atmospheres (when they show a marked tendency to oxidise to form silicate glasses which are often liquid at quite low temperatures), the results of exposures in H_2 or H_2-H_2S mixtures show that they have better resistance than the bulk Si_3N_4.

Figure below on page 3 illustrates the mass-change of Nd-N-apatite [Nd(H)], Nd-α-wollastonite [Nd(K)] and hot-pressed Si_3N_4 containing Nd-N-apatite as the principle secondary phase, as a function of time and environment at 1300°C. Figure above compares the resulting surface morphology of the monolithic Nd-N-apatite and the Si_3N_4. It is evident that the Si_3N_4 surface has become very porous as a result of the selective loss of Si_3N_4 grains leaving intergranular material, transformed to Nd-silicates or $Nd_2(O,S)_3$ depending on the environment.

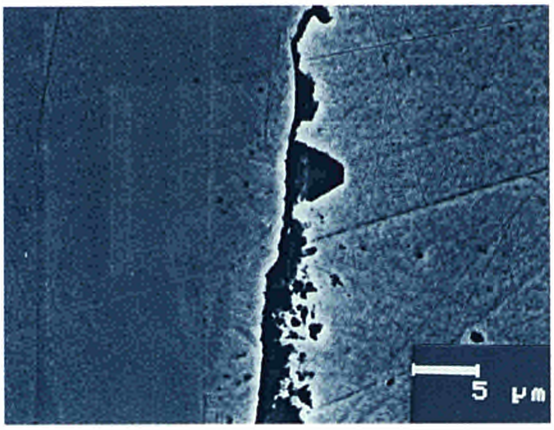
Studies are in progress to define the active/passive oxidation transition region for Si_3N_4's and SiC's. Figure below left illustrates the influence of pH_2O on mass-change of a hipped SiC for a fixed period of 50h at 1300°C in different environments - mass gains represent passive oxidation with the formation of SiO_2 whilst losses represent active oxidation to SiO. Clearly, the influence of water vapour is of greater significance than differences in oxygen content of the environment.



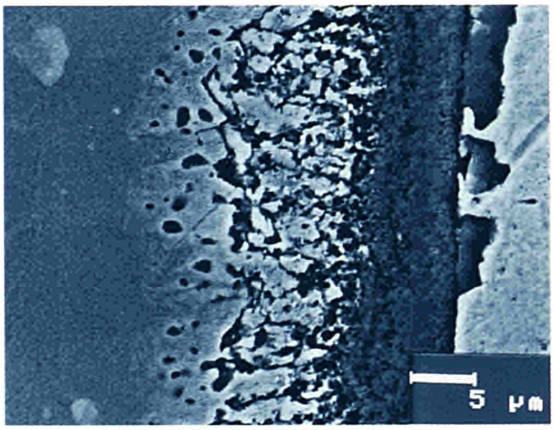




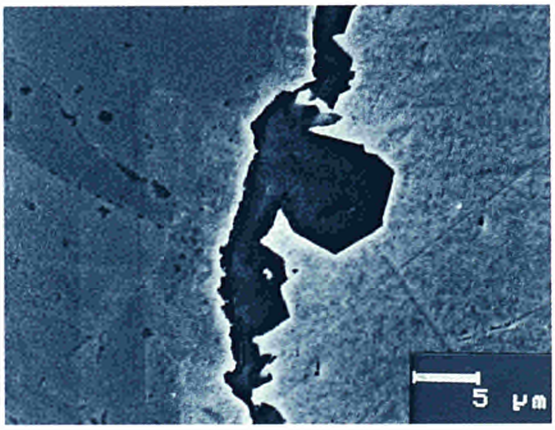
a. MA 956



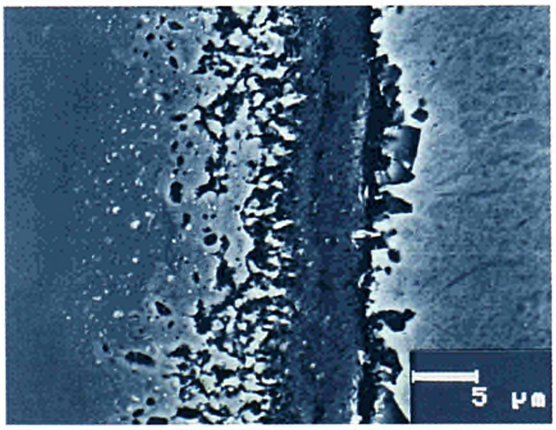
b. HR 160



c. HR 3C



d. Cronifer 45TM



e. 26/37/3V

Above: *Cross sections of all the alloys after exposure to 0.2 H_2S - 64 CO - 3.8 CO_2 - H_2 bal at 600^0 for 2000 h.*

Experiments using a CO-based gas mixture representing that of a dry-feed entrained slagging coal gasifier are being carried out in collaboration with EPRI, USA. Data for in this non-equilibrated atmosphere are being compared with data from a similar study using an equilibrated gas (which attempts to model the S, O, and C activities), thus enabling the validity and applicability of laboratory-derived results to be assessed.

Samples from candidate alloys, i.e. Ma 956, Hr 160, Hr 3C, Cronifer 45 and a newly developed 26Cr-37Ni alloy containing 3%V, have been exposed at 600^0C for 2000 hours. Corrosion kinetics, expressed as mass-change measurements, are compared in figure below right on page 3. Structural analyses have been completed on specimens discontinued after test durations of 100, 500, 1000 and 2000h. Cross-sections from samples of each alloy exposed for 2000h are compared in figure above. Tests in the equivalent equilibrated gas mixture have reached 1000h and are continuing.

High Temperature Corrosion Resistance of Alloys and Coatings

Table below: *Chemical Composition of Base Alloys*

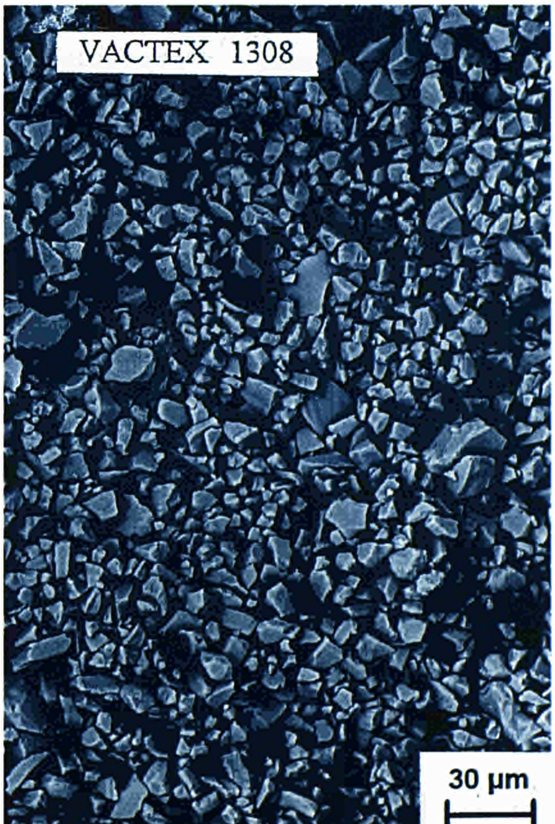
ALLOY	CHEMICAL COMPOSITION (wt%)																		
	Al	B	C	Co	Cr	Fe	Mn	Mo	N	Nb	Ni	P	S	Si	Ta	Ti	W	Zr	Altri
IN738	3.5	0.01	0.09	8.5	15.7	0.5		1.8			bal			0.2		5.0	1.25		
UD720	2.5	0.02	0.04	14.0	17.8	0.16	0.01	3.1			bal			0.21		4.8	1.3		

Chemical composition of Coatings

COATING	CHEMICAL COMPOSITION (wt%)						
	Al	Co	Cr	Ni	Si	Ta	Y
AMDRY 995	8.0	bal	21.0	32.0			0.5

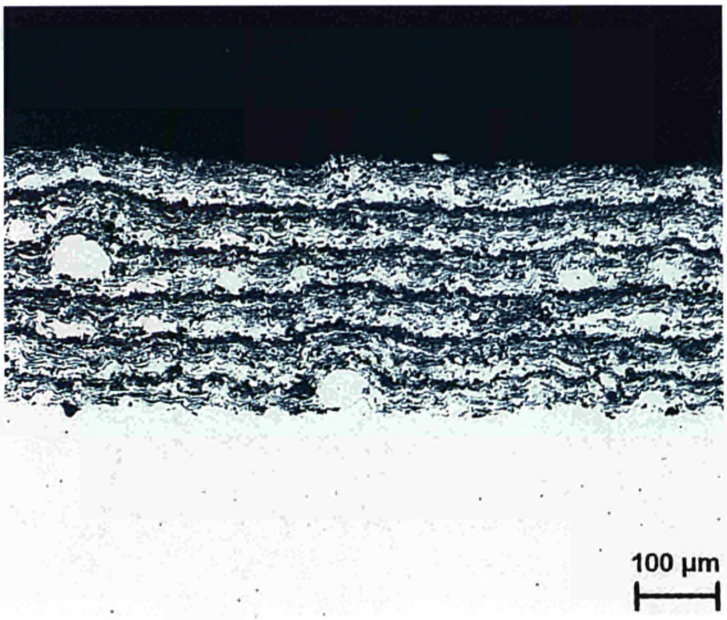
The objective of this work is the production and characterization of new coatings to reduce the oxidation and corrosion at high temperature in super alloys used in power plants. The alloys considered were: IN 738 and UD 520 (see table above) and metal-ceramic layers, of NiCoCrAlY/Al_2O_3 were used as coatings. The technique used for coating was Vacuum Plasma Spray (VPS). The ceramic component distributed in the metallic phase is expected to reduce the diffusion process in gaseous state (reducing the oxidation and corrosion of the outside of the coating), as well as in solid state, slowing down the interaction at the interface of the coating with the base material. Furthermore, these composite coatings should offer a better resistance to combined corrosion/erosion and thermal fatigue.
The composition of NiCoCrAlY powder (AMDRY 995) is described in table above. Two types of alumina, differing in particle size granulometry, were used. The larger grain size alumina was METCO 105 from METCO ITALIANA, while the smaller grain size alumina was VACTEX 1308 from PLASMA-TECHNIK AG SWITZERLAND. The morphology of both powders is shown in figure below.

Below: *Alumina powders, METCO 105 and VACTEX 1308 with two different grain size for composite coatings*



For both powders, coatings were produced with 25wt% and 40wt% Al_2O_3. During spraying, the two components tended to separate, producing stratified layers varying in oxide content. Figure above illustrates this phenomena: the grey phase is Al_2O_3 and from the flatness of the layers we can deduce that the ceramic had completely melted and was very fluid. The smaller grain size Al_2O_3 (VACTEX 1308) produced more homogeneous coatings as shown in figure below.

The surface of the samples was prepared by sanding at 6 atm, using 60 mesh corundum followed by sputter cleaning just before the coating in VPS as shown in table below. Finally, a bonding treatment was made at 1080°C for 4 hours at 2.10^{-6} torr. EDAX analysis of the coating surface showed an almost continuous layer of Al-rich material. It appears that the Al_2O_3 was floating on the NiCoCrAlY metallic phase. X-ray analysis indicates the presence of αAl_2O_3.



Above: *AMDRY 995 + 40% Al_2O_3 (METCO 105) VPS deposited on IN738*

Below: *Composite layer AMDRY 995 + 50% Al_2O_3 (VACTEX 1308) VPS deposited on IN738*

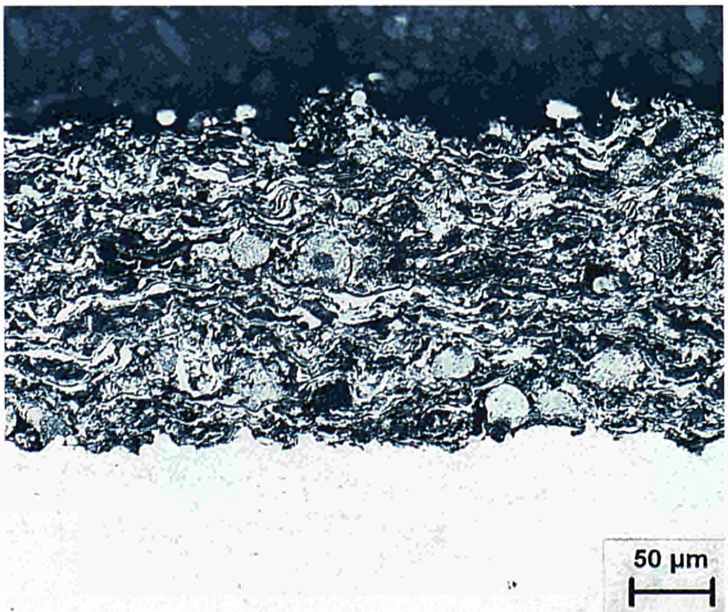
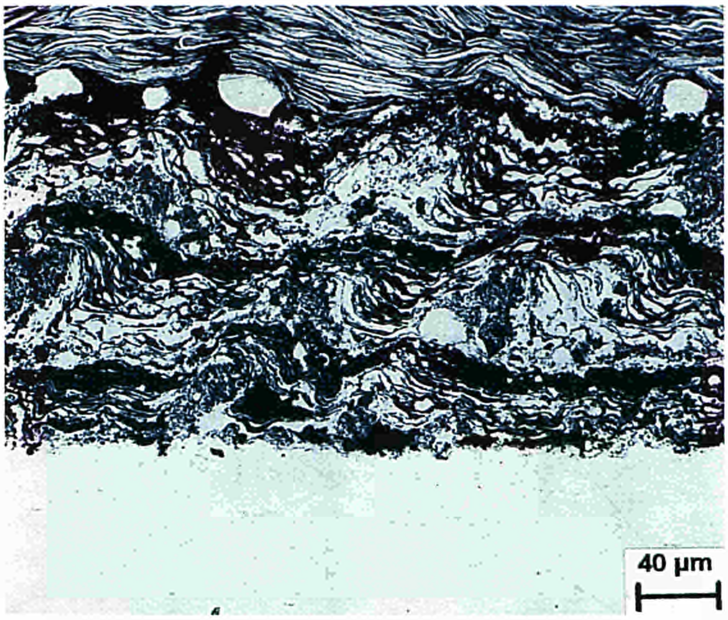


Table below: *Spraying Data for Metal-Ceramic Coatings*

	SPUTTERING	VPS DEPOSITION
Chamber pressure	20 mb	150 mb
Distance	240 mm	240 mm
Primary gas Ar	40 slpm	50slpm
Secondary gas H_2	1 slpm	1 slpm
Carrier gas Ar	-	2.2 slpm
Arc current	300 A	600 A
Sputtering current	40 A	-
Arc voltage	43 V	45 V
Powder feed	-	4 g/min

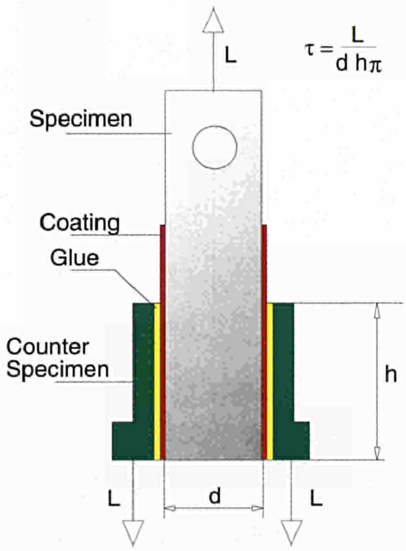


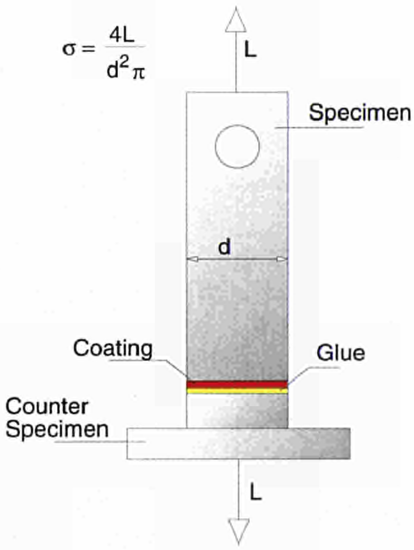
Above: *Coating shown in figure on page 7 below after thermal shock test (4 kW/cm² for 10 ms). The white zones are metallic droplets produced under thermal test.*

Below: *Specimens and set up for (a) shear stress and (b) tensile stress test*

The cohesion and adhesion of these coatings was tested on samples with 25wt% and 40wt% Al_2O_3 in the coating after the bonding process. The Pull Off test (ASTM C 633-79) was used for shear and tensile tests, using epoxy resin (ARALDITE AV 199). Figure below shows the set-up for shear and tensile tests. In most cases, rupture occurred at the resin/sample interface, with values of 40 to 50 MPa for the shear test and of 60 to 75 MPa for the tensile test. The samples of UD 520, coated with the two Al_2O_3 content coatings, were exposed to thermal shock using an Electron Beam Gun. The exposure time for all samples was fixed at 10ms, and the power density was varied from 2 KW/cm² to 12KW/cm².

A thermal shock test at 4 KW/cm² produced droplets in the metallic phase at the surface of the coating (figure above). Increasing the energy during the thermal shock, the surface of the film melted, with the separation of the metallic phase from the oxide in the melted zone. The lamellar structure and the presumably low thermal conductivity of such films appear to have contained the effects of the thermal shock. This interesting result will be investigated further with additional tests, including thermal cycling. Tests of thermal stability of these coatings are now underway and we are now considering the possibility of oxidation tests running in parallel.





Characterization of Intermetallics and Refractory Alloys

The investigations of the creep and creep damage of the ordered intermetallic alloy $Ti_5(Si,Al)_3/Ti_3$ (Al,Si) were completed. The material was developed in the framework of a German high temperature material programme under the leadership of the Max Planck Institute, Düsseldorf. Complementary characterizations of this alloy were carried out by the MPI, with respect to the room temperature mechanical properties and to tensile tests at elevated temperatures. Titanium aluminides based on Ti_3Al or TiAl are generally promising candidates for use at higher temperatures, mainly because of their high specific creep resistance. Problems however always arise from insufficient creep ductility. The investigated alloy showed a creep resistance slightly better than Ti_3Al but the addition of Si, which leads to the second phase $Ti_5(Si,Al)_3$ did not give the expected level of improvement. Moreover, as illustrated by figure below, extensive investigations of the creep ductility have shown that serious problems can arise at 700^{0}C when high creep rates are applied.

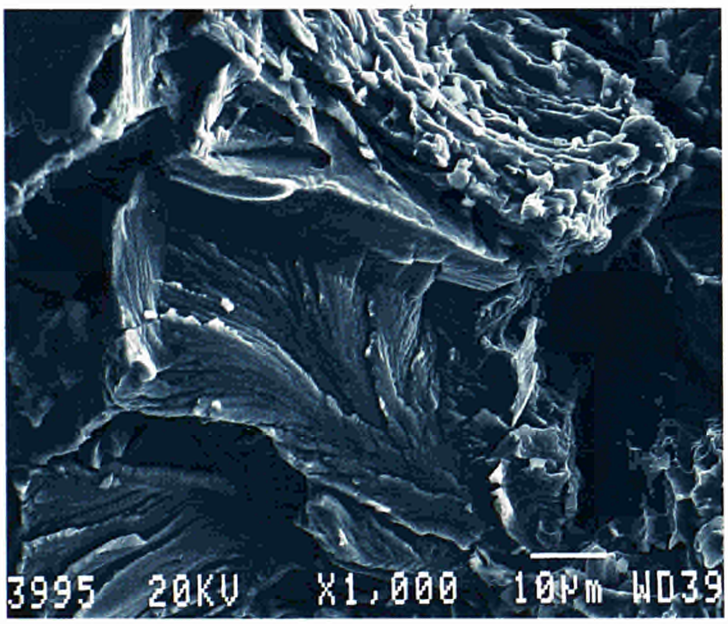
In contrast, at 700^{0}C at lower creep rates, or at any creep rate at 800^{0}C the ductility is relatively high. In the case of low ductility, a large part of the rupture surface shows brittle fracture whereas, for instance at 800^{0}C, brittle fracture occurs locally and only in regions with unfavourable grain orientations.

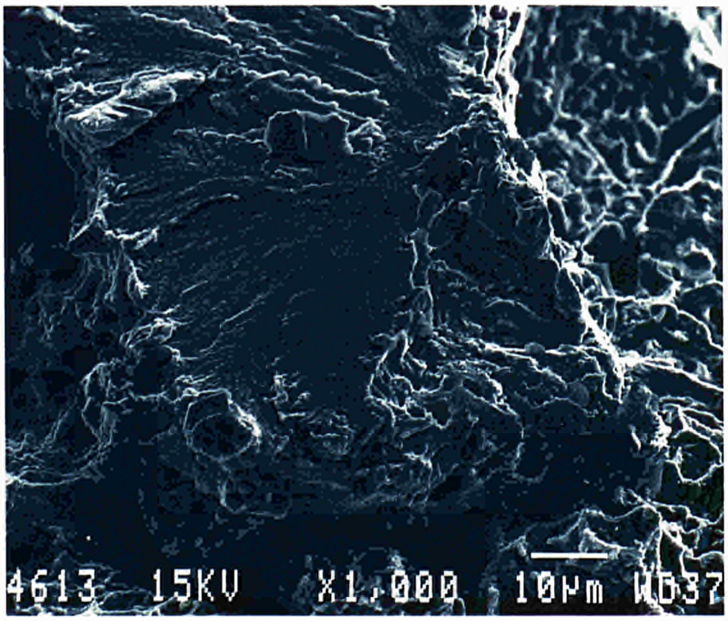
This feature of reduced ductility was previously unknown. There was previously no indication that grain boundaries play a decisive role for embrittlement and that intergranular fracture was the main fracture mode at lower temperatures.

Further investigations have been started in collaboration with the Max Planck Institute, Düsseldorf, on a newly developed Ti-Al-(Cr) alloy which is expected to offer improved creep strength and creep ductility.

In this context a comparison between the creep properties of a cast and a forged version of the material will be made.

Below: *Fracture surfaces of $Ti_3(Al,Si)$-$Ti_5(Si,Al)_3$ after testing at a) 700^{0}C, 35 h; b) 800^{0}C, 2103 h.*





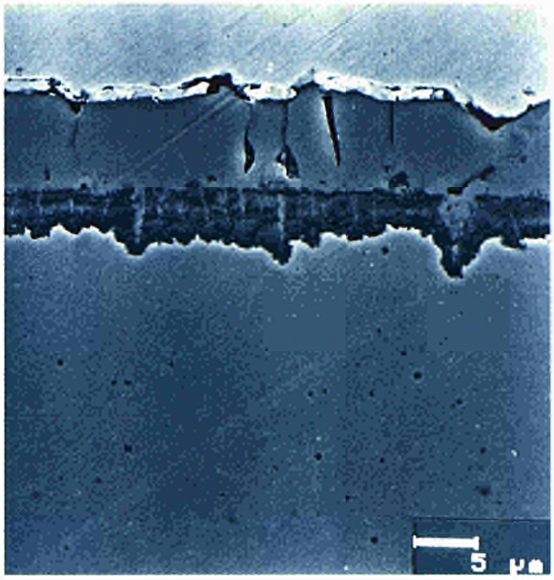
Characterization of H.T. Alloys

Investigations were started on the interaction of deformation and corrosion for a series of advanced high temperature alloys at 600°C using a sulphur-oxygen-carbon containing environment.

This work aims to simulate the corrosive conditions in heat exchanger tubes of the first heat recovery system in coal gasifier plants. This work requires that the atmosphere is maintained in a non-equilibrium state.

Under service conditions, the gas cools from the reaction temperature of about 1300°C too rapidly to achieve equilibrium. The test facilities were upgraded to handle the non-equilibrated gas. Creep strain values of 1-5% have been applied; 1% constitutes a characteristic design value.

The investigations were carried out on ODS alloy MA956 at 600°C for times up to 2000 h. Whereas at 1% there was no indication of accelerated attack, acceleration of corrosion seems to result for a higher deformation, figure above.
Thus enhancement of attack was restricted to strains which exceed the uniform elongation.



Above: *Internal and external corrosion of MA956 after testing in an S-O-C atmosphere at 600°C*

Performance Improvement of Ceramics, CMC's and Alloys

Alloys

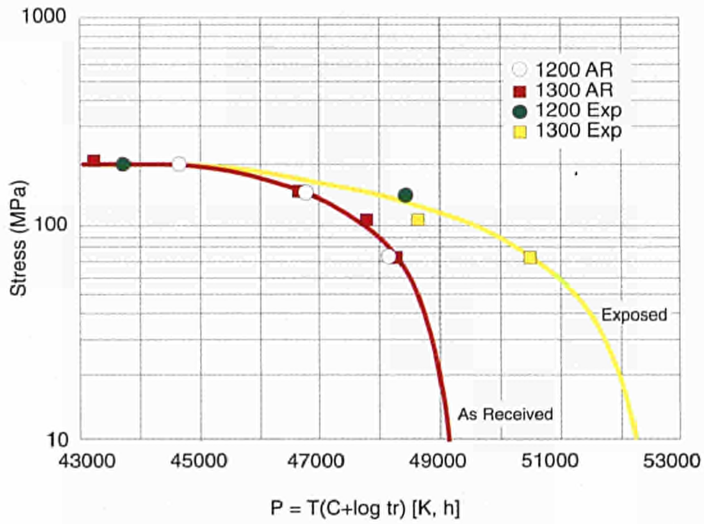
The potential of the ODS alloy MA 760 for application in uncooled vanes of stationary gas turbines, was investigated in the context of COST 501. The assessment of the damage and failure modes in LCF and TMF tested ODS alloy MA760, completed the performance evaluation of this material.
Simultaneously, the network grouping 29 research institutes, universities and industrial partners in eight EU and EFTA countries, collaborating in the framework of COST 501-round II/WP5 under the leadership of the Institute for Advanced Materials, concluded its research activity on the prediction of lifetime under simulated service loads. The associated final report will be issued early 1994.

Ceramics

One aim of the work on monolithic ceramics is the determination of the synergistic effects of sulphidation and mechanical loading at high temperatures on a hot pressed silicon nitride in a simulated coal gasification environment.
Pre-exposure followed by high temperature loading resulted in a considerable improvement of the long term creep properties under uniaxial loading (figure on page 11 above). In practice, however, the ceramic has to withstand chemical attack and mechanical loading simultaneously. A more realistic simulation thus requires performing long-term uniaxial tests in which the gas can react with the bulk of the material by ingress through cracks which

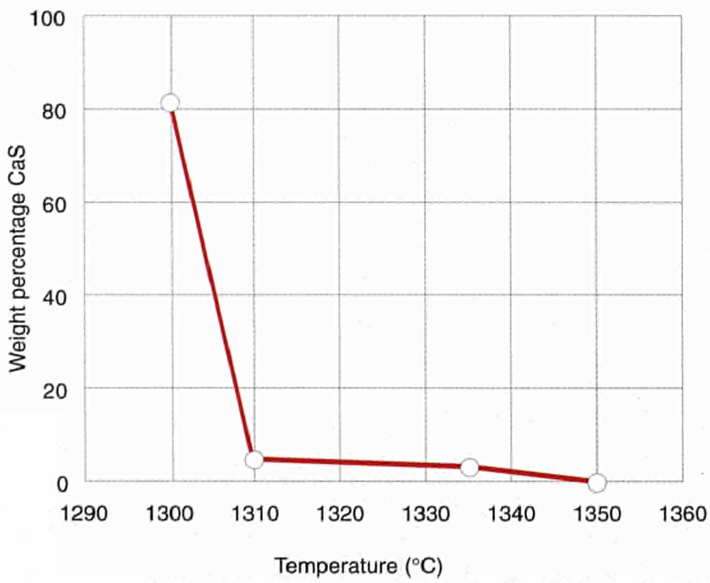
may form as a consequence of mechanical loading. An experimental set-up to perform such in-situ uniaxial testing under simulated environments was built and tested. Particular attention was given to determining the corrosive conditions experienced by the test specimen, particularly with respect to the chemical potentials of sulphur and oxygen.
To check whether the calculated partial pressures were actually established, calcium oxide, which exhibits a compositional change under the anticipated test conditions, was mounted at the position of the specimen. At temperatures below 1325^{0}C, thermodynamics predict that for the used gas composition CaO should transform completely into CaS, whereas at higher temperatures it should remain stable. The experimental results are plotted in figure below. They indicate that CaO remained stable to slightly lower temperature than theoretically predicted. In terms of the partial pressures of p_{O_2} and p_{S_2}, the experimental values are found to lie within 1 % of the theoretical values.
At the moment, the first in-situ creep tests are being performed.

A second, and more generic aim of the work is to investigate the existence and the conditions for the occurrence of a cyclic mechanical fatigue effect in monolithic ceramics. On the basis of a literature review, a classification of cyclic fatigue mechanisms has been proposed.
Experimental work during the reporting period covered the design and construction of a reversed bending jig which allows through-zero flexural fatigue at high temperatures. The design has gone through a number of iterations, and the performance of the final version is currently being assessed.



Above: *Larson-Miller representation of creep rupture data for as-received and pre-exposed material*

Below: *Experimentally observed weight percent of CaS after exposure to the anticipated simulated coal combustion environment*



Research Area: Reliability and Life Extension

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

Advanced materials can only be used if their behaviour under operational conditions can be predicted correctly. In order to achieve correct predictions, it is necessary to study experimentally the behaviour under well defined conditions, to observe the defects which form under these conditions and to develop models which can reliably predict the behaviour for conditions beyond those studied experimentally.

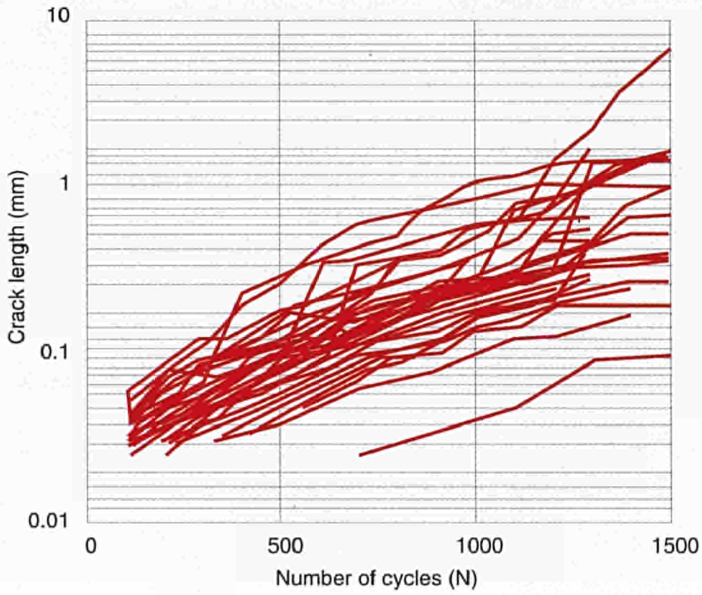
The studies in this research area all contribute to this objective. A major common feature, in most of the work is the fatigue behaviour and possible influences of specialised surface treatments on the fatigue life of components.
The changes in microstructure, the development of internal stresses and the growth of defects have been examined for different materials.
The work is also closely linked to other Institute projects. For example, thermal fatigue is an important life determining aspect of the behaviour of materials for the first wall of a thermonuclear fusion device. Fibre reinforced ceramics are candidate materials.

The problem of the ductile-brittle transition of bcc alloys needs to be addressed in the two major areas of nuclear energy: in pressure vessels for fission reactors and for advanced materials in fusion. The study of crack propagation in surface coatings is intimately linked to IAM's work on surface engineering.

Structural Performance of Advanced Materials

The demand for economy of operation of modern gas turbines has boosted the burner gas entry temperatures and the compression ratios to ever increasing levels in order to improve their thermal efficiency. The trend is to further reduce the operational costs by increasing both the component lifetimes and their reliability. The project contributes to achieving these goals by experimentally evaluating the mechanical performance of advanced materials for hot path components in gas turbines, and by formulating and validating performance prediction models. Two classes of materials are studied. Coated single crystal nickel based superalloys find increasing application in blades of the high and intermediate pressure stages of aero-gas turbines, and are being considered for application in land based turbines.

Continuous fibre reinforced ceramic matrix materials (CMC's) on the other hand have potential for application in specific gas turbine components.



Above: *Evolution of crack lengths with cycle number of all the microcracks detected on a thermo-mechanical fatigue tested specimen of a single crystal nickel-based alloy*

Single crystal nickel based superalloys

T(hermo)-M(echanical) F(atigue) testing of various combinations of bare and coated single crystal alloys is used, in the framework of third party contracts, to mimic the in-service behaviour of critical blade volume elements.

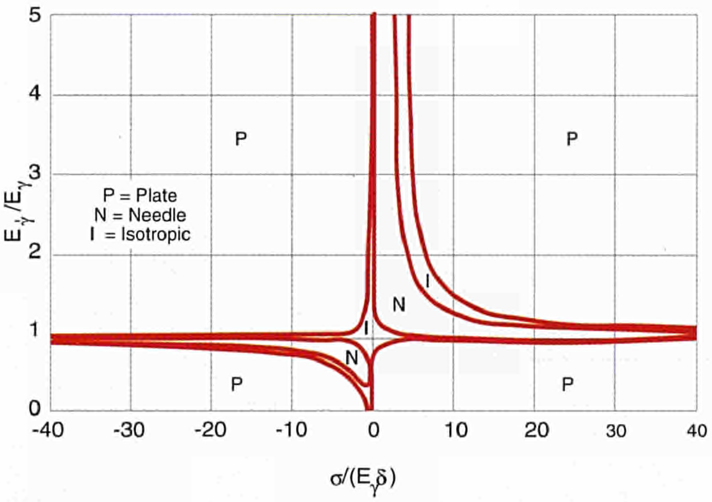
One of the substrate-coating combinations i.e. single crystal SRR99 coated with a high activity aluminide, has been singled out for an in-depth study, in the framework of the JRC Specific Programme, of a range of processes which are anticipated to influence the lifetime and the performance of the substrate-coating composite.

The evolution under TMF loading of the dislocation and precipitate microstructures in the substrate, the degradation of the coating, the initiation and the growth of microcracks and the residual stress state in the coating and sub-coating zones are the major processes of interest.

Experimental techniques for evaluating these processes were implemented in 1993, and first results became available. A mechanical testing machine was converted to TMF testing, the thin foil preparation technique for TEM specimens was optimised and an X-ray diffraction unit dedicated to the measurement of lattice misfit and residual stresses was installed and commissioned. In collaboration with ENSAM (Paris), special software for the analysis and fitting of X-ray diffraction peaks was implemented. The initiation and the growth of microcracks on sample surfaces during TMF was assessed in-situ for a first, limited set of testing parameters.

A vibratory polishing technique for the controlled removal of thin layers of TMF tested samples was employed in order to enable the aspect ratio of these microcracks to be determined. Specific conclusions with respect to the mechanics of crack initiation and crack growth under in-service blade conditions can already be drawn from the microcrack data base derived from measurements on the specimen surface. An example of the evolution of the length of each individual crack observed on a single specimen is shown in figure above.

Investigations concerning the microstructural changes triggered by TMF loading focused on the rafting of the strengthening precipitates. The modelling of the mechanical performance and of the lifetime of the coated blade material is based on an analysis of the mechanisms of microstructural



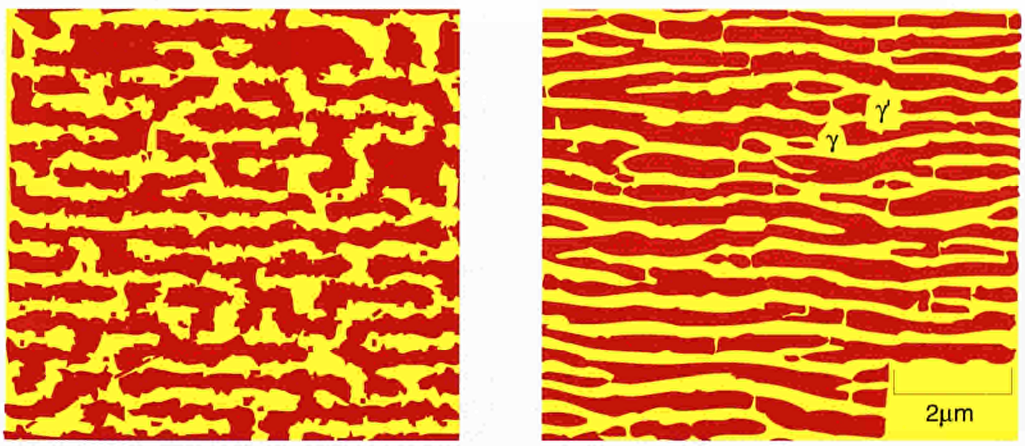
degradation and on the mechanics of microcrack initiation and growth. The first step set on the road of modeling concerns the development, in 1993, of a new criterion for the prediction of precipitate rafting in superalloys, based on the anisotropy in the interfacial dislocation energies.
In combination with Monte Carlo simulations, the criterion was used to model the microstructural evolution of single crystals at elevated temperature, with and without an applied load. The results compare well with experimental data.

Figure above shows a map predicting precipitate particle shapes as a function of the ratio of E moduli of γ and γ', of the applied stress σ and of the lattice-parameter misfit between γ and γ' together with a Monte Carlo simulation of the resulting γ/γ' microstructure.

a) Prediction of precipitate rafting in single crystal nickel-base alloys (P=plate, N=needle, I=isotropic)

b) Simulation of rafting (left) compared to actual rafting observed in single crystal nickel-base superalloy (right)

Above: *Model prediction of rafting of precipitates in single crystal nickel superalloy*



Continuous fibre reinforced ceramics matrix composites

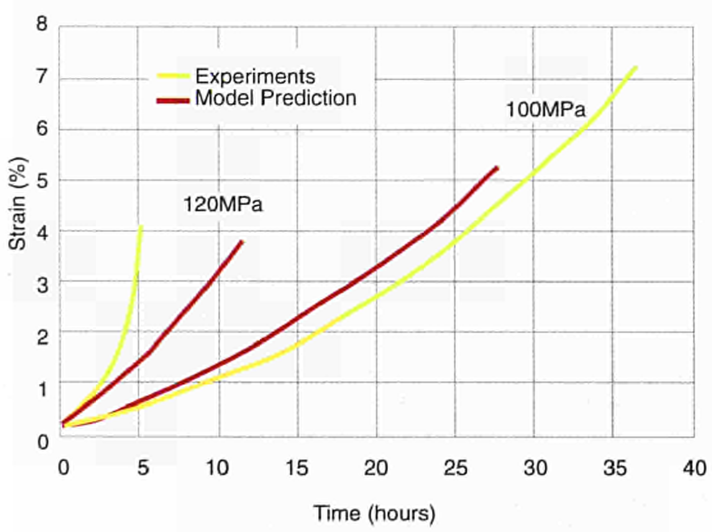
The research projects focus on the long term high temperature creep and fatigue behaviour of 2D reinforced materials. The emphasis lies on the study of the intrinsic material behaviour, i.e. without the disturbing effects of environmental factors.
Uniaxial tests are therefore performed under high vacuum conditions, thus avoiding chemical and/or physical changes of the fibre-matrix interfaces. In the creep project on an Al_2O_3(f)/SiC composite, the previously generated data base reference has been extended by a number of tests including periodic unloading and reloading cycles. These cycles give rise to a hysteris behaviour.
Detailed analysis of the loops provides valuable clues with respect to the interpretation of the damage mechanisms occurring under sustained loading at high temperature.
The evolution of the average elastic modulus as a function of accumulated creep strain indicates that progressive debonding of the fibre-matrix interfaces takes place during creep.

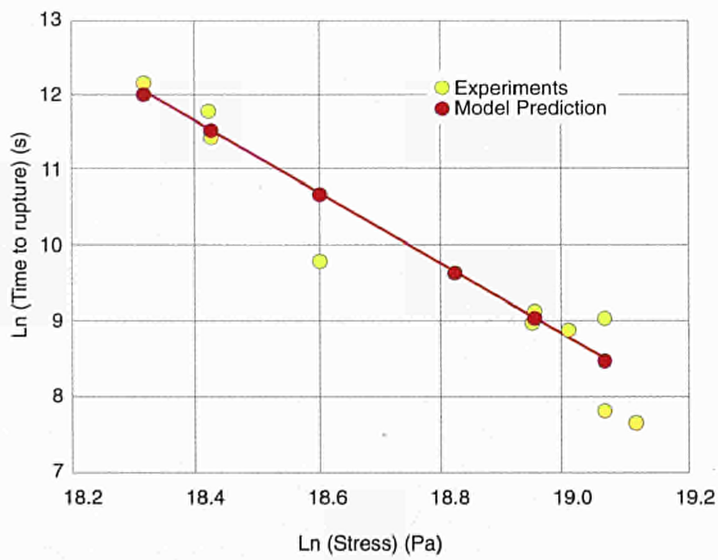
This causes the creep rate of the composite to increase, although the fibres themselves are creeping stationary. This explanation of the composite tertiary creep has been successfully incorporated in a model, which predicts the stress and time dependence of tertiary creep accurately (figure below left).

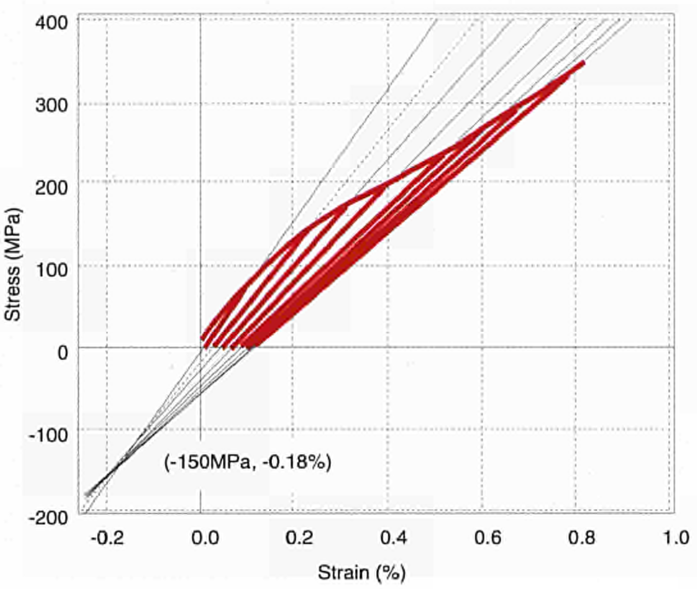
The model also includes a probabilistic analysis of fibre failure, which also makes it possible to predict the creep rupture life of the composite as a function of applied stress (figure below right).

Below left: *Comparison of experimental and model predicted creep curves of Al_2O_3/SiC composite at 1100ºC*

Below right: *Rupture life as a function of creep stress at 1100ºC: comparison between experiments and model prediction*







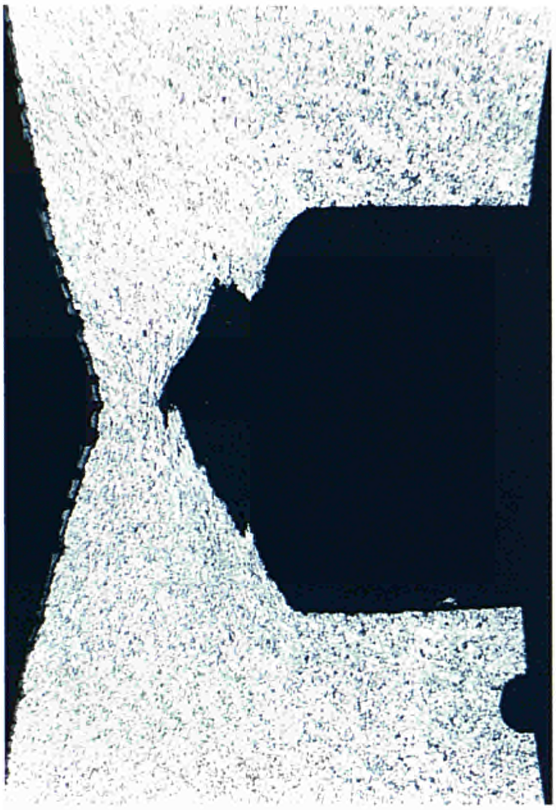
In the fatigue project on a 2D reinforced C(f)/SiC composite, room temperature stress controlled tests have been performed over a range of stresses and frequencies during the reporting period.
In order to define the stress amplitude for the tension-tension fatigue tests, the tensile behaviour at room temperature has been documented. Intermittent unloading-reloading cycles reveal a slight hysteresis, accompanied by a residual strain which saturates when the load is taken up by the fibres. It is worthwhile noting that the best fits to the unloading-reloading cycles have a common intersection point, which can be related to the presence of residual stresses in the as-processed composite (figure above).

Above: *Tensile test at room temperature on C(f)SiC composite*

Component Integrity Testing and Evaluation

In order to study the phenomenon of hydrogen attack in petrochemical plant steels, a major upgrade of experimental facilities to handle the explosive gas at high pressures and temperatures had first to be completed. Subsequently, a series of internal pressure creep tests using hydrogen as the pressurising medium was carried out on 2¼ Cr1Mo ferritic steel tubes containing either circumferential or longitudinal internal starter defects.
In addition, to support the component tests, a novel experimental facility was constructed which allowed conventional creep crack growth experiments to be carried out on compact tension (CT) test pieces in high pressure hydrogen environments.
A reduction in ductility and loss of rupture strength of the CT specimens was found to provide evidence of hydrogen attack. The effect was found to be even more marked for the component tests, where metallography revealed a dramatic change in failure mechanism as a result of the hydrogen attack (figure on page 19 above).

The enhancement of hydrogen attack caused by stress is illustrated by the increased development of damage at the notch tip where the stress is the highest. The influence of hydrogen on crack growth rates was evaluated in comparison with inert environment testing. Interpretation, using the C* method leads to some difficulties due to the high ductility of this alloy in the component tests in inert environments, but good correlation of the crack growth rates in the less ductile hydrogen-attacked state has been found.
A further activity contributes towards the objective of a COST 501 project which is to construct a high temperature heat exchanger capable of operating up to temperatures of 1200°C. In order to achieve these high temperatures, a ferritic oxide-dispersion-strengthened (ODS) alloy has been selected. The marked anisotropy of this material requires that an assessment of multiaxial creep resistance be made before the material can be considered in the design. Two batches of the alloy MA 956 have been studied in this respect using both internal

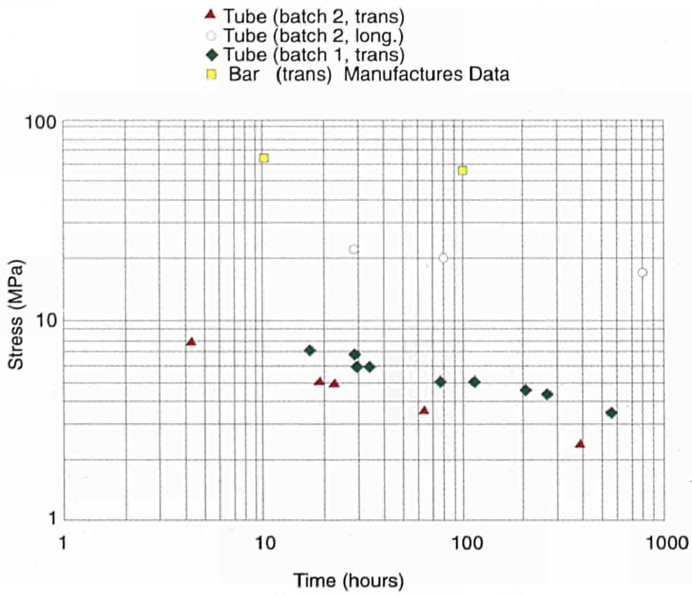


a) argon

b) hydrogen

Above: *Influence of pressurizing environment on the crack growth morphology of a notched component*

Below: *Stress rupture results for tube and bar MA956 at 1100ºC*



pressure and axially loaded creep tests on tubular components of dimensions representative of the heat exchanger.

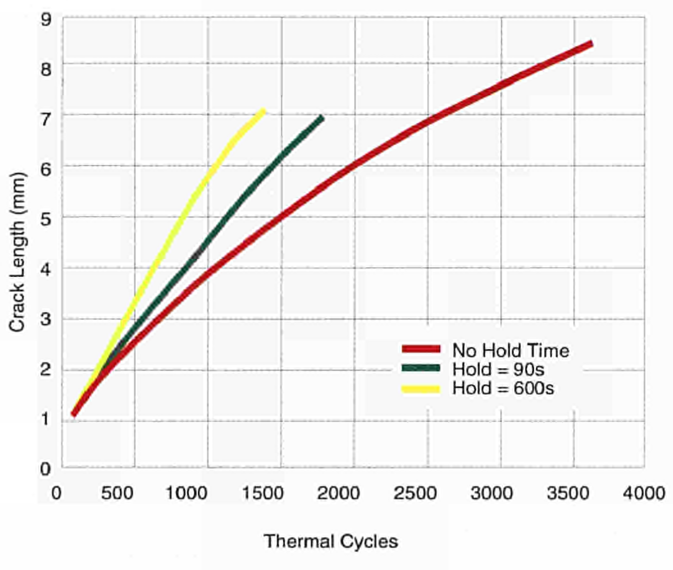
Figure below shows that the creep strength in the hoop direction, which governs the design, is reduced by a factor of approximately three from that measured in the longitudinal direction. Furthermore, the creep rupture lifetimes are considerably shorter than the manufacturers published data.

Under internal pressure, the tubes fail through crack initiation in the longitudinal direction at the inner surface, thus propagating in an intergranular mode through to the external surface. As this limitation in strength may be further exacerbated through sensitivity to defect, an additional programme of experiments has been devised. Initial results confirm that MA 956 tubes are not particularly sensitive to circumferential defects as would be expected; longitudinal defects will be studied at the earliest opportunity.

Thermal Fatigue of Components

The project aims at the numerical modelling of the life spent in propagating a crack to failure in components exposed to cyclic thermal gradient fields, and at the experimental verification of the model. Because of its widespread technological use, the tubular geometry has been selected for the purpose of modelling and experimental verification.
An experimental facility for the testing of tubes in a cyclic thermal gradient field has been completed previously. As a first application, the first wall of a first generation tokamak fusion reactor has been selected. In order to simulate the conditions seen by the first wall, the project also includes a nuclear aspect, for which an in-pile rig for the in-situ measurement of crack growth in the HFR has been designed.

Below: *Effect of hold time on the thermal fatigue fracture behaviour of 1 mm notched, 14 mm wall 316L components*



The development of the numerical model and of the associated computer code PREDI-N, and the preliminary experimental programme in the out-of-pile thermal cyclic testing rig have been completed in 1992. PREDI-N is essentially a code for linear elastic applications.
For elasto-plastic and creep loadings, a general purpose finite element code focussing on fracture mechanics aspects is in the process of being implemented on a personal computer. This FE code is an in-house development, offering the advantage that the source can be easily tuned to specific needs. In contrast to commercially available codes, the dedicated FE code is cost efficient both in terms of required hardware and run time. With respect to the in-pile rig, heaters for the control of the tube temperature have been incorporated in the design.
A new probe for the AC potential drop measurement of crack growth has been designed and constructed. Tests performed on components at IAM Ispra have given good results. Manpower shortage inhibited progress with respect to the construction of the in-pile rig.
Adaptation of the thermal fatigue testing facility to examine the influence of creep during the thermal cycle is realised through the development of a more sophisticated temperature based control system which allows for hold times at the maximum temperature of the cycle. In experiments where the external surface of the thick walled 316L stainless steel cylinder is cycled between 120 and 600^{0}C, higher crack growth rates for external longitudinal starter notches were obtained with increasing hold time (figure below). This is somewhat surprising, as analysis of the thermal loading using the Abaqus finite element package reveals that on reaching the maximum temperature, the external stresses are compressive, although they relax early in the hold time. As a result, a decision has been taken to modify the test equipment to allow the provision of an additional tensile creep load during the hold time to enable the study of creep/thermal fatigue interaction. In the Round Robin verification testing programme for the Internal Pressure Testing Code of Practice, the three internal pressure tests on 9Cr1Mo tubes, foreseen in Phase 1, have been successfully completed. A further six tests on the same material are planned in Phase 2 in which the influence on rupture life of hoop/axial stress ration in the tube wall will be evaluated.

Performance Improvement of Composite Materials

The investigations in this project are aimed at the development of methods to identify and to describe theoretically the principal deformation mechanisms and the lifetime determining defect state in metals. In particular, the effect of the microstructure on the macroscopic mechanical behaviour is discussed whith special emphasis being laid on materials with a designed microstructure. In principal, the activities started in 1992 were continued and further developed. A new subject dealing with the ductile to brittle transition has been taken up with regard to future contributions to the fusion programme and the AMES network.

Fatigue of Laser Surface Treated Materials

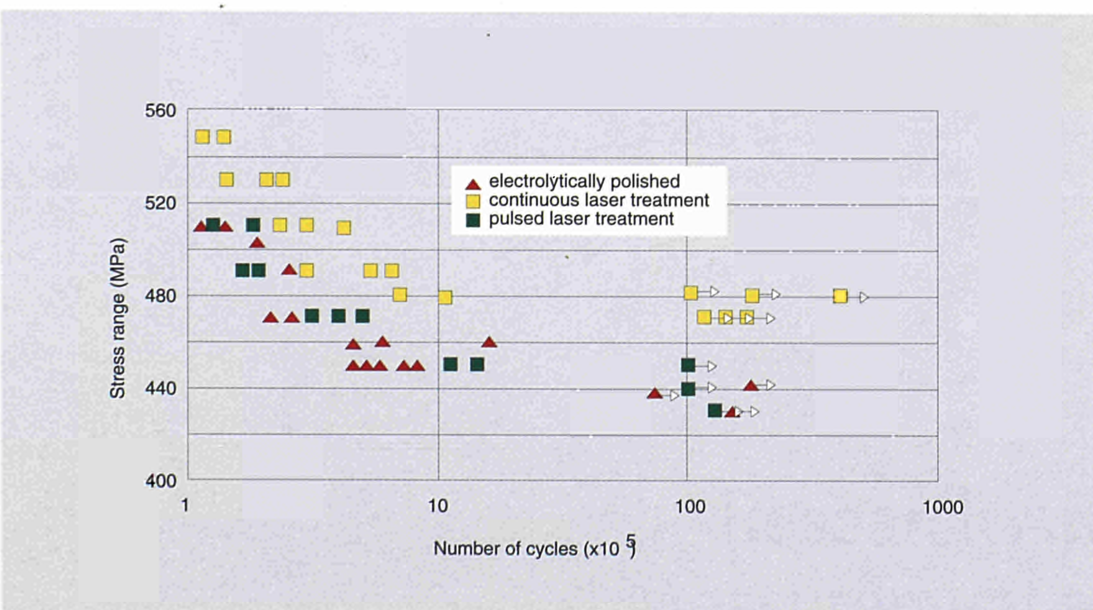
The effect of laser surface remelting on the high cycle fatigue properties has been further investigated. During surface remelting a fine grained surface layer is formed by exploiting the effect of high solidification rates due to self quenching.
Fatigue experiments were performed using flat specimens of 2 mm thickness.

Below: *Fatigue behaviour of laser surface treated 316L stainless steel. R = maximum stress/minimum stress = 0.01*

The surface treatment included a pickling pretreatment in order to improve the absorptivity and a subsequent treatment with a high power CO_2 laser. Two types of laser beams, a continuous and a pulsed one, were used to melt the material in a spot with a diameter of about 2 mm. Laser tracks in the longitudinal direction of the specimen i.e. parallel to the load axis with different degrees of overlapping were generated. The fatigue limit was defined as the stress level where half of the specimens reached 10^7 cycles without rupture.

As shown in figure below an improved fatigue resistance could be obtained with surface remelting by a pulsed laser beam. Using a continuous beam, there was still a significant improvement with respect to the as-received conditions which was comparable to the one achieved by polishing (see figure below). The microstructural investigations showed a fine grained surface layer with a thickness of 40-200 µm depending on the laser surface treatment conditions.

The cracks which cause the final failure seem to nucleate at small inclusions appearing at the surface after remelting. The increased surface roughness of the laser treated specimens is of minor importance, since the electrolytically polished specimens with a smoother surface exhibited a lower fatigue limit.



Metallographic Characterisation of the grain morphology and grain boundary damage

The quantitative assessment of grain boundary damage in the form of pores or microcracks from metallographic sections has been automated. This includes processing of images in order to elaborate the relevant information. In addition, programmes for determining the average properties characterising the grain shape have been developed which allow the comparison of grain structures with mathematical models for spatial tessellations.
Based on these results it is possible to derive damage parameters which can be used in constitutive laws appropriate for lifetime predictions.

Creep

The creep tests in MA 6000 at 750°C and 950°C have been completed. In addition a series of tests at 1050°C has been performed in collaboration with CNR-ITM. Metallographic inspection revealed a very pronounced tendency towards intergranular pore growth.
Preferred nucleation sites appear to initiate on the short transverse boundaries of the columnar grains, i.e., on the boundaries inclined perpendicularly to the stress axis or to the extrusion direction. Unrecrystallised regions evident in the form of fine grains are more susceptible to grain boundary damage.

Ultrasonic velocity measurements exhibit a rather large scatter and are inconclusive with respect to their potential for damage detection in this material. By performing creep damage measurements in specimens wich are uniform along the gauge length, a rather inhomogeneous damage distribution with a peak towards the specimen centre is frequently observed. This indicates an instability in the damage evolution in the later stage of the specimen lifetime. A new general stability criterion which is based on the temporal evolution of fluctuations with respect to the characteristic length scale of the evolving microstructure has been developed. The predicted critical damage values were in good agreement with those measured close to the rupture surface.

Ductile to brittle transition

The ductile to brittle transition as observed in many body-centred-cubic materials has been described by an approach based on dislocation dynamics. The synergistic effect of dislocation generation at the moving crack tip and the plastic deformation in the crack tip vicinity has been studied.

The stability investigation of the coherent propagation of a cleaving crack and its plastic zone leads to an expression for the ductile to brittle transition temperature and a prediction of the fracture toughness.

Research Area: Measurement and Validation Methodologies

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

The research area "Measurement and Validation Methodologies" is this Institute's principal area of activity which has specific standards related and prenormative research objectives in the fields of measurement, testing and characterisation. The institute has made strong efforts to develop a position of support to the increasing interaction between European R&D and standardisation which manifests itself in the setting up of a working group on "Standardisation and R&D Links", STAR, by the European Standardisation Committee, CEN, and in the enhanced scope of prenormative research objectives included in the 4th RTD Framework Programme. Measurement and validation methodologies for advanced materials are the core of a significant contribution that the Institute makes to this development. To categorise the standards related activities of this research area, several levels should be distinguished which support each other naturally and which constitute a joint structure for effective contributions to standards research:

- standards activities involving co-operation with or membership in technical committees of CEN and ISO.
- co-normative research providing support to ongoing standardisation by round robin testing and co-operation in international networks.
- pre-normative research addressing future standards objectives, new needs for standards development and standards activity to be initiated.
- development of reference methodologies which are in the particular focus of this research area.
- targeted fundamental research as the general research basis for these activities.

A research structure of this nature cannot be limited to one single area, and it should be understood that besides this core area, many other standards related activities are horizontally distributed throughout the Institute's programme. The number of projects addressing prenormative and standards objectives has significantly grown in 1993. They provide the basis for the development of a central position which the Institute should assume in the interaction between standardisation and R&D on advanced materials during the next multiannual programmes and which should make the transfer of research results to standards activities more efficient by the double concept of

- co-ordination of research focusing on the needs of the European standards bodies, and
- setting up European networks of excellence to address standards related research.

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 pressurized components (in particular vessels and piping) using Non Destructive Examination techniques (NDE).

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 improve the assessment of the capability and reliability of procedures for Non-Destructive Examination of structural components.

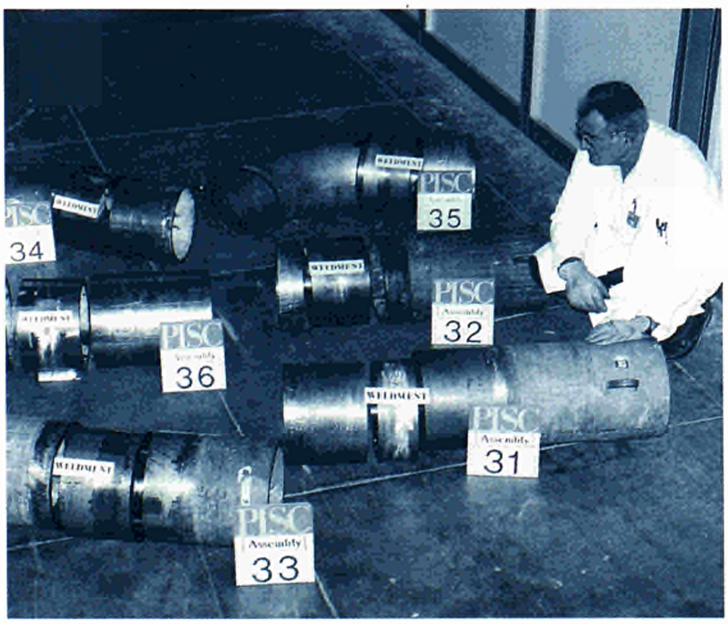
Three projects are centred on the Joint Research Centre which, in its roles of Operating Agent and Reference Laboratory, manages the programme and provides with participating EC countries approximately 60% of the programme funding; the other 40% come via contributions in kind from the non-EC participating countries. OECD/NEA provides the Secretariat of the PISC Managing Board, which consists of representatives of 14 countries (8 EC and 6 non-EC countries).
The programme is now at the very end of its third phase (PISC III); the activities concentrate on the evaluation of inspection results of pressure vessel structures containing service defects and of important structural components of the primary circuit made of different materials. Most of the PISC test assemblies and structural pieces were representative of (or came from) nuclear reactor components.

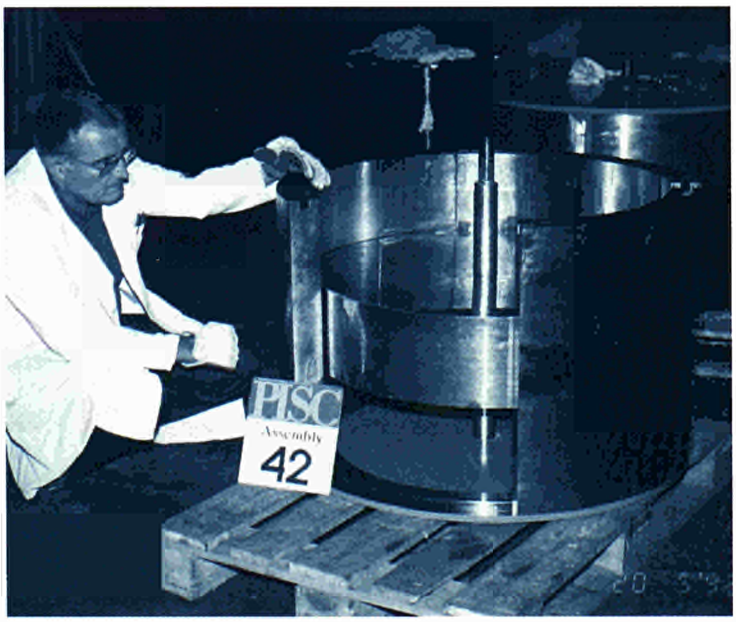
PISC III Programme Status

The Round Robin Tests are by now completed, destructive examinations are near to an end and the evaluation of results is conducted in parallel for most of the programme actions.
This evaluation will probably be continued by other programmes with different objectives: from NDE effectiveness evaluation to inspection procedure qualification or optimization. The present report appears thus as a closure of the PISC experimental work which generated a large data base now avai-

Below left: *PISC III assemblies 31 to 36 made of wrought stainless steel, Phase 1 of destructive examination: extraction of the weldments*

Below right: *PISC III assembly 42. Modular assembly simulating welded pipe sections of typical PWR primary circuit. Different materials and weld techniques are present in one assembly*





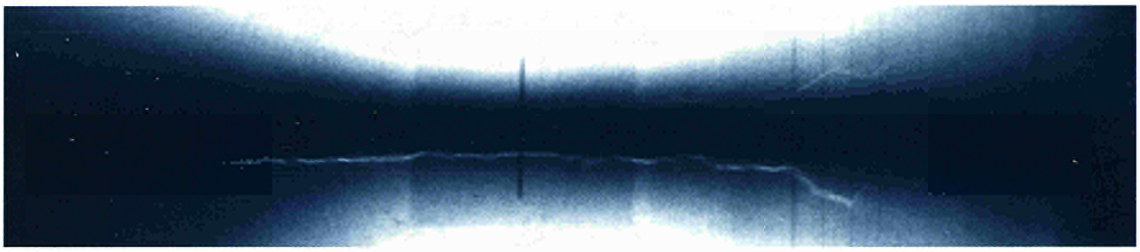
lable for various evaluations, trials and simulations. The objectives of Actions of PISC III which evaluated, their status of the evaluation of results in 1993, leading to the first conclusions as follows.

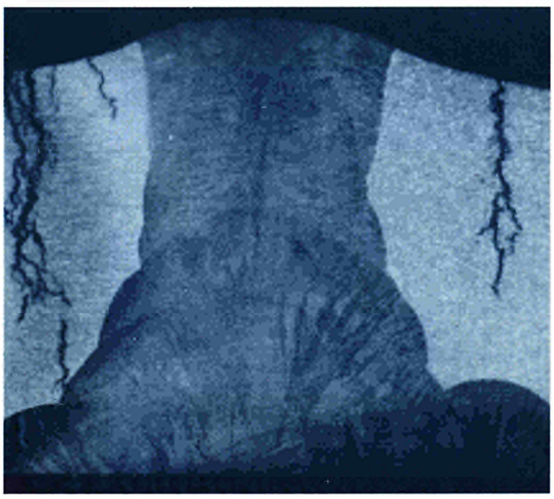
Action No. 4 (Austenitic Steel Testing) applied the PISC II methodology to the primary circuit piping of LWRs. Round robin tests for the capability assessment and parametric studies started in 1990. Twenty five teams have participated in one or more phases that extended up to September 1993.

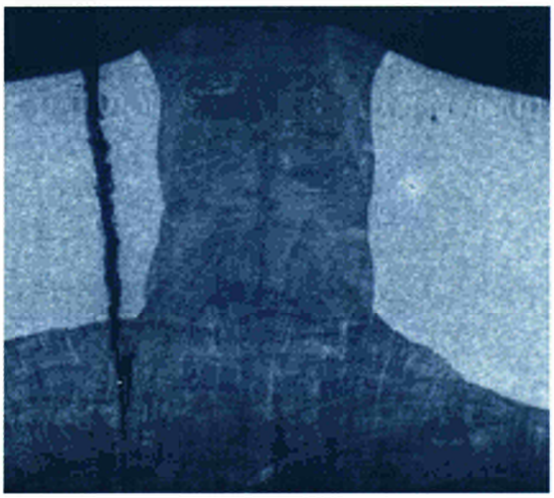
The Wrought-to-Wrought pipe sections RRT was the first one to be completed, the assemblies having been examined by 20 teams from 10 countries. These assemblies Nos. 31 to 36 were destructively examined in Japan (figure left on page 25).

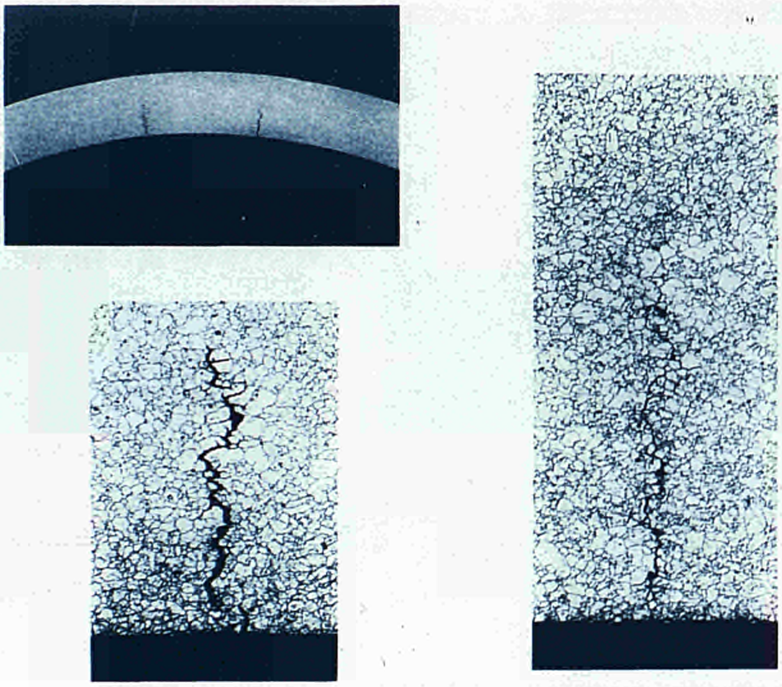
The Assembly 51 (Wrought-to-Cast) is being destructively examined by the JRC at Petten as are the Cast-to-Cast mock-ups (Assemblies 41, 42 and 43, figure right on page 25).
A reliability exercise has been performed in the USA and will be repeated in Europe in 1994 on pipe sections coming from nuclear plants but funded outside the PISC programme budget. Parametric study blocks have been made for the study of the influence of the metallurgical structure and of the flaw characteristics.

Below: *Typical large IGSCC in wrought to wrought steel welded pipe assemblies*









Above: *Steam generator tube wall with typical corrosion defects (PWSCC)*

The inspection of the assemblies made of wrought steel, provided good detection results of IGSCC's, fatigue cracks and notches. Branched IGSCC's and defects in the weld are difficult to detect (figure below on page 26). Sizing results were generally disappointing but four teams showed good sizing performance. Several team made several and severe false calls.

The major conclusion of this exercise is that the capability exists to inspect these assemblies but it is represented by 10 to 15 per cent of the teams only.

Clear conclusions are available as well on the negative effect of the counterbore on detection of defects and on possible mitigation measures.

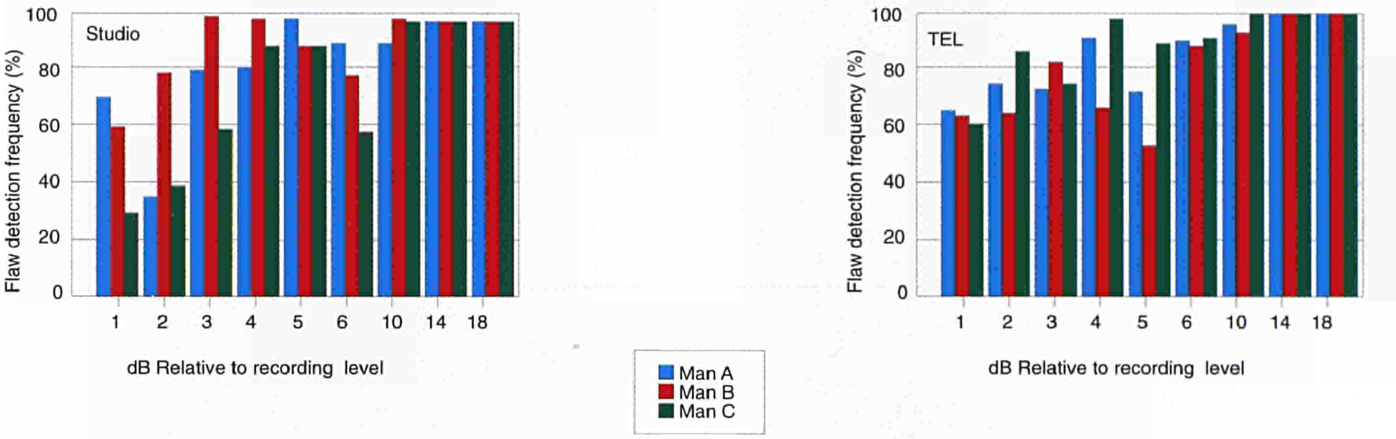
The effect of the structure of cast Austenitic steel is not as systematic as expected but some inspection techniques which are often used are shown not to be adaptable.

In several cases artificial notches of the type A correctly represent real defects, for the purpose of ultrasonic inspection.

Action No. 5 (Steam Generator Tubes Testing) involved round robin tests of individual tubes of steam generators containing realistic and artificial defects. About thirty teams from 11 countries participated in the RRT.

The validation of artificially introduced defects has involved mainly experts of France, Italy and Japan. The Reference Laboratory (JRC, IAM/NDE helped by ENEL/DCO) has prepared many artificial defects in tubes.

Orders have been placed by the Operating Agent to get realistic corrosion defects from CEA, MITSUBISHI, CEGB, KEMA. Emphasis has been put on corrosion defects (IGA, SWSCC, PWSCC,



Above: *Human factor studies; Manual inspection results of three inspectors: performance in detection of rejectable flaws as a function of the amplitude of reponse of the flaw in laboratory (studio) and industrial conditions (TEL)*

figure above on page 27). The RRT ended in September 1993.

The evaluation of results started in Paris in December 1991; the establishment of criteria and bench marks for this evaluation represented significant progress. Effective evaluation of inspection results is ongoing: first trends are that detection is always lower than 75 per cent. Worst and best results are obtained in turn by UT or ET. Combination of techniques (UT with ET) gives results in the upper range. A large dispersion is found in the correct sentencing of defects and the average performance seems to be of the PISC I standard as obtained in 1979 for the ultrasonic inspection.

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. The experimental studies involving 6 inspectors was completed by AEA Risley for PISC. These studies began with tests in a studio aimed at establishing the base-line capability of the inspectors under laboratory conditions. They were completed by studies in the environmental laboratory, TEL, of PISC to investigate the effect of working conditions and duration of shift/programme. The assessment of the working conditions and duration of shift/programme. The assessment of the results is available [9] which shows that these types of test could identify, illustrate 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, the ambient conditions (figure on page 29 above and below).

Action No. 8 (Support to Code and Standard (C/S) Organizations) gave direct support through the PISC group of experts to C/S organizations [10].

Action 8 closes with PISC III but has synthesized the legacy of PISC to networks or programmes for the implementation of inspection qualification.

Conclusions

Starting in 1974, 20 years ago, several studies of NDT effectiveness and reliability in the nuclear industry were initiated in a number of EC and OECD member states. One of the most important, extensive and thorough of these studies was PISC which has virtually become a watershed in the development of such work. Several literature surveys made by national institutions and international organizations have shown that the PISC conclusions are supported by other work.

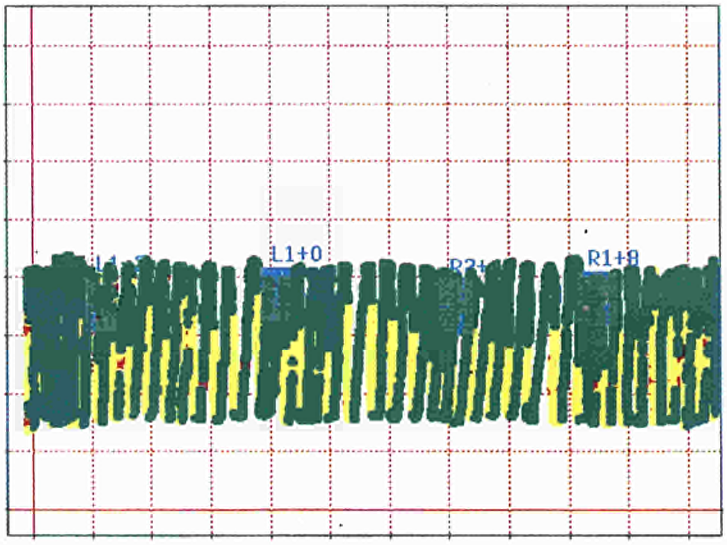
This programme showed that generally neither the effectiveness nor the reliability of inspection procedures were sufficiently evaluated in many cases of industrial applications.

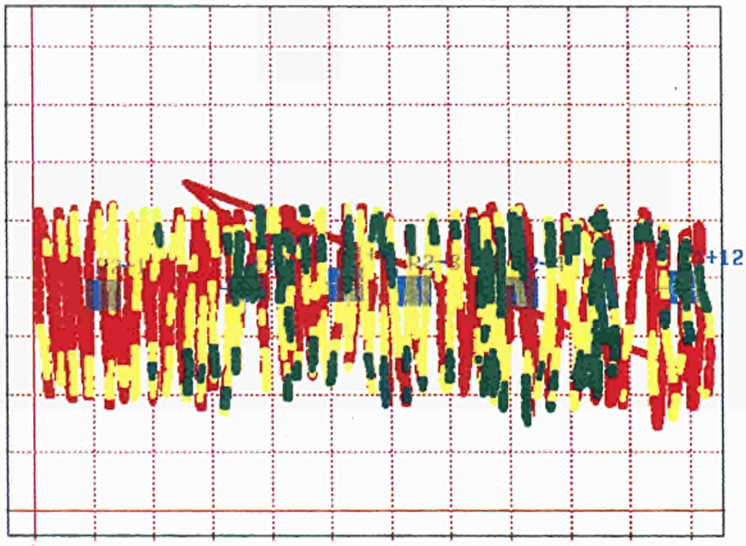
At present, it seems that inspection of many pressure components could be effective and reliable; it is probable that a definite conclusion could be drawn when particularly difficult inspection problems will be solved and the relevant inspection technique qualified.

It is thus fair to consider that Inspection will be able to play its role in the Structural Integrity assessment scheme and therefore in Pressurized Components Safety Assessment Inspection techniques are, or will soon be, able to furnish the information needed about the "Status of the structure" concerning the presence, location, size and characteristics of defects.

The effectiveness and reliability of these inspections will depend very much on the way in which they will be qualified and PISC III contributed significantly to the development of qualification schemes and in particular to the performance demonstration of inspection procedures.

The experimental work of PISC III is now at an end and with it the funding of the PISC programme. However, the evaluation and use of PISC data generated by the different Round Robin Tests will certainly continue for several years as well as the co-operative spirit which characterized these 20 years of the NDE effectiveness evaluation.





Above: *Manual scanning. Diference between correct and uncorrect coupling: green means good transmission of energy; yellow means unacceptable transmission; red means no transmission*

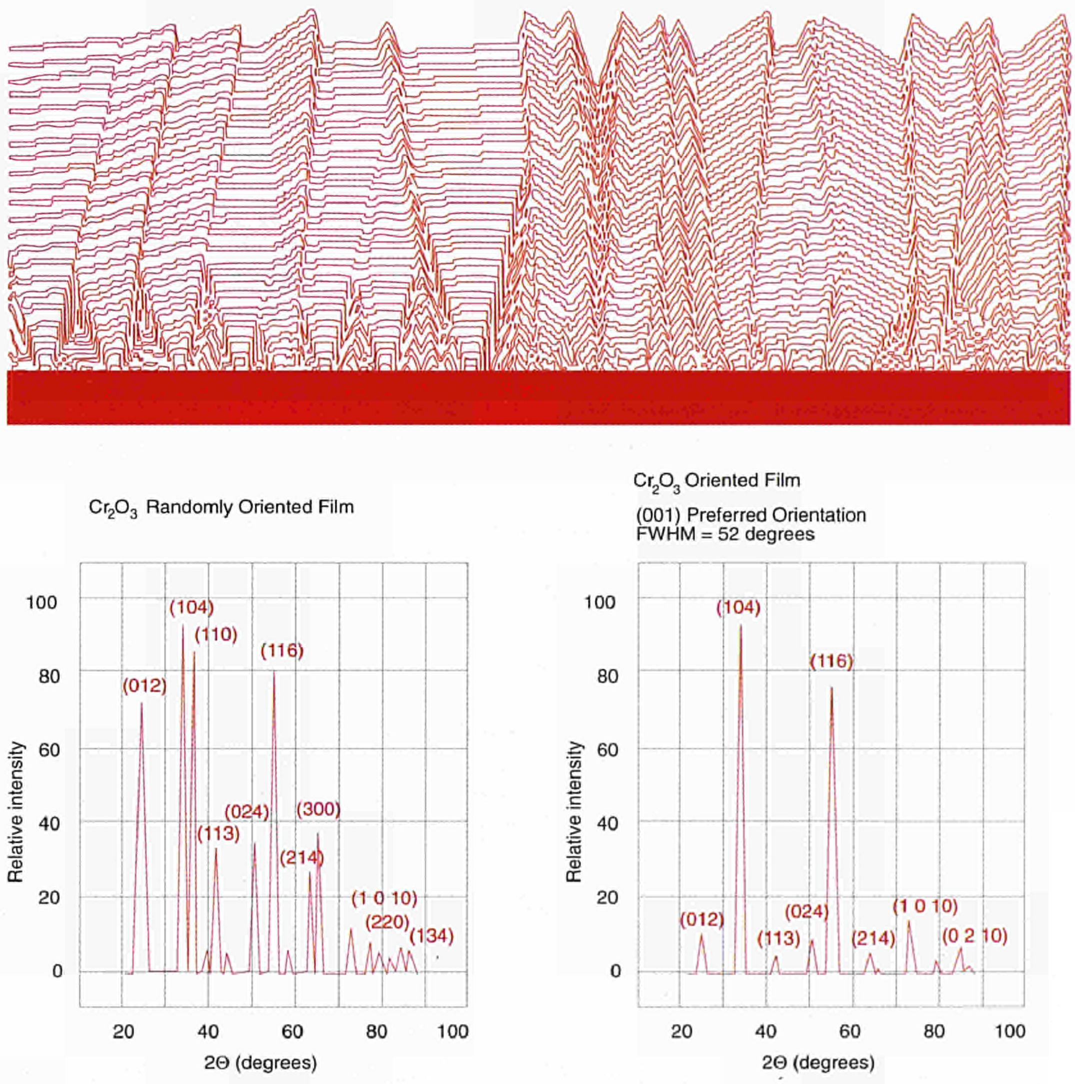
Physical Properties of Coatings

During 1993, new efforts were devoted to the development and improvement of methods for the identification and measurement of properties relevant to wear and corrosion resistant films and coatings. Main emphasis was given to the development and refinement of measurement methods which can be applied to very thin films such as nano-indentation for mechanical characterization, glancing angle x-ray techniques including diffraction, reflection and x-ray fluorescence for structural and microstructural characterization, high resolution electron spectroscopy for chemical characterization, and positron annihilation doppler spectroscopy for non-destructive film thickness measurement. Furthermore, the R&D as well as the service deposition activities in the Thin Film Laboratory were continued.

Nanoindentation and Thermal Drift

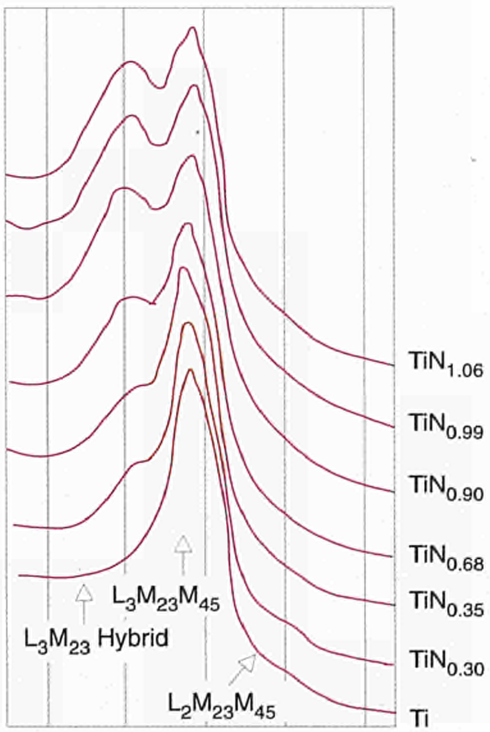
The highly sensitive method of ultra-low load, depth-sensing nanoindentation (installed in the Thin Film Laboratory of the IAM Ispra) is usually

Below: *Calculated picture of the crystal surface subjected to preferred orientation selection (above), and corresponding diffraction spectrum (below right) in comparison to a spectrum which would be obtained with randomly oriented crystallites*



applied to determine hardness and Young's modules, but it has also the potential to be extended to the measurement of other properties such as film adhesion, residual stress and time dependent plastic flow (creep).
In an attempt to apply the method to creep measurements, a load and time dependent thermal drift was observed. This type of drift (Joule heating drift) has not been recognized up to now either by the instruments manufacturer or by its users. The drift was found to be due to the Joule heating in the load coil during the indentation procedure. It was studied in detail and new recommendations for the measurement and data evaluation procedures were suggested. Presently, a method is being elaborated to compensate for the effect by the development of suitable codes and by hardware modifications.

Below: *$TiL_3M_{23}M_{45}$ direct mode high resolution raw spectra for various concentrations N from 0 to 1.06*



An active cooperation in the BCR project "Hardness (Mechanical Properties) Testing of Surfaces" (BCR Contract No MAT-CT92-0027) was initiated in 1993.

Glancing Angle X-Ray Techniques

Development work was focussed on the elaboration and testing of specialized software necessary for the interpretation of glancing angle XRD and reflectivity spectra. Three programs are now either completed or nearing completion: REX is a program which can be used for the interpretation of X-ray reflectivity data. A third option has been added to the possibilities for simulating the effects of surface and interface roughness.

FLUX is a program, developed over the past two years, which is used for deducing simulated curves of the intensity of X-ray fluorescence or diffraction from either films or substrates against angle of incidence. It has been found that a fairly accurate knowledge of the spectrum of the incident radiation is required if a reasonable accuracy is to be obtained.

FOX is the main new software development program of the past year. It is used to simulate glancing angle XRD spectra of surfaces with preferred crystallite orientation. The program can be applied to any crystalline system, and can also simulate the spectra of surfaces where the degree of preferred orientation varies with depth.

Figure on page 30 below illustrates schematically such a surface (generated with a 2-D crystal growth simulation program), together with the simulated diffraction spectrum.
For comparison the diffraction spectrum of a non-oriented surface of the same material is also presented in the diagram.

As well as these software developments, work has been continuing on the structural analysis of thin films produced by various groups of the IAM.
These include BN, TiBN and HfBN thin films, Ti implanted BN and Ti/BN multilayers, SnO_2 and Pt implanted SnO_2 gas sensors, chromia layers on silicon, Y implanted Cr and Ni-20Cr before and after oxidation (RefEXAFS analysis was also used for these studies), Nb implanted TiAl, and corroded SiC/SiC composites.

High Resolution Electron Spectroscopy

A new method based on high energy resolution Auger Electron Spectroscopy (AES) for the determination of the Ti:N ratio of TiN_x coatings has been elaborated. Titanium nitride coatings are presently the most widely used technical coatings (as decorative or wear and corrosion resistant coatings).
So far the analysis of TiN has not been performed with AES, because the main Auger peaks ($NKL_{23}L_{23}$ and $TiL_3M_{23}M_{23}$) are partially overlapping.
The new method is based on the analysis of the Ti $L_3M_{23}M_{45}$ valence band peak, which develops as a consequence of the bonding of Ti to N, a second peak, labelled L_3M_{23} hybrid peak, emerging at a 3.9 eV lower energy.
This peak grows with the grade of nitridation (figure on page 31 below).

Data evaluation reveals, after a simple Shirely background correction, a linear dependence between the ratio r of the $L_3M_{23}Hybrid/L_3M_{23}M_{45}$ peak height of the raw data and the N/Ti ratio ($r(\%) = 16 + 44.8^* N/Ti$). This relation can be used directly to evaluate the nitrogen content of an unknown TiN_x compound by recording only the Ti $L_3M_{23}M_{45}$ region.

Positron Annihilation Doppler Spectroscopy

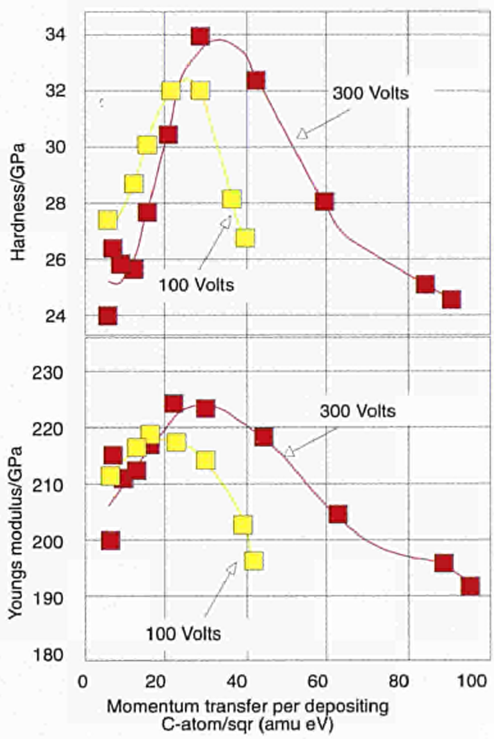
The measurement method has been further improved and extended. A study of the stopping law of positrons penetrating film and base material allowed the establishment of a rather general and accurate calibration procedure.
As a result, a simple function of the measured doppler parameter provides a linear measurement of film thickness from 5 to 50 mg/cm^2. With a modified calibration the sensitivity can be extended down to 1 mg/cm^2.
For the measurements of thicker films, up to 300 mg/cm^2, use of a more energetic positron source has been made, starting with the determination of the appropriate stopping laws.
Preliminary tests show that the method is suitable for mapping the film thickness over extended areas with a spatial resolution of the order of 1 mm.

The measurement speed has been increased by almost one order of magnitude by technical improvements. The technique is now suitable for practical applications.

Thin Film Deposition

The availability of thin film deposition techniques at the IAM-Ispra is indispensable as a service activity for the various characterization methods under investigation (see above) and for other IAM projects including surface modification by ion implantation and development of sensors.
However, thin film deposition technology is also a scientifically challenging discipline and requires a continuous development to maintain a high level of expertise.
The R&D activities of the Thin Film Laboratory Ispra were limited to the development of high strength boron nitride based transition metal coatings and on diamond-like carbon films.

Below: *Hardness and Young's Modulus of a-C films sputter deposited with different assisting argon ion intensities as function of the momentum transfer*



The development of ultra-high strength coatings based on titanium boron nitride was continued. A new sputter deposition technique using a heterogeneously composed Ti/BN target, the composition of which can be easily varied, was established. Coatings with hardness values up to 60 GPa were obtained.
These high values are caused mainly by nanometric grain sizes and by the occurrence of two Ti-B-N phases. Pin on disc and adhesion tests were performed indicating a good, but slightly lower performance when compared with TiN coatings.
Experiments to improve this behaviour are under way. An investigation to evaluate the effect of argon ion bombardment during growth of amorphous carbon on hardness H and Young's modulus Y was performed. The films were prepared and analyzed by a new procedure: the ion beam sputter deposited film was assisted by a focused argon ion beam.

This method produces a film with a high central zone and progressively decreasing zones of energy and momentum transfer.
By measuring H and Y in these different regions a large data set was obtained just from one film.

From the analysis of two films which were prepared at different assisting ion beam voltages of 100 and 300 Volts, respectively, it was found that H and Y display a uniform functional dependence only if plotted versus momentum transfer (figure on page 32 below).

Pre-standards 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, inclusive of the validation of these methods.
The deliverables serve as the pre-normative input to the subsequent development of new standards, or to the improvement or optimization of existing codes and standards in collaboration with European Standards Institutions.

Simultaneously, support in the form of participation to, or chairing of working groups involved in the drafting of standards and codes of practice is provided.

As an example of the latter type of activity, IAM Petten is currently providing the chairman and the secretary of a working party set up under the auspices of the High Temperature Mechanical Testing Committee, with a view of establishing a code of practice for the measurement of misalignment in static and dynamic uniaxial mechanical testing. The members of the working party are representatives of testing machine manufacturers and of research institutes.
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. Other testing activities with a pre-normative impact rank under the umbrella of Round Robin testing. In the reporting period we were involved in a Round Robin exercise in the framework of VAMAS on the measurement of the fracture toughness of monolithic ceramics at high temperature.

Application of NDE to Materials and Thin Coatings

Acoustic Microscopy

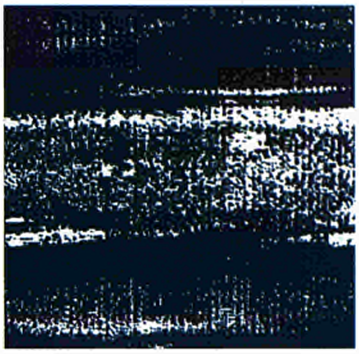
A common definition of the Acoustic Microscopy does not exist. In a very large range of frequency (from 1 MHz to 1 GHz), various ultrasonic techniques are used for the Non Destructive Inspection of small components (e.g. microelectronics, multi-layered materials,...) and for the measurement of some particular characteristics of advanced materials such as their microstructure (grain morphology and size), thin coatings thickness, . . .

The systems which are available on the market are generally specialized, following commercial criteria for their specifications. They are systems called "Acoustic Microscope" or "High Frequency Ultrasonic Scanning System" and "Low Frequency Acoustic Microscope" or "Ultrasonic Scanning system" but no one is suitable for the study of measurement methods where it is necessary to apply quite simultaneously various techniques using different propagation modes and different types of transducers.

In 1992, the Ultrasonic Development Laboratory undertook to develop a system in collaboration with the manufacturer Biosonic and the University of Valenciennes. The first prototype was delivered in July 1993 and a series of tests were performed in order to assess its performances.
Figure below shows an example of C-SCAN images on samples of MA 6000 without any surface etching. The grain morphology and size are clearly visible as some intergranular microcracks. Some tests on MeCrAlY coatings were performed as it is possible to see in the annual report concerning the NDE of Plasma Sprayed Coatings.

LT2 25 x 25 mm Step 0.1 mm

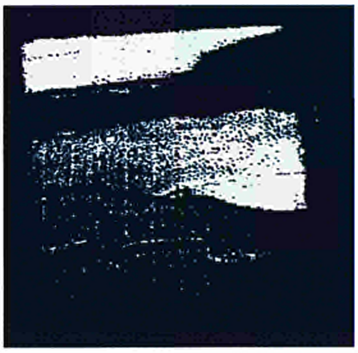
LT2 5 x 5 mm Step 0.02 mm



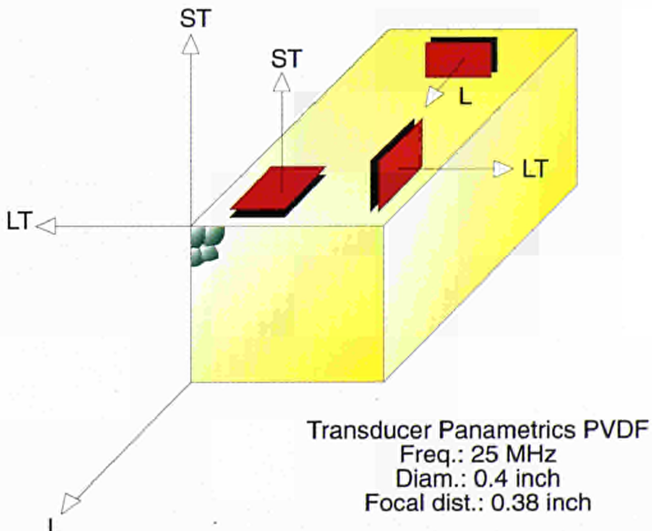
LT1 25 x 25 mm Step 0.1 mm

L2 25 x 25 mm Step 0.1 mm

ST 1 25 x 25 mm Step 0.1 mm



ST 2 25 x 25 mm Step 0.1 mm



Below: *Acoustic microscopy on MA 6000 samples*

Some modifications on hardware and software were requested to the manufacturer.
The second prototype has been delivered in January 1994 and is mainly used for the study of techniques for the inspection of metallic coatings. Some other applications are foreseen in the frame of both Work for Third Party and Shared Resources Actions.

Ultrasonic Techniques

Introduction

The purpose of the project is to develop and qualify non destructive techniques for characteristics measurements and for the inspection of metallic and ceramic coatings. The programme of work is mainly concerned with ultrasonic techniques that should enable the measurement of thickness, elastic constants and porosity of coated materials and the detection and sizing of defects such as bonding flaws and surface-breaking cracks. The activity has been initiated in 1993 by theoretical studies and experimental tests aimed to define the best course to be followed to attain our objective.
An important part of the work is performed in the frame of a third party contract with ENEL.SpA.

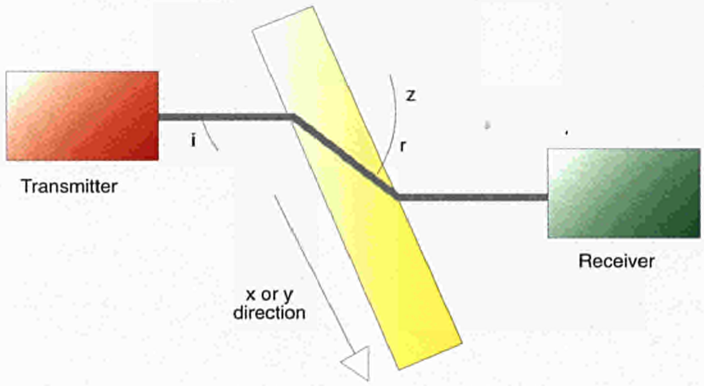
Experimental Work

Compression and Shear Waves Propagation Velocity

Before plasma spraying, the compression (CW) and shear waves (SW) velocities in substrate materials were measured as a function of the direction of propagation.

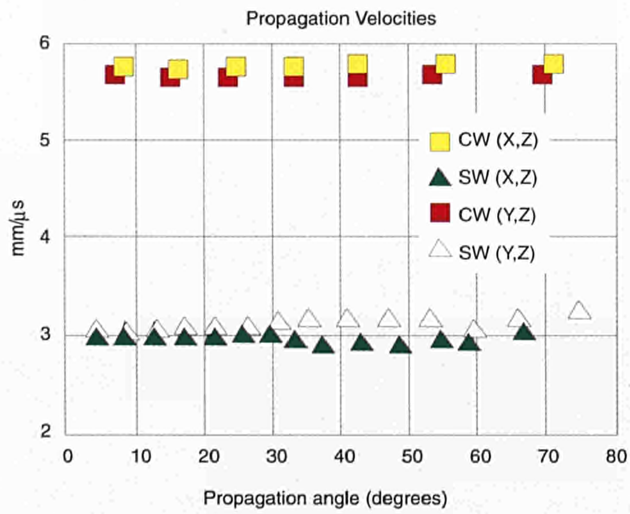
These measurements were performed in through mode (figure above). For each value of the incidence angle i, refraction angles and velocities were calculated from the difference between the corresponding time of flight and the one obtained in normal incidence.

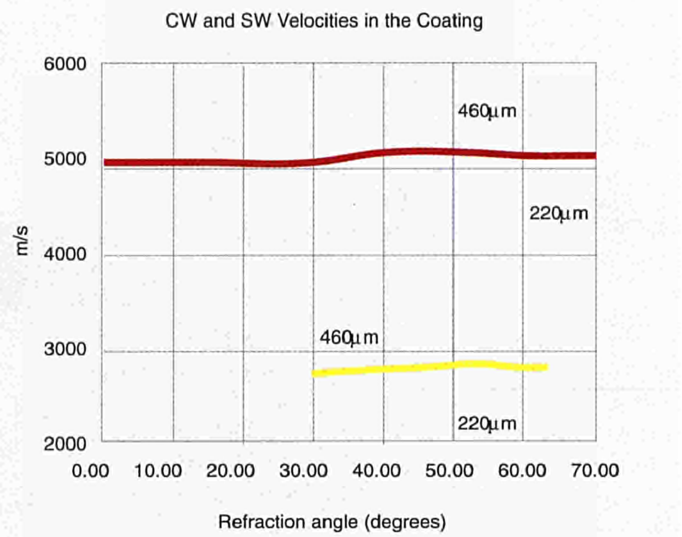
Figure below shows the case of an AISI 316 L substrate. The results were obtained with an accuracy of about 1%.
Propagation velocities measurements on IN 738 samples are presently in progress.
For measurements on disbonded MeCrAlY layers, the same method was used, completed by the frequency analysis of the transmitted signal.



Above: *Velocity measurements in through mode (x is the direction of lamination of the substrate material, y is the transverse direction)*

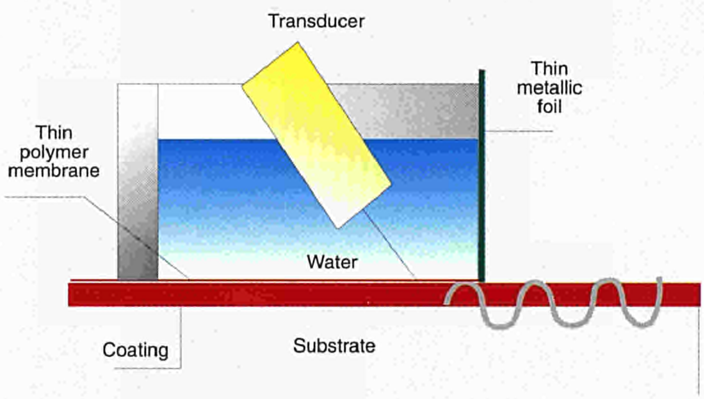
Below: *Propagation velocities in AISI 316 L*





Above: *Propagation velocities in a NiCoCrAlY coating*

Below: *Special transducer for Rayleigh waves*



The results are illustrated in figure above. The influence of the various sources of errors has been analysed:

- The main difficulty is due to the roughness. Using a simplified theory, mainly valid for long wavelengths, a rough sample behaves as a polished one having its mean thickness. It was verified by comparing the results before and after removing the roughness. Nevertheless, a theoretical work is developed in order to determine more exactly its effect.
- A second source of inaccuracy is the strong beam distortion which corresponds to large incidence angles. A computer program is being developed to calculate exactly this beam distortion.

Rayleigh waves propagation velocity

Rayleigh waves propagation velocity has been measured using a transducer especially manufactured for generating surface waves (figure below). The result of the measurement on AISI 316 L (2900 ± 10 m/s) is in good agreement with that calculated from CW and SW velocities.

Some interesting conclusions can be drawn from the results of measurements performed on coated samples using this transducer.

It is possible to predict that the minimum size of defects which can be detected by Rayleigh waves is about a few millimetres. More accurate modelling work is going on in order to improve the reliability of this prediction. In addition, when the experimental results concerning real defects will be available, the comparison with the theoretical prediction will reduce the probability of confusion with an experimental artefact.

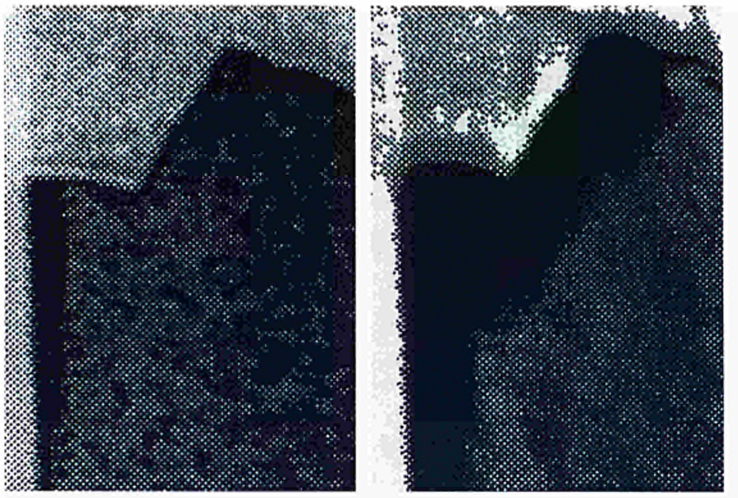
At high frequencies, the Rayleigh waves are completely contained in the coating and the velocity measurement is the most accurate. Anyway, the Poisson's ratio and information on anisotropy are required for a full characterisation of coating materials.

The results were generally obtained with a good reproducibility. Therefore, this method can be used, with reference samples, for accurate comparative measurements.

Anyway, the research of a good accuracy on absolute measurements presents an interest provided that the characteristics themselves are reproducible from one sprayed coating to another. Efforts on more accurate absolute measurements will be released after the experimental verification of the reproducibility of the characteristics by using relative measurements.

Tests on bonding flaws

Figure above illustrates an example of C-SCAN's on a bonding flaw using the Pulse Echo Technique. On the left hand side, the image acts as a reference. The presence of the bonding flaw is revealed by a strong diminution of the backwall echo amplitude, thus giving rise to an important contrast (the right hand side image).
The same sample will be used for testing new techniques using Rayleigh Waves, much more sensitive to the presence of such defects, and much more suitable for testing real components.

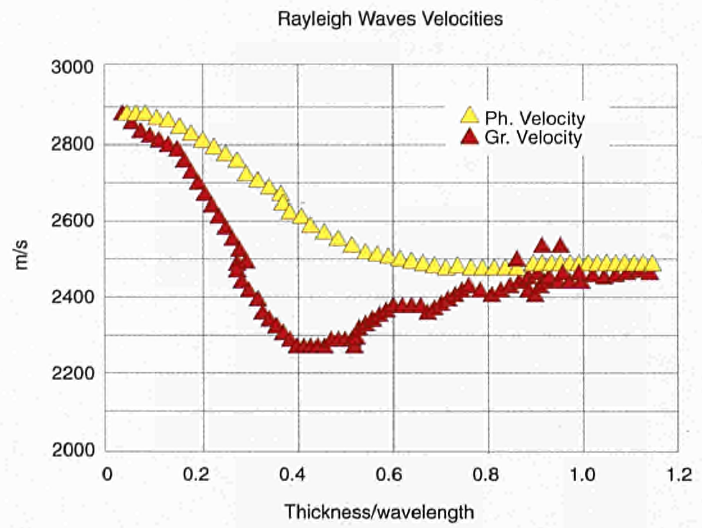


Above: *C-SCAN of a disbonded area (black on the right picture)*

Theoretical Study. Modelling

The theoretical study is used as a support to the experimental work. The actual characteristics of coating materials and substrate materials are the input to the computer codes that were developed for practical use. Figure below illustrates an application showing the group and phase velocities of Rayleigh waves completed by an experimental verification.
Such a result is very important. According to the literature review, this accuracy in the verification of the theory has never been achieved. Besides, it demonstrates that methods based on this theory can be applied successfully to the inspection of coated components.

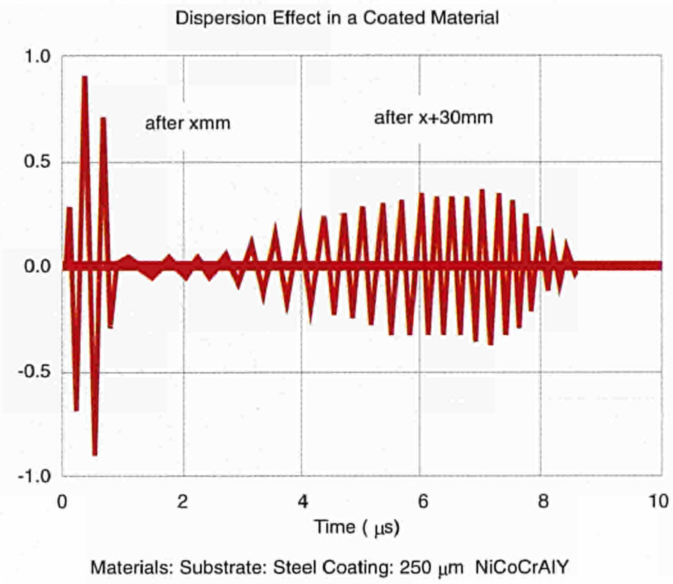
Below: *Dispersion curves*



Modelling

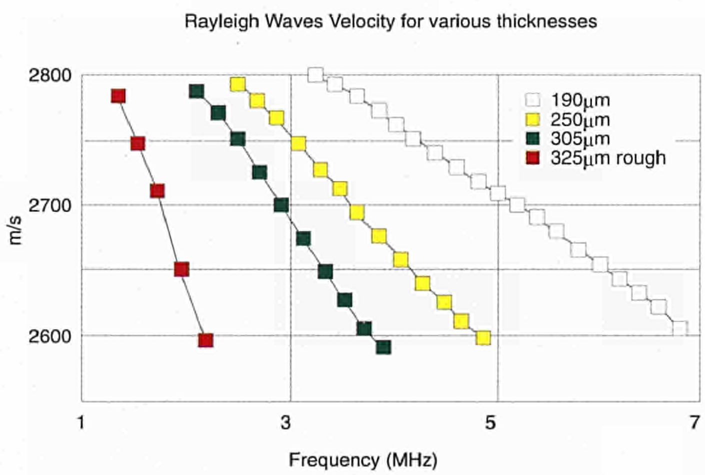
The actual ultrasonic beam can be reconstructed summing plane waves with various propagation directions; the mathematical tool used to find the magnitude of the plane wave is the FFT. Therefore, the previously mentioned computer codes for an individual plane wave will be used as subroutines in models for actual transducers.

A variant of the reciprocity theorem states that the sensitivity in reception in the case of a plane incident wave is proportional to the magnitude of the



Above: *Dispersion in a layered medium*

Below: *Phase velocities in a NiCoCrAlY coating*



plane wave generated in the same direction when the receiver acts as a transmitter. Hence, the same formulation for a plane wave in the case of a transmitter is used to calculate the electrical signal in reception.

Such a "system model" was already mentioned in the experimental part for the assessment of the beam distortion effect on the characteristics measurements. This kind of model will be used for the design of every future transducer.

Another practical interest is the prediction of the echo signal coming from the defect. Since the wave in the medium is already known as a part of the beam calculation, we think that the boundary element method is suitable to solve our problem.

A home-made computer code is in elaboration in order to apply the methodology to coatings with special beam profiles and various defect geometries. As previously mentioned, we can predict that defects larger than a few millimetres should give rise to signals higher than the noise.

The third point of interest is the prediction of parasitic signals. Modelling efforts on the subject will be undertaken.

Dispersion effect

The strong dispersive character of the propagation in a layered medium may lead to surprising effects as shown with the simulation of the signal shape change along the propagation path (figure above). That is why special signal processing has to be applied while measuring the propagation velocity of Rayleigh Waves in a coating.

The phase of a signal is defined modulo 2π but we know that all phase representations have the same derivative.

Therefore, the group delay that is given by the phase differentiation with respect to the frequency is measurable without any ambiguity whereas an infinite number of phase delays, regularly separated by one period are obtained.

In our case, we choose the phase delay that has the minimum difference with the theoretical phase delay calculated from the measured group delay.
Figure below shows an example of phase velocity measured on a NiCoCrAlY coating before and after

grinding at different thickness. These results prove that In Service Non Destructive measurements of the coating thickness variations are possible.

Conclusions

An intuitive reasoning has lead us to the conclusion that the use of surface Rayleigh waves propagating in a layered medium will enable its inspection. In fact, the vehiculated energy of such waves is concentrated underneath the surface, thus inducing a good sensitivity to the properties of the layer and also less parasitic echoes coming from deeper regions.
Theoretical models were developed and verified experimentally using a system enabling the generation of Rayleigh Waves and to measuring their propagation velocity in coatings.

Taking into account the results of this preliminary study, we can confirm that a technique based on the properties of Rayleigh waves is suitable for the inspection of metallic coatings.

During the next year, the work will go on with the measurement of the characteristics of metallic coatings deposited in the same conditions on various substrate materials in order to evaluate the reproducibility of their characteristics.
An important part of the activity will be dedicated to the improvement of the resolution in the defect imaging with the support of new models using the boundary element methods.
Successively, the results will allow the definition of the specifications of ultrasonic probes especially made for the purpose of measuring thickness and detecting defects on real coated components.

Research Area:
Surface Modification Technology

Assessment of Research Area Progress and Achievement: Surface Modification Technology

After some years of preparation and construction, this field of major activities of the IAM is now fully developed. In addition to processing studies on surface modification techniques an increasing number of characterisation studies on the properties of modified surfaces and interfaces are also under way. Surface modification requirements have also arisen in other research areas where specimens have been prepared for studying these applications. It is gratifying to note that this important area attracts much external attention and that more and more research contracts are awarded to the Institute by third parties in research and industry.

In 1993 the equipment for chemical vapour deposition was substantially improved. Similarly, there has been progress in the understanding of the processes taking place during laser cladding. In this treatment the liquid layer on the surface of the material is evidently in strong motion. For the understanding of the resulting layer it is therefore important to consider not only such phenomena as heat conduction and dendritic growth but also fluid dynamics. The parametric studies on the formation of the surfaces in the different coating procedures are aimed to develop new and more efficient coating procedures. In addition to these studies on the processes themselves, the laboratories were active in order to produce a large number of specimens for the characterisation studies.

This aspect of the IAM activities is constantly growing and will increasingly be devoted to the development of generally acceptable methods and procedures for quality control. Coatings can display a wide spectrum of improved properties, and they are used in quite different applications.

The Institute thus has to devote its attention to a wide range of properties and needs a large number of different methods for the characterisation of coatings. For example zirconia coatings have been investigated for their mechanical behaviour and for their resistance to thermal shocks.
Thermal barrier coatings are not only produced but also tested to characterise their properties and performance with respect to fracture toughness and fatigue life.

Surface modification research in the Institute includes the investigation of an important number of coating processes, and various activities which address surface phenomena and mechanisms affecting the interfacial behaviour in multi-material configurations such as joints and particle or fibre reinforced composites.

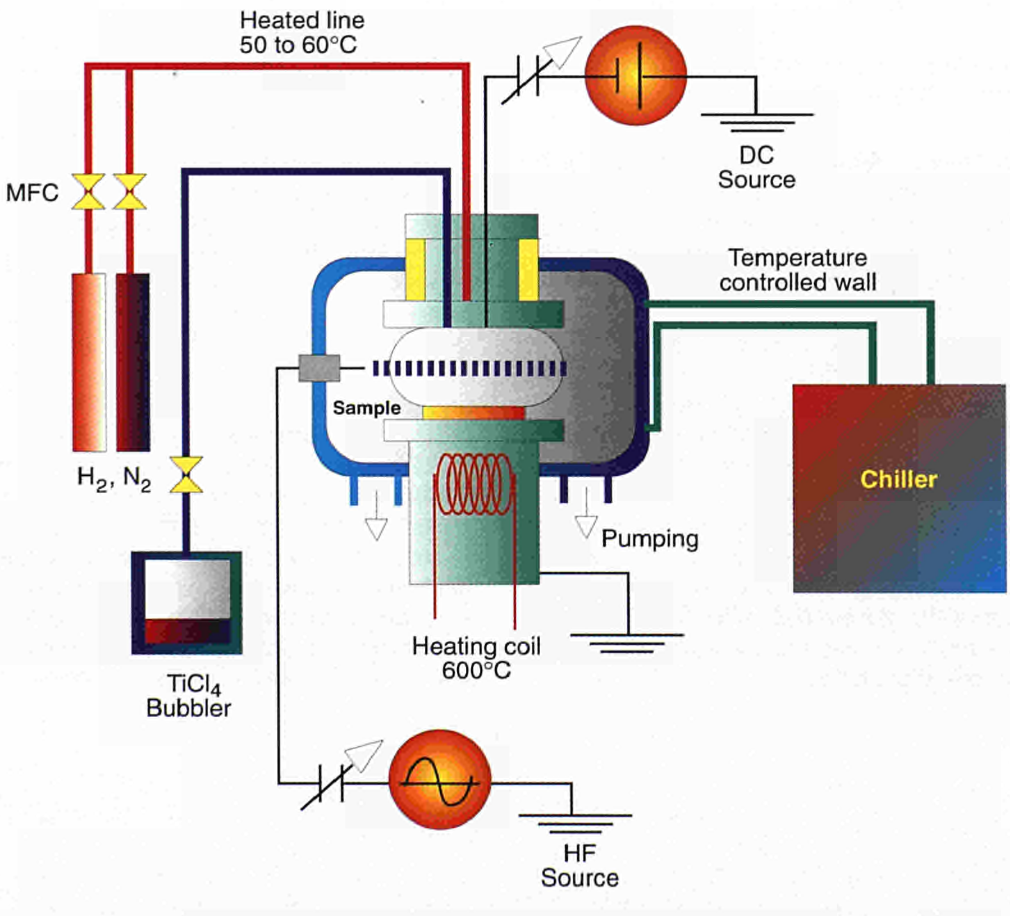
Investigation of surface spallation and wear performance by thin layer activation methods, and the study of flux line erosion due to Marangoni phenomena in refractory materials of metallurgical and glass making crucibles address surface studies of high industrial interest.
The electrical resistivity of ion implanted diamonds has been determined in different conditions.
Joints between ceramics and between ceramics and metals have been tested.
The oxidation resistance of implanted metals at high temperatures has been investigated by a new method. The surfaces of fibres for the fibre reinforcement of ceramics have been treated for an improvement of the properties of the composites.
Chemical sensors, aimed at the detection of environmental pollutants have been tested in a major European city.

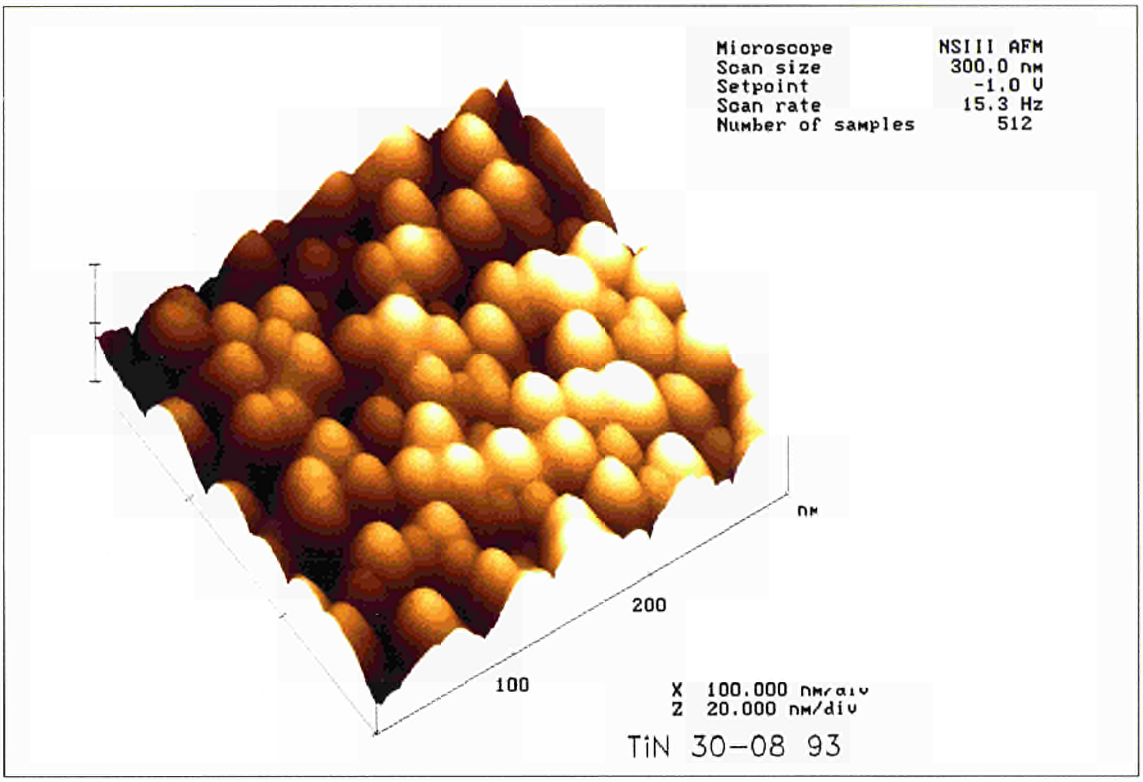
Wear and Corrosion Resistant Coatings

The facility is now fully functional for thermal spraying and chemical vapour deposition applications. Internal projects on new coatings, on coating process development, and the first external contract tasks have been started. The legal foundation of the Centre, as a co-operative venture between the Netherlands Energy Research Foundation (ECN) and the Joint Research Centre of the European Commission, has reached a conclusive phase. Chemical Vapour Deposition (CVD) processing activities were devoted to coatings for wear applications. The deposition and growth of hexagonal boron nitride for low friction properties was studied in a hot wall RF-excited PACVD equipment. In the evaluation of candidate precursors for continuous, dense coatings, a solid BH_3-NH_3 source proved to be to temperature sensitive, resulting in premature decomposition above 70^{0}C.

A gaseous BCl_3-N_2-H_2 precursor system, with Ar gas to generate a stable plasma and to encourage ion-bombardment processes has produced stable uniform coatings. The influence of the different deposition parameters on coating initiation and growth behaviour, are under investigation. Coatings are applied to Si wafers and to a standard tool steel. Coatings are characterized by friction/wear testing, hardness, scratch testing and by a wide variety of structural analysis techniques.
A parallel study into TiN and TiSiN, coatings is conducted in a cold wall RF-PACVD coating unit. The electrical configuration of this facility was modified to include a third electrode for the independent variation of the DC bias voltage. (Figure below). An extensive study of the influence of the influence of the deposition parameters, was preceded by a preliminary study of pressure and temperature effects. Variation of temperature between 400 - 550^{0}C has little effect on coating structure, while increasing gas pressure from 0.1 to 20 torr, produced a progressively finer coating structures of increasing

Below: *Triode RF-PACVD system for cold wall deposition of TiN coatings*





Above: *Atomic force microscope image of a TiN coating applied by RF-PACVD at 0.l. torr*

hardness value. Atomic force microscopy revealed a concomitant coating morphology variation from an egg-like topography (Figure above) to a columnar structure. Coating quality, hardness, wear resistance uniformity etc. compared very favourably with best reported values.

The nature of this work will proceed toward a study of plasma diagnostics leading to real time control of deposition parameters and "Intelligent Processing". This long term goal will begin with a study in depth into the parameters controlling initiation and early growth of TiN coatings.

Experimental studies will supported by the development of a model of the plasma, through the establishment of algorithms to describe the charged species of the plasma and referred to the deposition parameters, pressure, temperature, RF and DC potential and gas ratios.

The development of TiSiN coatings for improved hardness, wear and corrosion resistance has been started with a thermodynamic evaluation of the stability of the various coating species with respect to temperatures and gas ratios.

Thermal spraying activity has participated in a Round Robin test on spraying equipment performance through the Netherlands. Coatings were deposited by atmospheric plasma spraying, low pressure plasma spraying and high velocity oxygen flame spraying. ACC results ranked high for almost all coatings and were ranked first for deposition of Cr_2O_3.

Temperature control was seen to be an important controlling factor. A high speed pyrometer to measure particle and surface temperatures with a 1ms acquisition time is being developed to allow real-time control, and related to porosity, oxidation levels and residual stresses in sprayed coatings.

Increasing efforts are devoted to the development of coatings and coating technology for Third Parties.

Surface/Bulk Structural Properties Modification

In the course of the year the following activities have been performed:
- Deposition of a Hastelloy layer on a stainless steel surface
- Implantation of nitrogen on diamond
- Tests on stabilized zirconia thermal barrier using high power laser shots.

1. Deposition of a Hastelloy layer on a stainless steel surface

Hastelloy alloys combine good mechanical properties with excellent resistance to corrosion. In particular their application has been studied for deep gas wells with high concentrations of H_2S. In such conditions, resistance to very high pressure is required together with a good resistance to the acidic environment. Their application however is limited by the 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 indicates that a cost reduction factor of 0.5 to 0.6 is to be expected using cladded tubes with respect to bulk tubes of Hastelloy.

In order to deposit a layer of Hastelloy C 276, a 5 kW CO_2 laser was used, supplied by Rofin Sinar Laser Gmbh. The Hastelloy C 276 was fed in continuously using a wire feed supplied by Kematig. The wire was 1.2 mm in diameter and the composition was: C $\leq$0.02%, Co 2.5%, Cr 15%, Mo 16%, W 3.7%, Fe 5.5%, Ni bal.

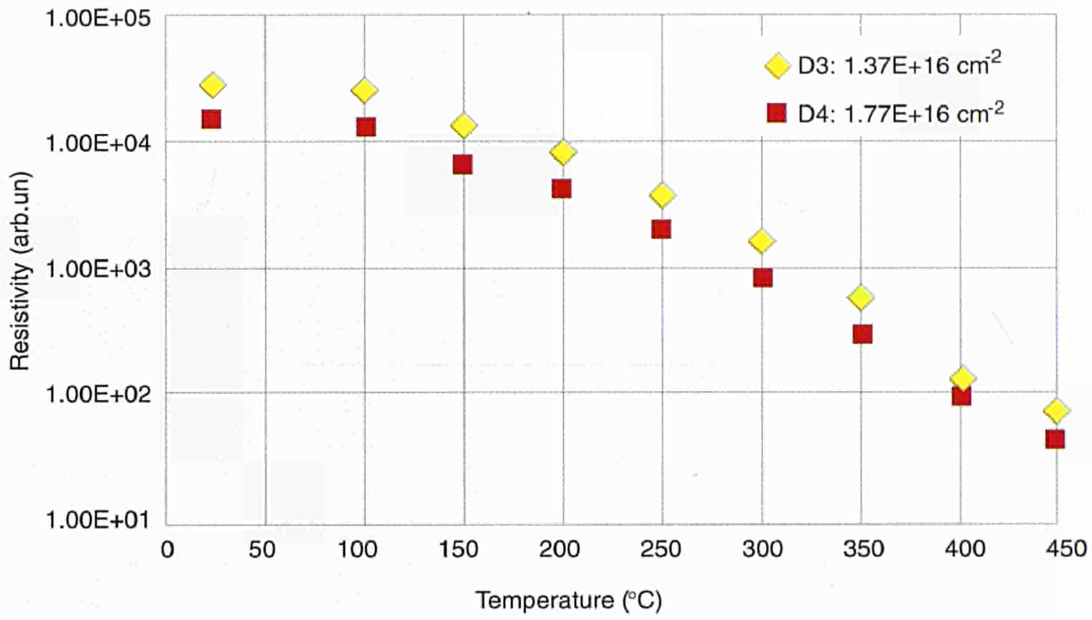
The base material was a 5 mm thick plate of AISI 316 stainless steel. During the coating operation, the plate was displaced under the lase beam at a speed of 650 mm/min, while the wire was fed at a slightly higher speed, 880 mm/min. The plate was covered by successive parallel passes at a pitch of 1.4 mm.

A first test was conducted at a laser power of 3.2 kW. To obtain a good adherence it is necessary to have a good melt pool where the two metal intermix and form a strong metallurgical bond. However it appeared that there were heterogeneous currents inside the melt pool, probably due to Marangoni flow. As a consequence a small stream of the molten stainless steel entered the mass of the molten Hastelloy. This effect was more marked when the laser power was increased, probably due to the larger thermal gradients inside the pool. The composition of the layer will be altered both microscopically due to interdiffusion of the different elements, and also due to infiltration of the molten base material into the deposited layer.
Microprobe analysis of the composition at four different positions within the layer showed that a large amount of material was transported into the layer, even when the total thickness was obtained by four successive passes. It is interesting to note the different behaviour of the various elements.

Molybdenum, which has a lower concentration in Stainless Steel than in Hastelloy, reaches rapidly the highest value. On the contrary, nickel on approaching the external surface begins to reach the concentration typical of Hastelloy. Iron behaves differently and is enriched in the Hastelloy region.

2. Implantation of nitrogen on diamond

Diamond is a wide gap semiconductor, with a very high electrical resistance. Due to its excellent thermal conductivity it would be an ideal material for high power transistors. However, it is rather difficult to dope diamond, because it transforms to graphite at about 1200^{0}C. However, even diamond with just surface conductivity would have interesting applications. In particular a conductive hard surface would drastically simplify the measurement of hardness in metals. The size of the indentation made by the diamond pyramid could be determined from a simple resistivity measurement. From literature data it is known that implantation of diamond induces an amorphisation of the surface layer. The resistivity of this layer is typical of glassy graphite. If the implanted layer is annealed, the resistivity decreases tending towards the resistivity of well-crystallized graphite. To maintain the hardness of the diamond, it was decide to implant with a light ion, namely nitrogen, so that at the surface the impinging atoms lose energy only by ionization, producing displacements in the interior of the diamond. Four industrial diamonds were implanted with nitrogen ions at four different energies, 50, 100, 150, 200 Kev, for total nominal doses respectively of $6x10^{15}$, $1x10^{16}$, $1.4x10^{16}$, $1.8x10^{16}$ respectively. The resistivity was measured using the four electrode method. Due to the difficulty of measuring the thickness of the amorphized layer, the



Above: *Variation of resistivity after 1 h isochronal annealing*

resistivity was calculated in arbitrary units. The resistivity decreased with increasing dose of implanted ions. To follow the variation of the resistivity with temperature, the diamonds were annealed for one hour at progressively higher temperatures.
Figure above shows the values obtained for the higher irradiation doses. It can be seen that the resistivity decreases sharply with temperature.
However, at 500-550^{0}C, the resistivity suddenly increased to the value of the unirradiated diamond and the diamond surfaces, which were darkened by the implantation, recovered their initial transparency. It appears that implantation produces interconnected island of disordered graphite inside the diamond which are kept under pressure by the surrounding diamond. When the temperature allows sufficient atomic mobility, the damaged structure reverts to diamond which is the thermodynamically stable phase due to the high local pressure.

3. Test on stabilized zirconia thermal barrier

The use of thermal barriers to increase the working temperature of gas turbine components has been under study since 1970. Whereas final testing needs to be performed under real conditions, much effort has been devoted to developing tests which could allow a rapid evaluation of the different materials and coatings procedure.
A complete theoretical analysis of the coating behaviour is possible only if the physical and mechanical properties of the porous ceramic coating are known. At present this is not the case and testing can only establish a comparative ranking between the different coatings.

The acceptance standard for coatings adopted by the industrial manufacturers is based on the measurement of the adhesion strength, on the measurement of crack extension in a cupping test, and finally an examination of the morphology of the external surface. These tests are performed on plane samples which are prepared in parallel to the turbine components. The acceptance value are purely empirical and have been defined on the basis of previous testing experience.

In our laboratory some tests were conducted on plane samples covered with 0.5 mm of ZrO_2 in order to simulate the very rapid heating experienced by an airplane model when subjected to a hypersonic air flow in a shock tube. These tests were performed using short pulses from a high power laser.

The effect of single pulses of different powers, and of a series of 100 pulses were investigated. The metallographic examination has been performed on the original samples, on the samples treated with only one shot, and on the thermally cycled samples.

The metallographic investigation on the samples exposed to a single shot has been performed only on samples from one manufacturer.

The power level of 2 kW/cm^2 does not changes the grains size significantly. The porosity appears to begin to coalesce in larger pores and some laminar porosity is observed.

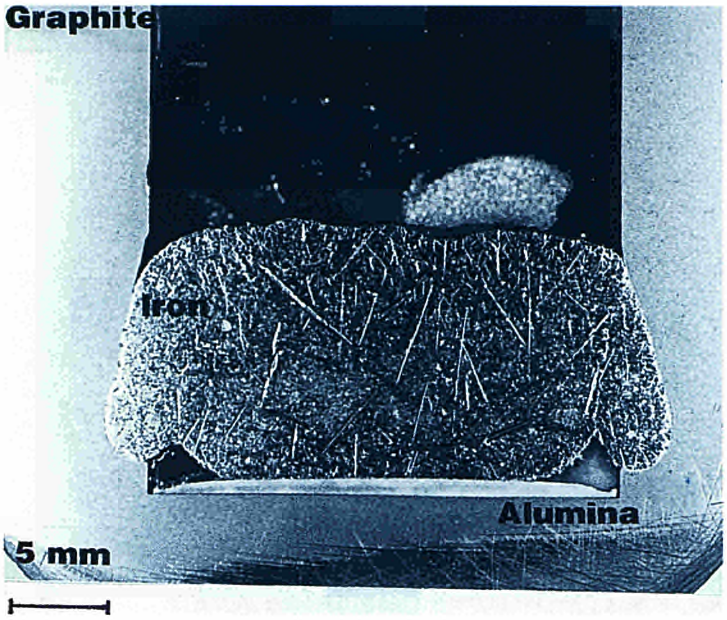
Higher power level, 4 kW/cm^2, resulted in a larger grain size at the surface, the small porosity tended to disappear, and a small dome was formed.

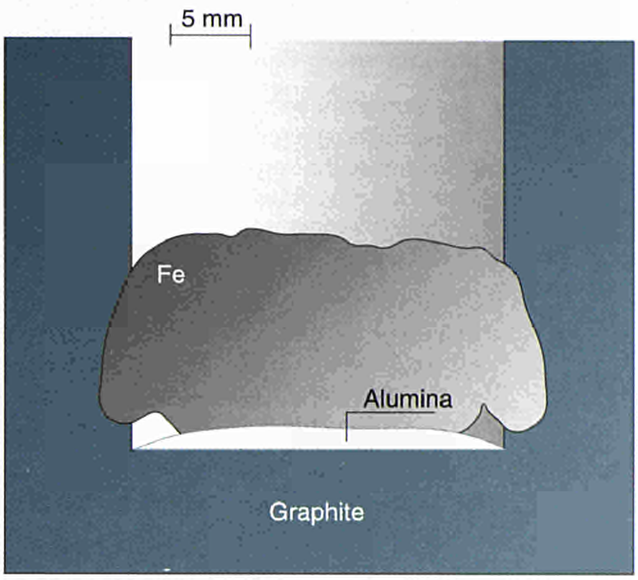
Finally, at the highest power level, 6 kW/cm^2, it can be seen that a thin layer of the outer surface has been melted, leaving a perfectly dense layer in the form of a dome with radial fissures. The thermal cycling tests have been applied on all three types of samples. In the first type of samples, the cycling tended to enhance the phenomena observed after the first shot. In particular the laminar porosity is more pronounced and the coalescence of the porosity in the outer layer is more marked. The general appearance of the layer however was not significantly changed. The second product behaved better. After cycling only the first stages of the formation of structured laminar fissures could be observed. There was also some coarsening of the porous structure at the outer surface. The third product had the poorest behaviour. After cycling extensive fissures were present at the interface between the ceramic layer and the intermediate layer. In conclusion, it can be said that such a test, followed by metallographic investigation, was able also to give a general evaluation of the resistance to thermal cycling. Due to the inherent simplicity of the test, it appears worthwhile to investigate its possible extension to more general uses.
The detailed results are described in the JRC report EUR 15406 EN.

Marangoni Phenomena in Materials Processing

The enhanced erosion of refractory walls in the glass and steel making industries at the flux or slag line is a costly phenomena. It is thought that interfacial tensions between the two liquid phases at the solid wall lead to surface tension driven flow otherwise known as Marangoni flow. These relatively large flows sweep away the dissolved refractory wall exposing fresh refractory wall to renewed attack. The interplay between the refractory wall and the melt was determined by the rate of refractory dissolution and the flows generated determined the overall erosion profile. A mathematical model recently developed at the Institute of Advanced Materials, Petten, predicts the erosion profiles for different flow conditions.

The present experimental work aims to measure the erosion shapes and erosion rates of a three phase gas-liquid-solid interface system in order to determine the dominant erosion mechanism. This work is of a basic nature in that better understanding of the mechanisms and their relative importance to the erosion will be determined.
The system under investigation is the iron-carbon-sulphur ternary system where iron-carbon alloys are melted in graphite crucibles under different temperature regimes.
The initial set of experiments looked at the effect of buoyancy driven flow induced in the iron-carbon melt by the dissolution of the graphite walls creating density gradients.

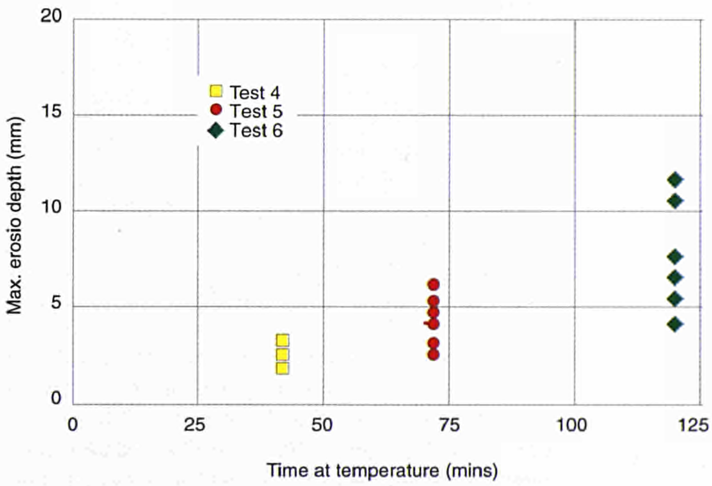




Above left: *Cross-section of solidified iron in graphite crucible*

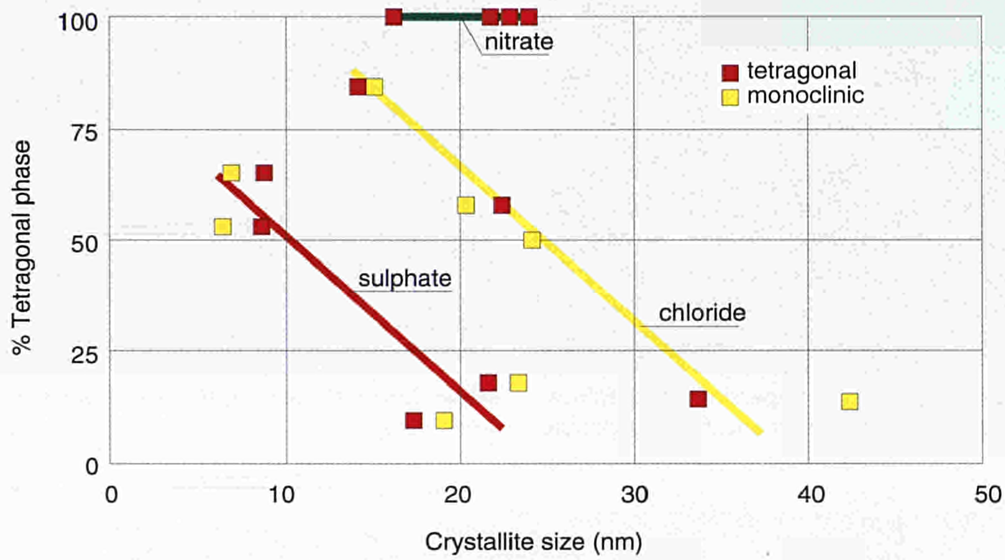
Above right: *Erosion profile magnification x 4*
- - - - - = original crucible shape —— = eroded profile

Below: *Erosion rate of C crucible containing pure Fe at 1550ºC*



These tests were performed using a pure iron sample under reducing atmospheres at 1550ºC. The erosion of the refractory wall was enhanced at the bottom as expected because carbon is less dense than molten iron and so, upon dissolution, is swept upwards creating proportionately more fresh graphite wall at the bottom of the wall than at the top.
A typical erosion profile of a cross section of the crucible with the solidified iron is shown in figure above. The enhanced erosion at the bottom of the crucible wall can clearly be seen; erosion of the base of the crucible itself is prevented by a layer of alumina thus simplifying the erosion profile.
The effect of carbon on the surface tension of the pure iron is minimal and so enhanced erosion at the surface was not expected. The erosion rate is illustrated in figure on page 49 below where the maximum erosion depth is shown as a function of temperature. It can be seen that the erosion depth increases with time.

Sub-microstructural Engineering



Above: *The dependence of the size and stability of the tetragonal phase crystallites on the precursor anion species*

Zirconia powders were produced from acidic aqueous solutions of various zirconium salts dispersed as water-in-oil emulsions. Precipitation was induced by ammonia, either directly by bubbling ammonia gas through the emulsion, or indirectly by the release of ammonia resulting from the thermal decomposition of a suitable organic reagent. In the emulsion, each droplet acted as an individual chemical reactor thereby ensuring the uniformity of the precipitate on a microscale, even for mixed-cation solutions in which precipitation occurs at widely differing pH values. The principal aim, however, was to control the size of powder agglomerates through control of the emulsion droplet size to obtain powders with characteristics suitable for typical powder processing operations such as pressing and sintering.

The work reported here has progressed as far as investigating the powder compaction characteristic. These characteristics were studied for powders prepared by direct precipitation from different precursor solutions, and dried by various techniques. The nitrate, chloride, and sulphate of zirconium were used as precursors. Powders were dried by distillation, oven-drying, and freeze-drying. The morphology and microstructure were characterized by SEM, TEM, HREM and XRD, and the compaction characteristics were measured using an instrumented hydraulic press. The precursor solution strongly influenced the primary crystallite size and the tetragonal to monoclinic phase transformation of the calcined powder (Figure above).

The compaction behaviour showed a dependence on precursor species, drying method and calcination temperature. The direct precipitation method, however, tended to produce hollow spheres due to the precipitation reaction taking place preferentially at the surface of the droplet. In contrast, the thermally induced precipitation method results in uniform precipitation throughout the volume of the droplet and thereby avoids the phenomenon of hollow spheres.
Primary crystallite sizes of about 10 nm and powder granules of 2-200μm were obtained.

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

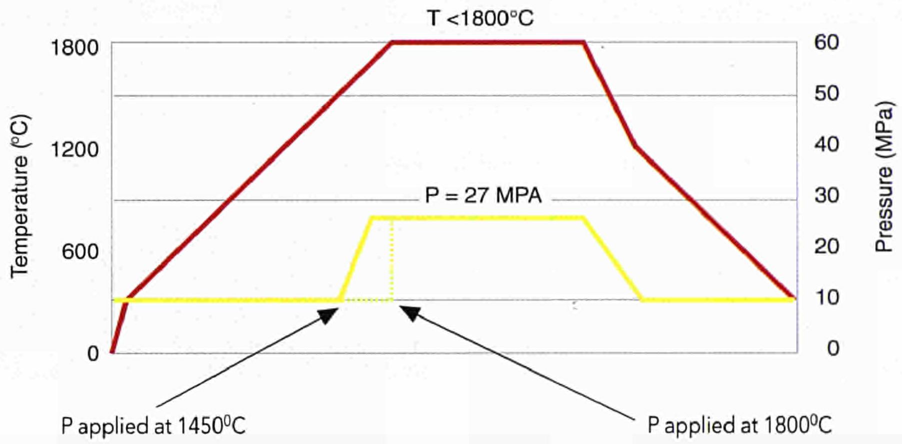
The activities of the project are directed to the engineering of fibre-matrix interfaces to:

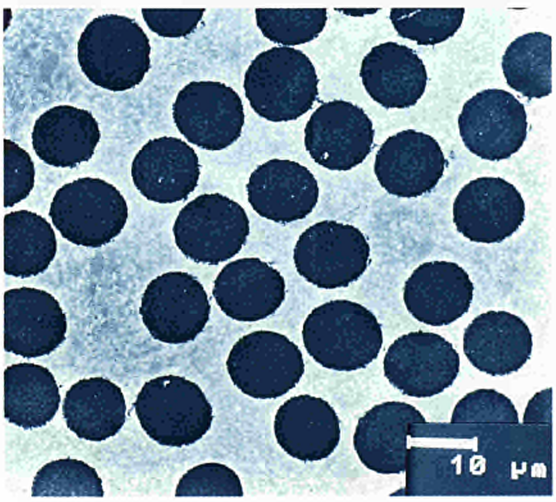
- improve composite fracture toughness
- minimise fibre-matrix chemical interaction
- accommodate interface stresses induced by thermal expansion mismatch

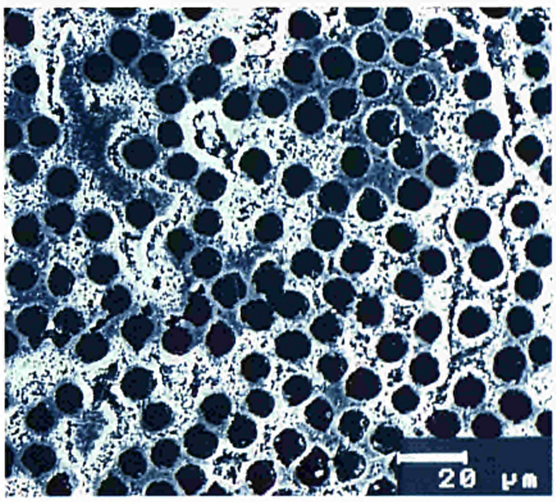
Composite Processing

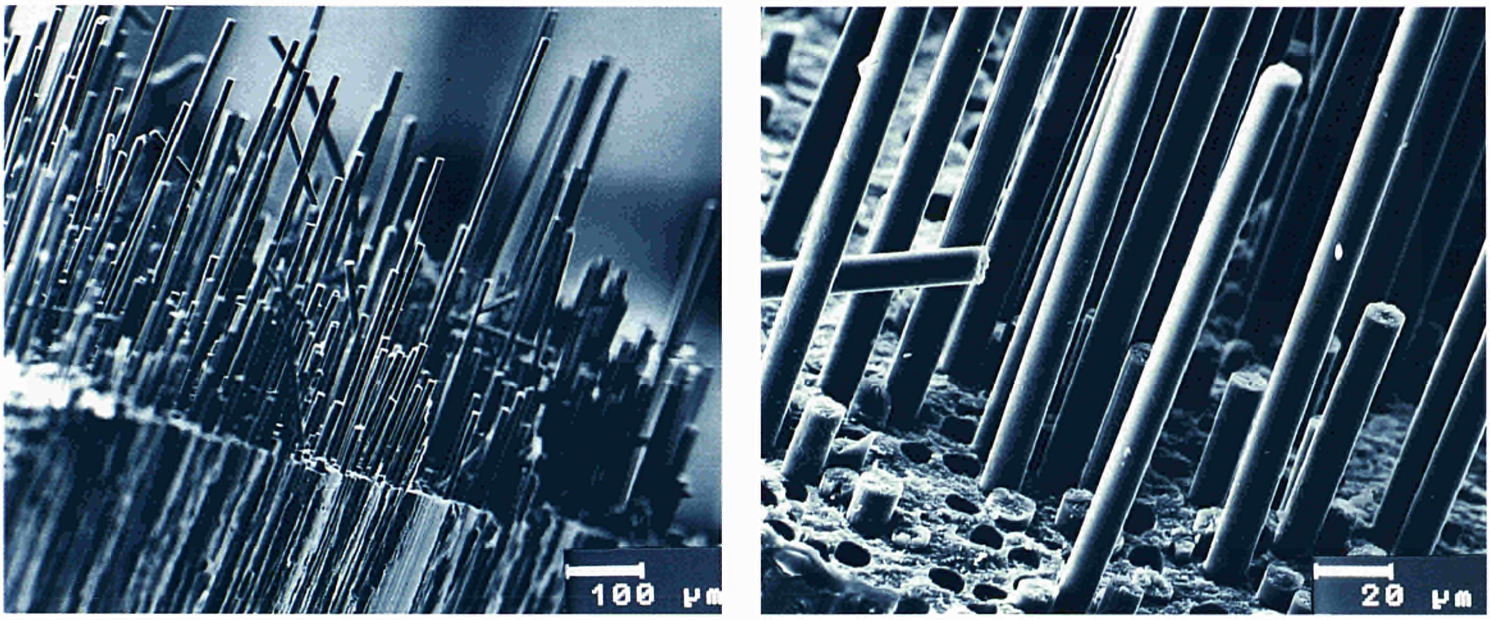
The first phase of this project has addressed the reinforcement of silicon nitride by high modulus, pitch-based carbon fibre. Optimisation of the microstructure has been achieved by iterative improvement of the green forming methodology, ie. fabrication of thin uniform fibre tapes by spreading and weaving, tailoring of dispersion viscosity/wetting characteristics, development of a novel double layer impregnation technique, and pressing and drying of the green composite, leading to a 30 ply green body of some 55% density. Subsequent hot pressing yields a dense composite, with a 35% fibre content. An optimised pressure/temperature cycle inhibits Si_3N_4 decomposition without damaging the fibres (figure below), and minimises thermal stress cracking on cooling the composite.

Below: *Optimisation of the Hot Pressing Cycle for a Carbon Reinforced* Si_3N_4









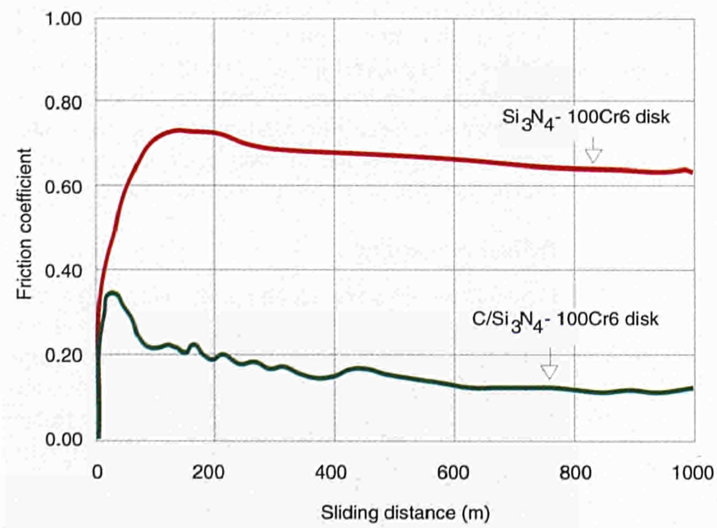
Above: *Fibre pull-out in a Carbon-SiC-Si_3N_4 Composite*

Influence of Interface Chemistry on Mechanical Properties

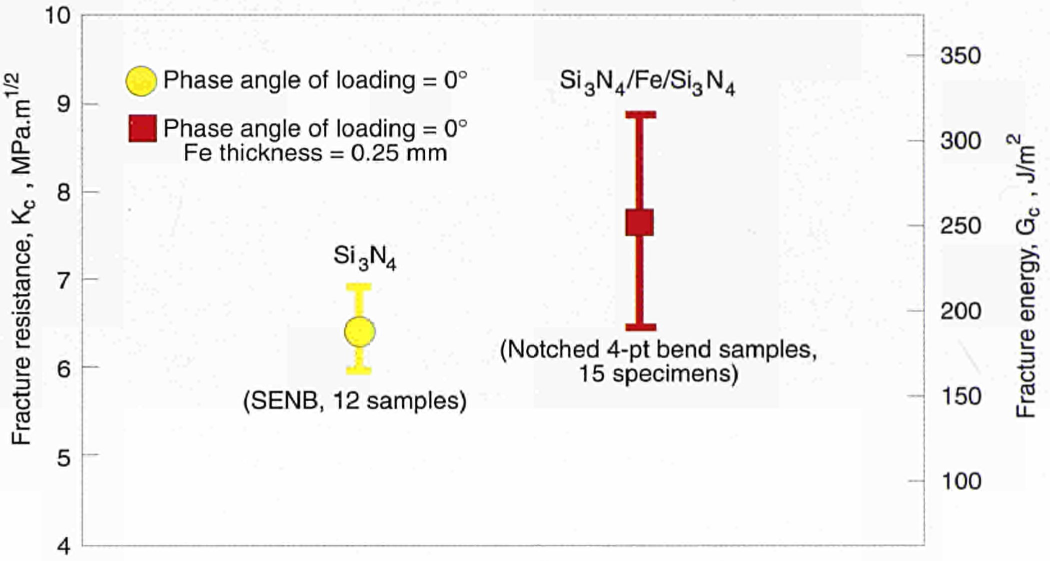
Composites have been fabricated with four different interface conditions, with fibres uncoated, and precoated with pyrolitic C, with SiC, and with a duplex pyrolitic C + SiC. Fracture profiles and fractured surfaces showed clearly that graceful failure of the composite was linked to the propensity for fibre pull-out. Of the four combinations, the SiC coated fibre composite exhibited the highest degree of toughening with a fibre pull-out length up to 1 mm, (figure above) while retaining a rupture strength of 500 Mpa. Microstructural observation and chemical analysis of fibre surfaces (SEM/EDAX, EPMA and AES) suggest that fracture occurs between the fibre and the coating. The composites also showed improved wear and friction behaviour in dry sliding on steels, as compared with a monolithic Si_3N_4 (figure below). Wear properties are influenced by macrodefect population and fracture toughness of the composite and the formation of a lubricating debris film between the contacting surfaces.

Next studies will concentrate upon the influence of interface chemistry and structure and of matrix chemistry to achieve improved composite properties at high temperature and under thermal cycling.

Below: *Friction Coefficients in dry sliding on Steel Disc, monolithic Si_3N_4 and Composite C-Si_3N_4*



Advances in Ceramic Joining



Above: *Si_3N_4/Fe joint interface fracture energy and fracture toughness compared with that of the monolithic ceramic (4-pt bend specimen)*

The aim of this project is to enhance the joinability of Si_3N_4(SN) ceramics through a better understanding of the mechanisms of ceramic/metal(C/M) bonding. Improved joint properties have been achieved by controlling fabrication parameters to optimize interface microstructures and fresh studies have commenced on interface fracture mechanics and on the modelling of residual stresses.

Diffusion Bonding

Previous work had produced joints as strong as the parent ceramic by using Fe, Fe-Cr alloys as the bonding interlayer, as shown by the interface fracture energy measurements (figure above). However, diffusion bonded joints commonly display relatively large variability in strength values. Work on the origin of this variability has indicated the relative importance of central or edge locations of the bonded assembly on factors such as reaction kinetics, degree of contact and residual stresses.

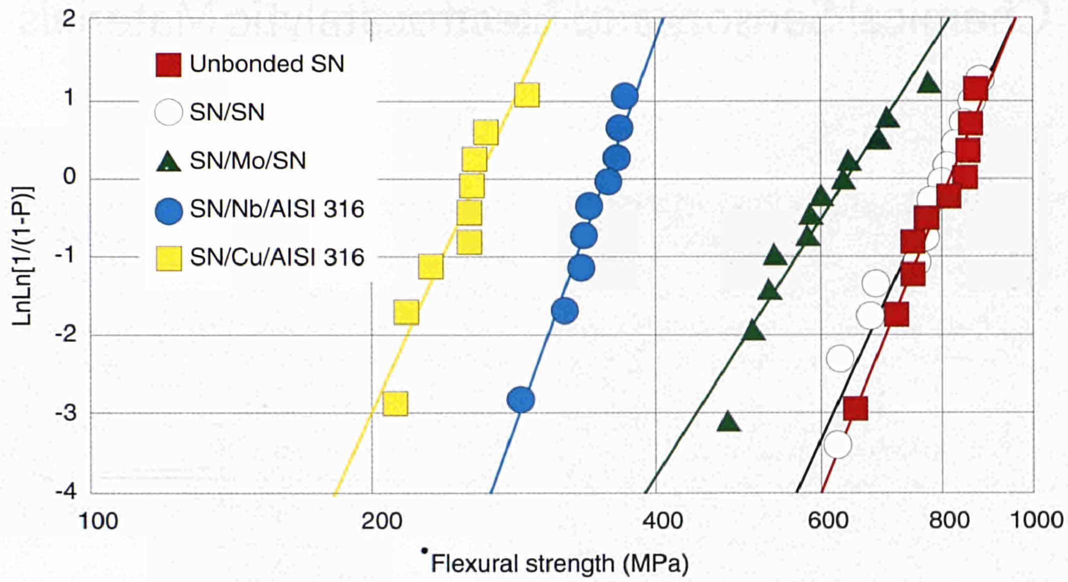
Brazing

The usefulness of joining SN to itself or to metallic alloys with commercial AgCuTi and NiCrSi brazes, and the criteria for selecting effective interlayers in C/M brazing were assessed.

Ceramic/ceramic joints with similar strengths to that of the monolithic were produced using the Ti-active braze, while the strength of C/M joints were less due to residual stresses (figure on page 53 above).

NiCrSi brazes performed less well because of excessive-pickup of Si from the ceramic and embrittlement of the braze seams. Experimental phase diagram and thermodynamic studies have confirmed the unsuitability of these alloys for SN brazing.

To overcome the high temperature brazing problems the development of a new technique was started. Early trials of bonding SN with Au/Ni-Cr interlayers produced joints with strengths comparable to those achieved by diffusion bonding, but at lower temperatures and without the necessity to apply uniaxial pressures.

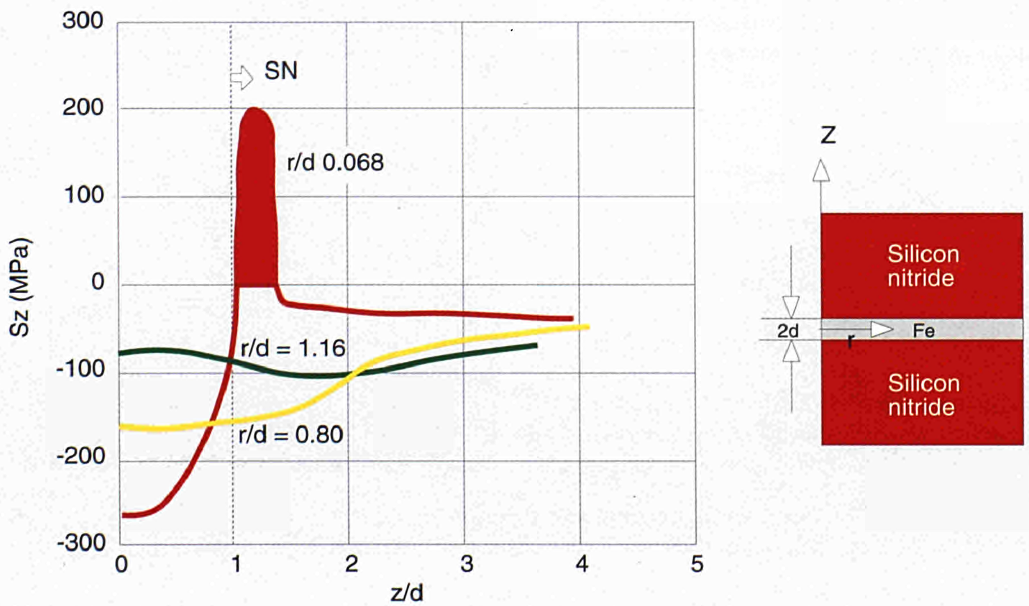


Above: *Strength distribution of Si_3N_4 brazed (Cusil-ABA) joints*

Modelling and Measurements of Residual Stresses

The residual stresses in C/C and C/M joints were calculated by FEM for both 2D-and 3D-geometries taking into account thermal and elastic mismatch, metal interlayer yielding, and work hardening. The 2D- and 3D-analysis results were in fair agreement, and they highlight the importance of metal interlayer plasticity on the critical tensile stress at the ceramic edge (figure below). Residual stress measurements using the XRD method(triaxial analysis) have been performed on SN/M(=Cu, Nb, Mo, Fe, W, AISI 316) joints; the results show good qualitative agreement with the FEM calculations.

Below: *The residual σ_{zz} stresses near the edge of the joints*



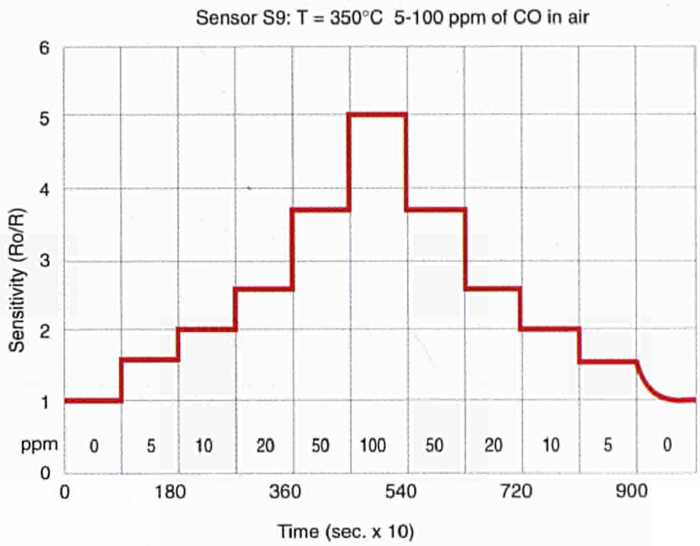
Chemical Sensors and Electrocatalytic Materials

The work on "chemical sensors" for monitoring the atmospheric pollutants was extended to carbon monoxide. As for NO_2, the sensitivity corresponding to the first alarm threshold, as fixed by the E.U. regulations, was easily attained. The response rates to CO concentration changes were faster than for NO_2.

Figure above shows a typical "stepwise" response curve. The possibility of measuring both NO_2 and CO with the same sensor has been demonstrated. This is possible because the temperature of maximum sensitivity is very different for the two chemical species. For CO it is around 350^{0}C, whereas for NO_2 it is ~200^{0}C, at which temperature the interference of CO is very low (figure below).

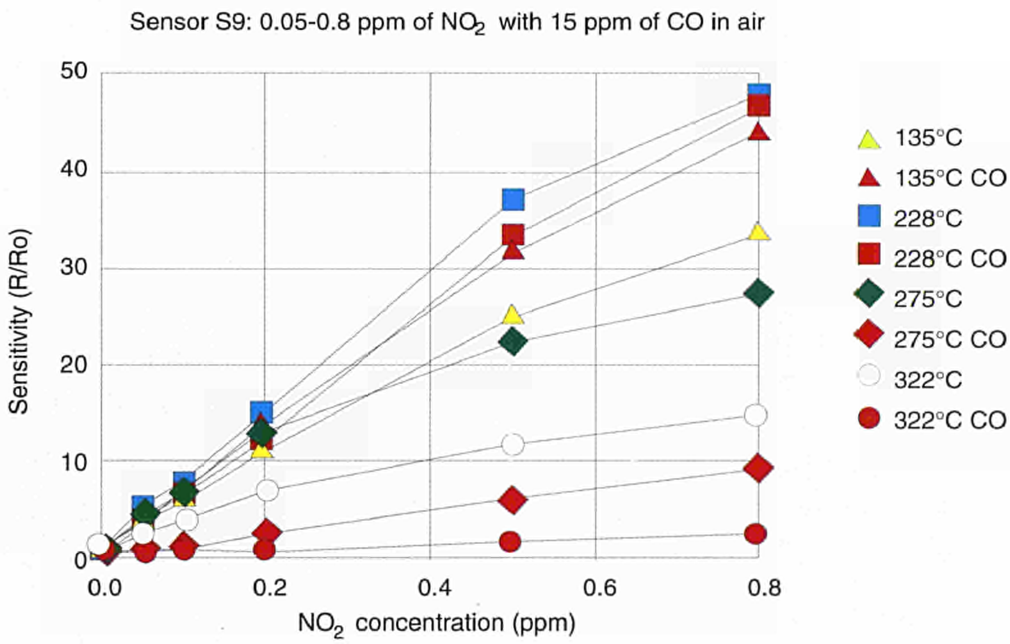
To better understand the conduction processes that have led to such extremely high sensitivities, contacts have been established with CSTE (CNR) at Pavia in the frame work of the recently signed CNR-iAM agreement. Preliminary experiments are in progress with the impedance frequency spectroscopy technique.

Theoretical models are also contributing to elucidating the prevailing mechanisms of the charge carrier transport.



Above: *Typical response to CO of a tin dioxide monolayer sensor*

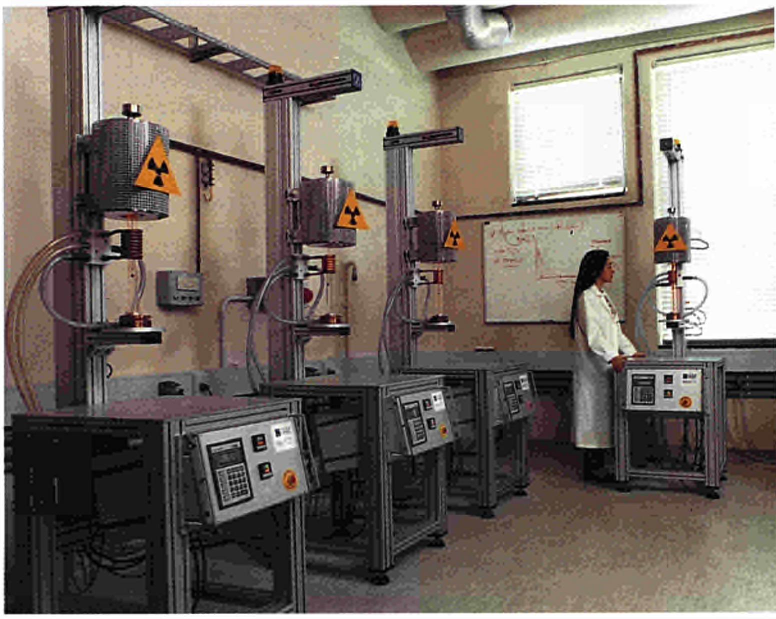
Below: *Sensitivity to NO_2 in a CO containing atmosphere at different temperatures*



Surface Scaling

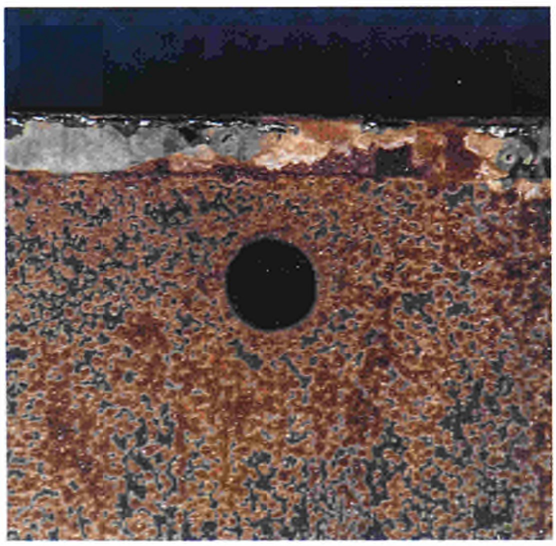
The objective of this project is to study surface degradation processes due to thermal cycling and wear by a combination of various methods, with emphasis on Thin Layer Activation (TLA) and to study by surface modification, the possible improvements of material performance. The principle basis of TLA is the creation of radionuclides in a surface layer by exposure to a high energy particle γbeam, from, for example, a cyclotron. The nuclides disintegrate with emission of nuclide specific γ-radiation. Any loss of the activated material due to a material degradation process will result in a loss in γ-activity of the activated component. This reduction in signal can be directly related to mass or depth loss. The specific properties of TLA include area selectivity, high sensitivity, speed and applicability as a noncontact in-situ method.

In the year under review, a research agreement was signed with the International Atomic Energy Agency (IAEA) within the Research Programme "Nuclear Methods in Monitoring of Wear and Corrosion in Industry".



Above: *The fully automized test facilities employing Thin Layer Activation for thermal cyclic corrosion testing*

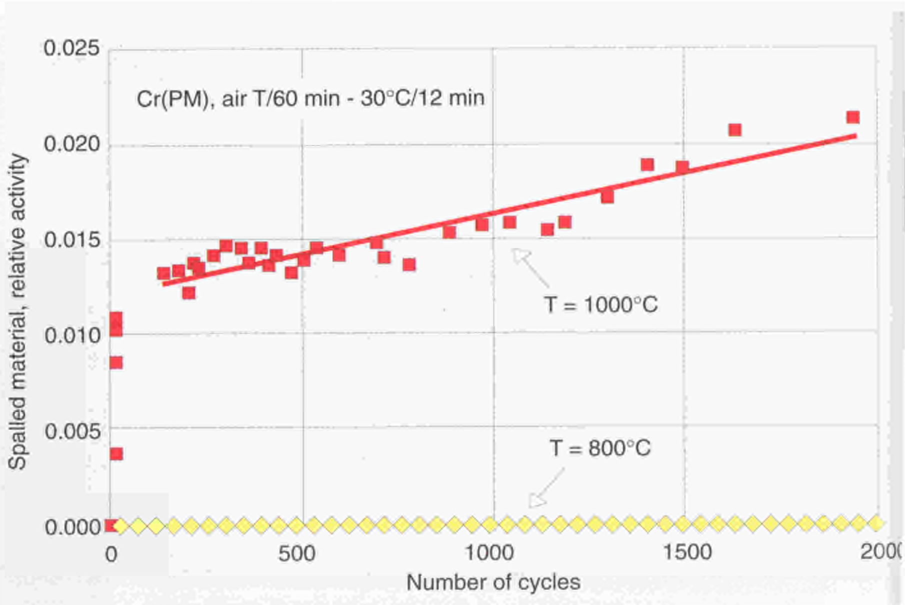
Below: *Surface-scale morphology near the boundary of unimplanted (top) and Nb implanted (bottom) TiAl-based intermetallic after 600 thermal cycles of each 1 hour at 800°C. Extensive scale spallation present on untreated material is absent on implanted part*



Various discussions and collaborations with universities, research centres and industries in Europe were initiated. Such collaborations are especially appropriate in view of the very limited number of research centres in Europe with facilities to employ TLA for surface degradation studies.

Spallation due to Thermal Cycling

Cracking and spallation of protective oxide scales and coatings due to thermal cycling is a subject of large concern in a wide range of technological applications, ranging from coatings on gas turbine blades, heat exchanger applications, to oxide fuel cells.
The understanding of the phenomena of spallation is generally poor and is insufficient to predict the material behaviour adequately.



Above: *Influence of maximum temperature on the material loss due to spallation caused by thermal cycling of powder metallurgical Chromium as determined by Thin Layer Activation*

TLA can significantly contribute to a better scientific understanding of the underlying processes and to a more reliable assessment of the service lifetimes of technical components. The application of TLA in thermal cyclic testing was developed further by our Institute. A laboratory with unique TLA/cyclic corrosion facilities became operational.
Protection of the intellectual rights by patent application covering both testing method and test facilities was undertaken in collaboration with DG XIII.
One barrier to progress in the understanding of cyclic corrosion is the large differences in testing conditions employed in various laboratories. This makes meaningful comparison of data from laboratory to laboratory almost impossible. A critical study of the various tests revealed that the important testing parameters are still inadequately known, thus preventing the formulation of test guidelines or test standards. A beginning was made with research in the relevant test variables of cyclic corrosion testing. This activity is linked to an initiative of the European Federation of Corrosion. This line of pre-normative research will be expanded.
A collaborative action was initiated with KFA-Jülich and our Surface Modification Centre on the effect of surface treatments on the scaling and spallation behaviour of TiAl-based intermetallics. The effect of Nb ion implantation led to a significant increase in oxidation resistance at 800^{0}C. This improvement was also maintained under thermal cycling.
Further work is dedicated to elucidate the underlying mechanisms.
The effect of minor additions of reactive elements on oxide scale spallation is performed in a joint activity with the University of Delft (The Netherlands), University of Bourgogne (Dijon, France), Plansee GmbH (Reutte, Austria) and our Surface Modification Centre. Various chromia forming high temperature materials were tested under thermal cycling in the temperature range of 800-1000^{0}C for testing times up to 3000 hours. Tests are performed using conventional gravimetric measurements and employing our TLA facilities. Additionally, the installation of acoustic emission facilities is in preparation. TLA results revealed that the rate of material loss during thermal cycling of powder metallurgical produced chromium could be reduced by a factor 1000 by the addition of small amounts of yttria to the material.
Due to its specific properties TLA was able to quantify accurately these and other differences. The TLA data on spallation will form the basis for lifetime assessment of technical components. A computer programme developed for this purpose by the NASA-Lewis Research Center was placed at our disposal. The TLA data is expected to lead to a significant improvement in the accuracy of the life time assessment calculations in comparison to the conventional weight change data.

Wear Testing

During the reporting year a start was made with the use of TLA in wear testing. A task force analyzed the various potential application areas with a strong emphasis on industrial applications. A marketing study is ongoing and various possible projects were formulated. It was observed that TLA has significant potential especially in cases where small wear losses have to be measured or for non-contact or in-situ measurements. A specialized facility for lubricated wear testing using TLA, including a hydraulic and a difference measurement system, became operational.

Coatings Characterisation

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

Results are reported here on the mechanical properties of YPS ZrO_2 thermal barrier coatings deposited on a Ni-base alloy (C263) provided with a NiCrAlY bondcoat. For this purpose 4 point bending tests under constant head displacement rate combined with AE-analysis were carried out. Some typical results are shown in figures below.

From these 4-point bending test results, it can be concluded that:

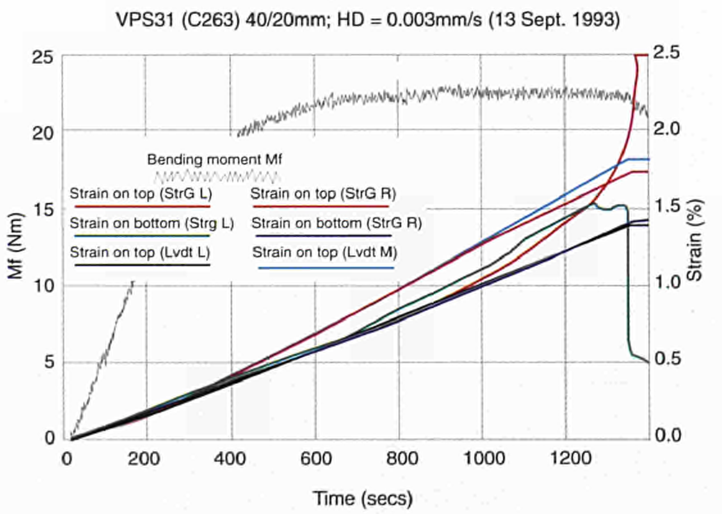
i) the onset of transverse cracking of the coating can be identified by a deviation of the top/bottom strain ration from its initial value whereby no change in the rate of the bending moment is occurring. For the present TBCs typical values for the transverse failure strain of 0.3-0.4% were found.

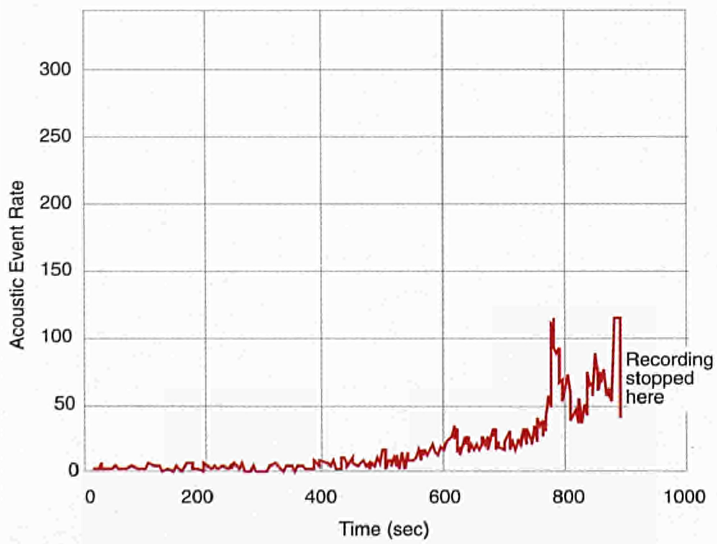
ii) the onset of parallel cracking (i.e. delamination) is characterized by a deviation of the ratio of the deflections measured at the centre and the left (or right) side of the specimen from the initial value. This is accompanied by a slight decrease in the bending moment. On basis of the above criteria, failure strains of 1.0-1.2% were determined for the onset of delamination of the studied TBCs.

iii) the chosen experimental set-up appears to be very promising as a standard procedure for the determination of failure strains in plasma-sprayed coatings.

Below left: *Bending moment and resulting strains at top and bottom of TBC-coated C263 test-pieces as function of time (head displacement rate 0.003 mm/s)*

Below right: *Rate of acoustic emission vs. time during 4-P bending test*





Research Area: Materials Information and Data Management

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

The research area "Materials Information and Data Management" comprises two functions:

- the development and operation of computerised data management and evaluation systems
- a support service for the organisation of meetings/conferences, publicity and internal communication.

In the first of these two functions the Institute addresses the computerisation of materials information in the two principal lines of the Advanced Materials programme: Materials Characterisation and Materials Processing.
The High Temperature Materials Databank (HTM-DB) is designed for the mechanical properties of high temperature alloys. An analogous databank for aluminium alloy has been derived from the HTM-DB for a group of automobile manufacturers.
In the materials processing line, a computerised data system for coatings technology is under development.
The Coatings Process and Property Databank (CPP-DB) is designed for process parameters, coating and interfacial properties and special features for in-situ data collection from process sensors. Its design has the basic ability to contribute to an intelligent processing development.

The projects develop own designs of ergonomically advanced user interfaces, data evaluation programmes and data input and output formats, making use of commercially available, advanced database software. There is extensive co-operation with industrial and university partners and European research programmes such as COST, BRITE/EURAM and the thermonuclear fusion programme. Speçial efforts are made to develop the potential for the provision of data services and software to paying customers. The HTM-DB has been successfully demonstrated to interested parties and has become widely known.

The diversity of relationships, contacts and collaborations of the Institute with external partners will in future be extended to the setting up of networks with research institutions on joint tasks. The function of the information support service will therefore increase in importance. It has made beneficial contributions to the related tasks of progress assessment and evaluation of research projects and to the collaboration between the Units of the Institute at Ispra and Petten.

Coatings Processes Data Banks

A computerised relational Plasma Spray Coating Properties and Processes Data Management and Manufacturing System (CPP-DB) has been developed. Its objective is to assist coating users and manufacturers to manage their data or to assist the manufacturing process by process monitoring and optimisation.

To achieve this objective, the system provides the following tools: a coating database, a process sensors data acquisition system with data transfer to DB facility, a process control interface, a database for experimental data of processing or post-characterisation tests (multi-spreadsheet and multi-graphics environment) as well as data analysis/evaluation and graphical procedures. The coating database includes: the process, coating properties, thermo-physical and mechanical properties of powder, publications and coating industries sub-databases.

All these tables are interrelated and a combined search is possible. The coating properties include: adhesion, residual stresses, porosity, electrical conductivity, microstructure and other layer properties.
Graphical on-line monitoring or display of instantaneous process sensors data are standard capabilities (process survey) of the Data Acquisition System.

The CPP-DB provides the capability to the user to create and interface the users own functions or to process mathematical models so that a computerised feedback process control loop can be established. The set points can be the DB data or the results of mathematical models. The data bank is a first step towards Plasma Spray Coating Intelligent Processing and is being developed within the framework of a human capital and mobility network programme.

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 client/server system. The database is implemented with commercially available Relational Database Management systems, the master database on the workstation with ORACLE and the local database on the PC of the client with SQL Base. The PC-based interface is installed on the PC-side under Microsoft Windows and is therefore compatible with other Microsoft Windows software such as spreadsheets, word processing software and help compilers. The data are retrieved from the local database, but for data entry and data exchange the user interface interacts with the workstation via Internet.

In 1993 work has continued on:

- the transfer of the evaluation program library from the PC/MS-DOS environment into Microsoft Excel for Windows
- improvement of the data entry and data retrieval functions through implementation of intelligent user guidance developed in co-operation with the University of Regensburg to optimize the ergonomics of the user interface
- data entry for COST and FUSION programmes
- integration of evaluation programs and extension of Microsoft Excel for Windows macros for graphical data presentation
- development of a PC-based user interface for Aluminium materials data

Information Centre

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

In 1993 the following activities were carried out:

- Organization, preparation and reporting on the Institute Programme Progress Reporting Scheme. Each of the Institute projects under the Specific, Exploratory and Support of the Commission were assessed by a panel of Senior Staff against a detailed programme of work set down earlier in the year. The report on this action will help to define the future direction of activities within the Institute.
- Organization and preparation for the Institute Visiting Group. At the request of the JRC Board of Governors external Visiting Groups were set up to evaluate JRC programmes. The IAM evaluation took place in December 1993.
  The Institute 1992-1993 activities were assessed against the programme objectives and the resources utilized. Details of the evaluation are given in the introduction to this annual report.
- Preparation and publication of issues 3 and 4 of the Institute news bulletin, the "Materials Challenge". This publication has been well received both inside and outside the Institute and forms an essential part of the Institute publicity material.
- Organization of the international workshop on "Materials for Coal Gasification". This meeting was held in Petten in June 1993 and was attended by more than 60 delegates from Europe, North America and Japan. It brought together materials scientists, plant operators and designers at the time that the first commercial plant came in to operation in the Netherlands.
- Organization of the IAM internal colloquium on Surface Modification. These meetings provide a forum for the consideration of major fields of activity in the Institute, with a view to coordination of research activities towards common objectives and also to provide an outlet for the expression of new ideas.

2. Contribution to the Specific Programme: Controlled Thermonuclear Fusion

Research Area: Fusion Materials

Assessment of Research Area Progress and Achievement: Fusion Materials

The worldwide development of fusion technology is currently concentrating on the design of the International Thermonuclear Experimental Reactor (ITER). The initial purpose of this design effort is to define tasks which will eventually fill gaps in the information base. The IAM has contributed to this effort in 1993 and it is expected that this work will play an increasingly important role in the coming years.

In parallel with this development, and as an extension to the NET activities, the Institute is also looking beyond ITER by participating in the EU long term programme.

For the ITER/NET activities IAM has been contributing by establishing the European Fusion Materials Data Bank, by the experiments on thermal fatigue and the blanket studies. The data bank is taking shape in the sense that the quantity of data is constantly growing and it has already been possible to start with the evaluation of the results delivered by the different laboratories.
To date, the work has concentrated on stainless steel AISI 316. Collaboration with the EU laboratories is of course an essential feature of this exercise.

Progress on the thermal fatigue work was determined by the delivery of test components from the industrial suppliers.
The long term activity has been directed towards the development of low activation materials.

During 1993 an extensive campaign on He implantation in SiC/SiC has been successfully completed. Even at large He concentrations, the changes in geometry and properties of the material seem negligible.
This implies that one of the fundamental questions on the use of these materials has been answered positively.

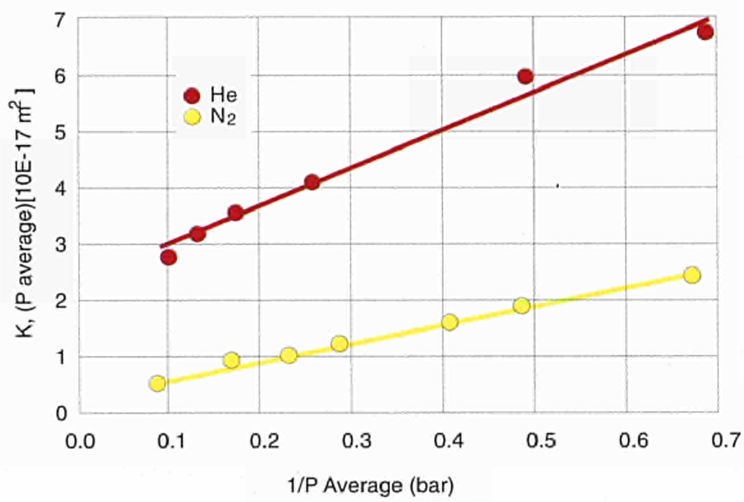
Low Activation Materials

Ceramic Composites

Gas leak tightness is an important issue for fusion reactor structural materials. If CMCs are to function as a pressure boundary, the coolant leak rate into the plasma and the tritium leak rate out of the blanket will be a major design criterion.

Measurements of leak rate have been made on SiC/SiC tubes with an internal diameter of 15 mm and 2 mm wall thickness. Pressure was applied from an external reservoir, and measured inside the test sample in function of time, in order to evaluate the leak rate.

The preliminary results (figure above) show that at room temperature and with an internal pressure of 6 bar of Nitrogen the leak rate is 2.10^3l h^{-1} in samples which were mechanically machined. The leak rate is not measurable or is of the order of 0.5l h^{-1} maximum when using samples with a sealing coat from an additional CVI process. Using helium, the leak rate increases by a factor from 3 to 6, whilst if the temperature is increasing from room temperature to 573K a reduction by a factor 2 in leak rate is observed.



Above: *Room temperature permeability of N_2(•) and He(*) in SiC/SiC tubes*

As part of the long term European programme on Low Activation Materials, the compatibility of the solid breeder candidates $LiAlO_2$, Li_2ZrO_3 and Li_4SiO_4 with two commercially available SiC/SiC composites was investigated. The first material was 1D-reinforced with a SiC matrix formed from a wet impregnation technique with subsequent pyrolysis, whereas the second was a CVI 2D composite.

The ceramic breeders $LiAlO_2$ and Li_2ZrO_3 (CEA Saclay) and the Li_4SiO_4 (KfK Karlsruhe) were supplied in the form of granular material, ranging in grain size from 70µm to 400µm. Simple experiments, in which the SiC/SiC composites (14 mm x 6 mm) stayed in contact with the ceramic breeders, were performed in closed capsules, dynamic vacuum and flowing helium at 873K and 1073K for 1000h.

The capsules were heated in an air oven, the other tests were carried out in a special apparatus. The results from the closed capsule tests show behaviour of Li_4SiO_4 different from that of $LiAlO_2$ and Li_2ZrO_3. It can be seen from SEM micrographs that extensive crystal growth had occurred on the composite surface in contact with the Li_4SiO_4 but not in the $LiAlO_2$ and Li_2ZrO_3 tests. However, glancing angle X-ray diffraction analysis indicated that the reaction product Li_2SiO_3 was present on the surface of each composite. Only the extent of Li_2SiO_3 formation differed, with the Li_4SiO_4 breeder producing the most while the $LiAlO_2$ breeder produced the least. The DG of reaction for the interaction of SiO_2 with the three breeder materials indicates that all the reactions are favourable, although the reaction of $LiAlO_2$ is much less exothermic.

Previous tests -though not in a fusion reactor atmosphere - have shown that the stability of SiC depends on the formation of a passive layer of SiO_2. From these results the "passive" oxidation of SiC to SiO_2 could greatly increase the generation of Li_2SiO_3. The formation of He in SiC/SiC by transmutation reactions due to fast neutrons is currently simulated by irradiations at the JRC Ispra-Cyclotron. Under helium jet cooling, 37.4 MeV to <1 MeV α particles were implanted in industrial CMC specimens up to high fluences (2500 appm in 25 and 50 mm^3). First results show no disintegration and no helium-induced swelling of specimens irradiated at

1023 ±100 K. Analysis of whether the inherent residual porosity of this type of material is an advantage is ongoing. The effect of possible microcracking of the matrix due to formation of neutron-radiation induced gas bubbles should not constitute failure of CMCs, however, it could limit the engineering design load. Meanwhile, 2D SiC/SiC specimens were sent to HFR Petten for neutron irradiation at different fluences at 973 K. Mechanical tests after these irradiation's are foreseen.

Non Ferrous Alloys

The ductile to brittle transition as observed in many body centred cubic materials plays an important role in candidate low activation materials such as pure chromium and chromium alloys.
Several plates of three alloys have been procured:

- Cr-Fe (2500 ppm) DUCROPUR
- Cr-Fe (5%)-Y_2O_3
- Cr-Fe (44%)-Al_2O_3(5%)-Y_2O_3(5%)-TiO_2(3%)

These materials have been characterized up to 1000°C by the manufacturer: tensile properties, hardness, creep rupture, DBTT. Specimens for tensile and fracture mechanics tests are being prepared. The experimental programme is accompanied by theoretical studies investigating the effect of dislocation dynamics on the ductile to brittle transition.

Irradiation Creep of 316L and Welds

Exp. E167-80

Low temperature irradiation of reference steels, 316L, AMCR, and US 316 at approx. 370 K are performed with stresses of 70 and 100 MPa.
Measurements of the elongation were performed after 0.1, 0.4 and 0.9 dpa. Measurements of the elongation after 1.5 dpa will be performed before the end of 1993.

Exp. E167-90

Low temperature irradiations on welded specimens, namely on EB welds and TIG materials started with the reactor cycle 93.11 on December 17th, 1993. These low temperature irradiation's are performed in channel H8 in which doses of 0.12 dpa are obtained during one reactor cycle, i.e. after 26 days. EB-materials will be irradiated only in the as-received state. TIG materials will be irradiated in the as-received state, and after annealing at 800°C prior to irradiation. The E167-90 rig also contains specimens of 316L steels and of pure AMCR type materials with certain additions.
Stresses of 70 and 100 MPa are used in this irradiation run.

Exp.E167-100

The preparation of this rig were finished at the end of January 1993 and irradiations will start during the first half of 1994 depending on the availability of reactor positions.
High temperature irradiations will be performed on 49 steel specimens for irradiation temperatures ranging between 340 and 400°C and for stresses of 70 and 100 MPa.
All EB weld specimens will be irradiated in the as-received state. TIG-weld specimens will be irradiated in the as-received state and after annealing at 800°C prior to irradiation.
This irradiation rig also contains 316L and AMCR 0033 steel specimens in the solution annealed state.

In-Beam Creep-Fatigue of 316L Stainless Steel

The Effect of 19 MeV deuteron irradiation on the fatigue properties of 20% cold-worked type 316L stainless steel has been studied for in-beam and for post-irradiation conditions. The conclusions of this study are:

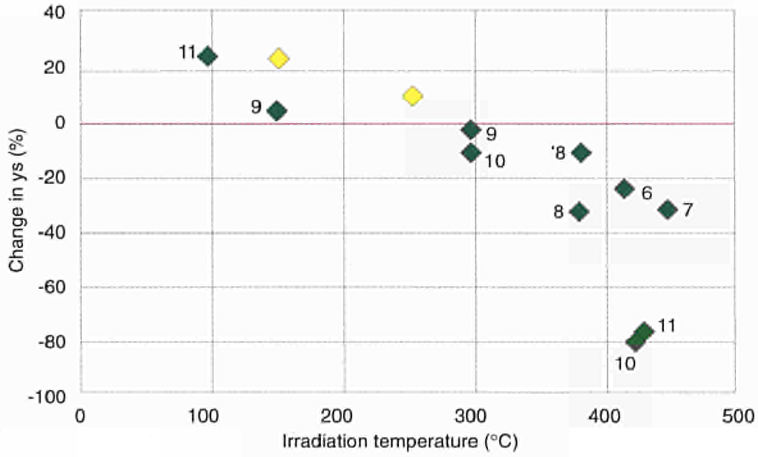
- In the High Cycle Fatigue range, the irradiation leads to an extension in fatigue life.
  a. by a factor of 6 for cycling under post-irradiation conditions
  b. by a factor of 12 for in-beam cycling
  c. the crack growth rate is equal for both conditions
- In the Low Cycle Fatigue range, by imposing a hold-time in the loading cycle, the creep induced stress relaxation leads to a drastic reduction of N_f for shear strain ranges less than 1.5%.
- The observed shift of the hysteresis loop on the stress axis may have negative effects for the fatigue life of the first wall, due to a transition from a compressive mean stress to a tensile mean stress.

Effect of Fast Neutron Irradiation on Tensile Properties of Precipitation Hardened Cu-Cr-Zr Alloy

High strength, high conductivity copper alloys are suitable heat sink materials for the divertor of magnetically confined fusion reactors subject to high thermal loads.
Tensile specimens of a commercial high strength Cu, Cr, Zr alloy were irradiated in the HFR reactor in Petten at 150 and 250°C. The total accumulated damage (10 dpa) and the damage production rate (3.3×10^{-7} dpa s^{-1}) correspond to the envisaged operating requirements of the ITER divertor.

Tensile testing after irradiation was performed in the LMA hot laboratory at JRC Ispra, at a single constant strain rate of 2.9×10^{-4} s^{-1}. At 155°C an increase of Yield Strength (YS) was measured and a decrease of Uniform Elongation (UE) from 5% to virtually zero. Persisting high Total Elongation (TE) values of about 9% indicate that the ductile fracture mode is not affected.

Irradiation hardening at 255°C is less pronounced. In the light of this study and of published results, the temperature of 275°C is proposed as a conservative temperature limit for use of fully precipitation hardened Cu-Cr-Zr (Mg) alloys in order to exclude irradiation softening (figure above).



Above: *Changes in yield strength of Cu-Cr-Zr-(Mg) PH alloys after various fast neutron irradiations reported in literature, indicated by numbers, are plotted against irradiation temperature to show the transition from irradiation hardening to irradiation softening. Open dots are for present HFR irradiations*

Thermal Fatigue

Thermal Fatigue Tests on First Wall Specimens

IAEA Benchmark on Lifetime Evaluation

The first phase of the IAEA Co-ordinated Research Programme (CRP) on "Lifetime prediction of the first wall of fusion machines" was completed. Its main objectives were the validation of FEM numerical methods and design codes for predicting the FW lifetime. The main results have been the following: (a) there is a strong sensitivity of the computed stress to the numerical procedure adopted and (b) different interpretations of standard codes lead to a large scatter in the evaluation of the maximum allowable number of cycles.

During 1993 the draft final report on the activities was written, it will be published as an IAEA TECDOC Report.
The basis for the second phase of the IAEA CRP has been settled. The reference component geometry was defined and manufacturing was completed. This phase is aimed at comparing the crack growth rate under thermal fatigue conditions with theoretical and numerical predictions.

Functional Validation of Prototype FW Mock-ups

Testing of two medium-scale prototype components was stopped after about 5000 thermal fatigue cycles because of water leakage from the

Ansaldo component. The NDT of both components was completed and the final report is being edited. The results obtained have given the following indications. A crack from the plate-to-plate braze started from one edge of the component and propagated through the braze until it reached the brazing of the cooling tube. Then it continued circumferentially around the tube and, at a distance of about 5 cm from the component end, penetrated the tube wall. The NDT on the Framatome component, performed by Framatome itself, has given indication of two parallel cracks running along both sides of the central electron beam weld at a distance of 5 mm from one to the other. The detected lengths were 197 and 22 mm, respectively.

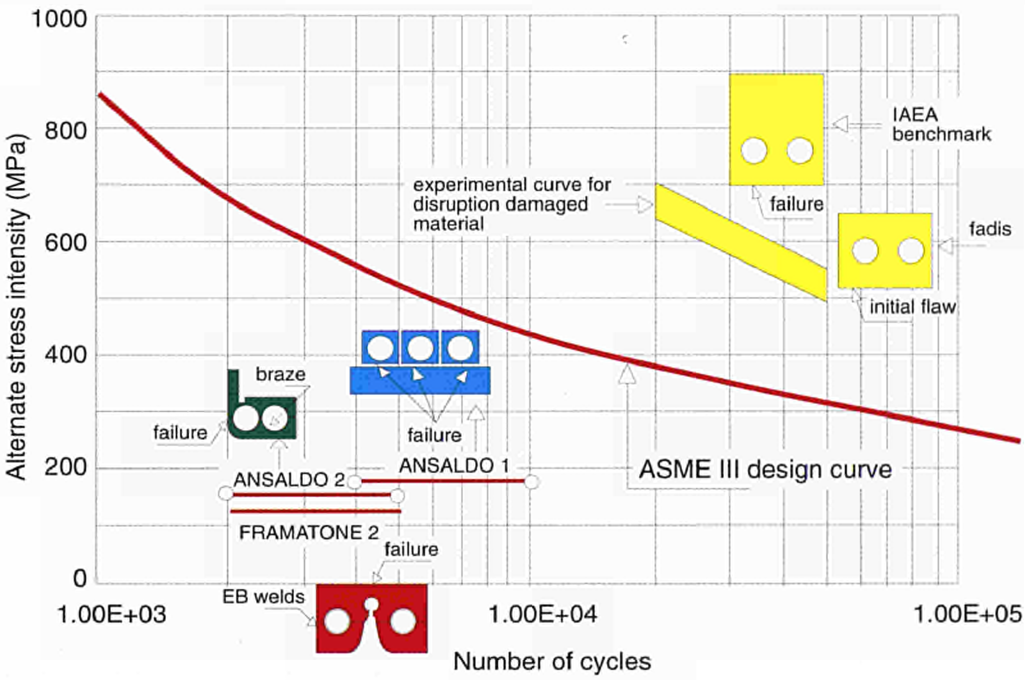
Below: *Experimental results of components subjected to thermal fatigue cycles. The figure gives the location in which failure actually occured and the experimental number of cycles to failure for each component. Numbers of cycles are given as a range, reflecting the uncertainly in defining failure. Therfore prototypical components are represented by a segment and simple component by the picture of the component itself located in correspondence to the scatter of the computed alternate stress intensity*

Interaction between Thermal Fatigue and Plasma Disruption

This experimental study was aimed at evaluation of the synergistic effects of thermal fatigue and plasma disruption induced cracks on a bare FW. The thermal effects of a plasma disruption were simulated by means of an electron beam gun. Three damaged components (Fadis) were then thermal fatigue cycled. In the experimental loading conditions, the ASME code envisaged for an unflawed component, a maximum allowed number of cycles of about 4000 in the spot location. After 50000 thermal fatigue cycles none of these components showed any evidence of crack propagation. At present the test is stopped and destructive examination is under way.

Contribution to Regulation and Standardization

All the thermal fatigue activities are aimed at assessing the possible extension of the presently existing nuclear standard codes to the fusion environment. The main achievements obtained so far are the following: (a) the thermal fatigue behaviour of a FW component without welded or brazed joints can be managed by standard codes with great



safety margins; (b) the application and standard design by analysis procedures may lead to highly unconservative results when applied to prototype components which contain welded or brazed joints.
Figure on page 64 summarizes the experimental results. A support activity for the ITER team is in progress to define the design criteria for the fusion reactor.

Development of New Divertor Concepts

High Thermal Performance CFC Divertor

The main distinguishing feature of this new divertor concept is the use of a single material, a carbon fibre reinforced carbon composite, for the whole structure. This should overcome the shortcomings of the ITER CDA reference design especially as far as the off-normal conditions are concerned. The same concept could also be usefully adopted for the limiter and sub-limiter to protect the plasma facing components. During 1993, a collaboration with the SEP company was established aimed at defining a suitable geometry which optimizes heat removal capability and ease of fabrication.
Several coolants have been investigated such as helium, organic fluids and liquid metals.
A scrutiny of the different options was completed. A study on the possible joining of CFC started in collaboration with the Polytechnic of Turin. The first results obtained consist in selection of joining materials to comply with the low-activation requirements and with joining needs (i.e. thermal expansion matching, wettability and chemical stability). They include mainly pure elements (i.e. Si, Ti, etc.) and glass-ceramic materials (borosilicates, leadborates).

Development of the "Brush" Divertor concept

This concept consists of copper matrix, tungsten fibre composite plate with fibres protruding out of the matrix facing the plasma. This configuration is expected to exhibit enhanced properties of thermal fatigue resistance since propagation should be prevented by the non-continuous interface between the plasma facing surface and the cooling channel. Plasma disruption heat deposition has been simulated by means of an electron beam gun. The results obtained showed that the fibrous surface is effective in increasing the thermal shock resistance.

Blanket Studies

Development of an Oxide Based H_2 Permeation and Corrosion Barrier

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 Pb17Li.
The study to determine the oxidation conditions necessary to form reproducible Cr-Mn oxide layers (as permeation barriers) on 316L stainless steels have been completed.
Due to the high temperature used in the oxidation of the steel, this process cannot be used for MANET steels. Therefore, aluminide layers produced on the surface by low temperature thermal treatment following the plasma spraying of pure aluminium have been investigated.
Aluminide layers on MANET have been produced while still retaining the mechanical properties of the steel. H_2 permeation measurements have been completed in the ETHEL laboratory, showing a two-three orders of magnitude reduction in the hydrogen permeation rate.

LME Susceptibility of Welded MANET

The constant load tests of MANET I and MANET II at 80% of UTS in Pb-17Li, Pb-17Li/H_2, H_2 and Argon at 450^{0}C are completed. Constant load tests at 70% of UTS are still continuing. Experimental difficulties delayed the tests. The constant load tests on welded MANET II have consequently been delayed. LME tests were performed on welded MANET II specimens, perpendicular to the welding direction in Pb-17Li and vacuum.
No LME effect was observed. Tests on specimens manufactured in the welding direction (welding and H;A;Z;) are close to completion.

The study of the LME effect in function of postweld heat treatments could not be performed because the welding manufacturer (Mannesmann, Dortmund) made a post weld heat treatment (750^{0}C, 4 hrs in air) of the welded plates, in contrast with the agreement with KfK and Interatom/ Siemens.

Fusion Materials Data Bank

The data bank has been equipped with the necessary facility for the digitization of older measurements, which are only available in graphical form, as, for example, tensile curves.
The data input has started for AISI 316, and at the end of 1993 already 110 tensile curves and 37 in reactor creep curves had been introduced in the data bank. In order to assure sufficient data flow to the ITER data handbook, the evaluation of the data had to be postponed.

3. Supplementary Programme:

- Irradiation Experiments in the High Flux Reactor Petten

Operation of the High Flux Reactor

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 research and services for third parties. In this chapter only the projects for German and Dutch institutions are addressed, whereas the specific programme activities are covered in other chapters of this report.

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

The irradiation programmes on light water reactor fuel were continued.
These programmes were focused on high burn-up MOX and UO_2 containing PWR fuel rod segments which had been pre-irradiated for several years in commercial power reactors. Increasing use is being made of a newly developed irradiation device which provides typical LWR fuel rod surface temperatures already at the low linear heat generation rates of high burn-up fuel (at about 150 W/cm).
As a valuable addition to the facilities available in the HFR pool for intermediate non-destructive investigation, a new technique and procedure for measuring and evaluating the diameter and eddy current profiles, derived from underwater measurements on irradiated LWR fuel rods, was developed and successfully introduced. Preparations were initiated for a new irradiation project on irradiation testing of fresh LWR fuel rods with various types of boron coating on the pellets. Preparative actions were taken for irradiation testing of refabricated high burn-up PWR fuel rods.

The irradiation programmes related to the high temperature gascooled reactor, comprising graphite and fuel irradiations, are close to their termination as a consequence of the termination of R&D on this reactor line in Germany.

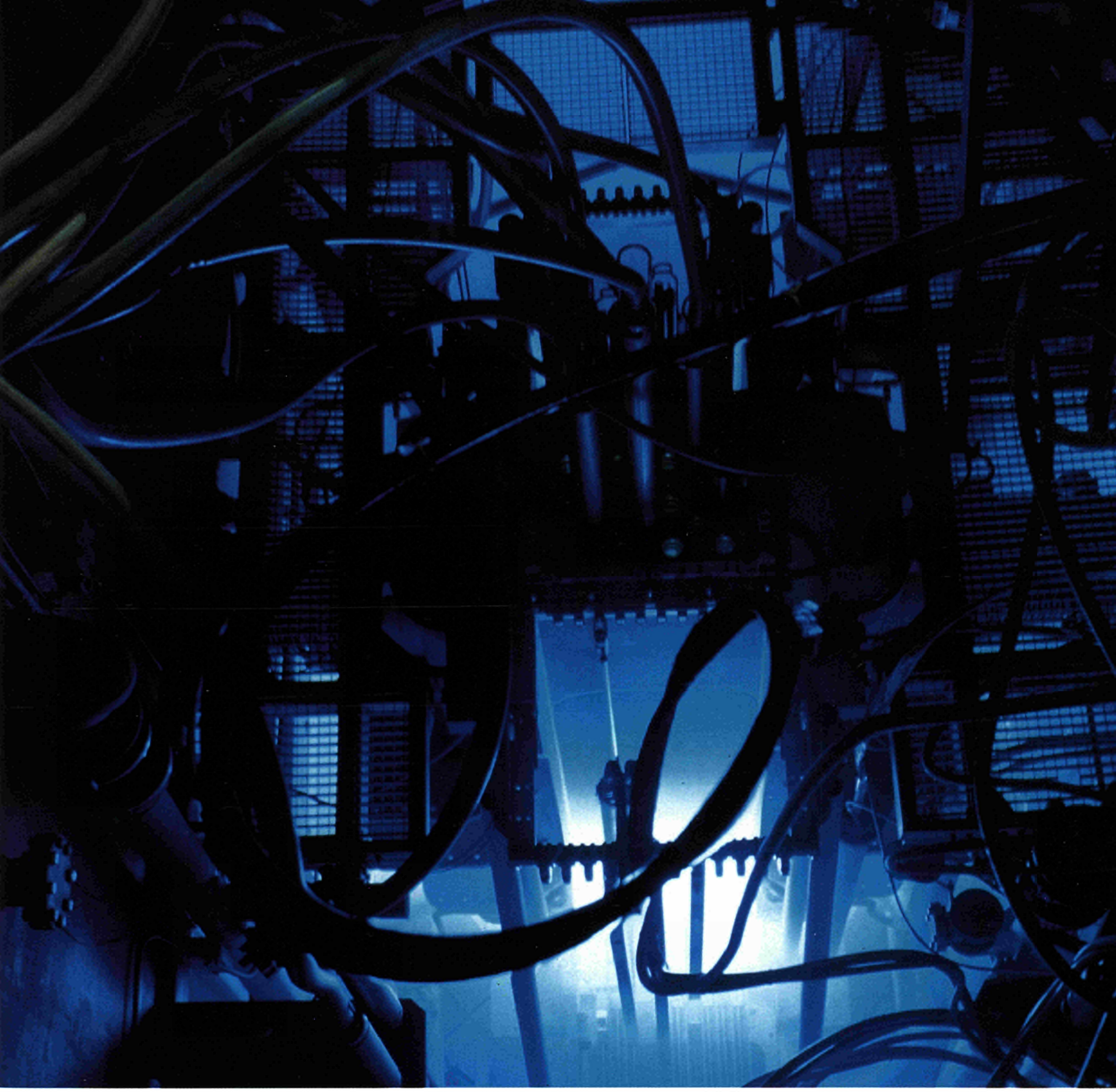
Ongoing projects have, however, been continued to schedule. Presently still three individual irradiations within the "fundamental properties graphite programme" are either being irradiated or being ready for irradiation. In the graphite creep programme one irradiation campaign is still planned. Within the HTR fuel irradiation programme, a full-size proof test of spherical fuel elements was successfully terminated after an operation time of 634 full-power days at operation conditions characteristic for an HTR-MODUL, while the last test in this series is still under irradiation.

In view of the phasing out of the R&D related to fast breeder reactors by the end of 1994, the fuel pin irradiations in the HFR were orientated towards the regular termination of ongoing irradiation projects. As such, the following experiments were performed and completed by the end of 1993. The sixth Power-toMelt experiment, whereby 3 fuel pins containing different quantities of Pu, were brought to overpower conditions, causing central fuel melting; the last 2 out of a series of 5 experiments, whereby during dedicated transients, the axial extension of both the fuel stack and cladding could be measured; and finally, and probably the most innovative and successful, the irradiation of 2 pre-irradiated POUSSIX fuel pins (having been irradiated in the PHENIX fast reactor; transported to Petten; decontaminated using a specially built electro-chemical facility; encapsulated, sodium-filled and weld-sealed, semi-automatically and remotely in the EUROS-cell; and then irradiated in the HFR).

In particular, the build-up of fission poisons, especially ^{149}Sm, during the irradiation phase in PHENIX, meant that a special pretransient irradiation phase was required whereby, as the ^{149}Sm was burned-up, the capsule was slowly withdrawn on the PSF trolley to maintain constant power. This was subsequently proven and was a considerable success for the HFR and the FBR programme.

KfK's remaining involvement in fast reactor activities, entails preliminary studies into transmutation of long-lived actinides and fission products.
As such an experiment to irradiate 3 fuel pins of different material composition is being designed and prepared for irradiation in the HFR, starting in 1994.

Fusion related investigations comprised irradiation projects for prospective constituent materials for fusion devices, in particular for first wall materials. The programme concentrated on optimized MANET steels. Irradiation on small scale of superconducting materials is continuing. The investigation of irradiation damage and tritium release



Above: *View into the reactor-pool*

kinetics from different ceramic lithium compounds under irradiation have been continued too.

The Boron Neutron Capture Therapy project, under the German contribution to the supplementary programme, continued its development towards its ultimate goal, namely, the treatment of glioma patients by BNCT at the HFR. The irradiation part of the "healthy tissue tolerance study" was completed in July. Thereafter, the facility was dismantled and preparations started to construct a new, enlarged patient treatment room. This part of the project will be completed in early 1994.

A small project in neutron radiography was executed in cooperation with the University of Stuttgart. A single detector tomography system was tested at the HB-8 neutron radiography facility. The objective was to investigate the system performance with regard to high neutron fluence rates and with various selected neutron beam combinations. For the first time in Europe, neutron radiography based tomography using sub-thermal neutrons was performed.

The Netherlands contribution to the HFR supplementary programme is managed via Energieonderzoek Centrum Nederland (ECN).

For the EFR programme, radiation damage studies on structural materials were continued. Compact tension specimens as well as specimens for creep fatigue testing were irradiated.

Within the fusion programme, martensitic steels, austenitic steels and vanadium alloys have been irradiated at different temperature and fluence levels. New projects are running to study the particular effect of neutron spectrum on irradiation induced property changes mainly on fracture toughness and charpy specimens. Emphasis is also on the effects of helium embrittlement.

The programme on irradiation testing of tritium breeding ceramic blanket materials, where ECN is co-operating with a number of European research centres, has continued with tritium release investigations under radiation for different zirconates, aluminates and silicates. In addition the influence of EXOTIC-7 was continued where radiation damage phenomena are 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 applicataions. 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 transumtation of the long half-life fission products ^{99}Tc and ^{129}I, 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.

With regard to reactor operation, the scheduled 11 operation cycles have been executed nearly to schedule. In order to avoid disturbances in radioisotope supply to the European radiopharmaceutical industry, the July/August cycle had to be rearranged. In total 279 operation days were achieved with not more than 8 unscheduled short interruptions of regular operation. The safety record was excellent with no significant incidents at all. Also, the irradiation dose to operation staff was kept at a very low level again.

Detailed information on the HFR programme, operation and well as utilization, can be taken from the Annual Report 1993: Operation of the High Flux Reactor, EUR 15219 EN

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

Standards for Advanced Ceramics

This project, in support to DG III Industry, deals with three types of activities. The first consists of contributing to and providing input to European (pre-) standardisation actions. In this area, IAM is actively involved in drafting standards for three working groups of CEN Technical Committee 184 on advanced technical ceramics dealing with terminology and classification, with test methods for monolithic ceramics, and with test methods for ceramic matrix composites. Another activity in this same area, launched in 1993, is the establishment of an IAM-led working group on ceramic corrosion test methodology.

The second part of the project consists of performing prenormative research into mechanical and corrosion test methods for ceramics. This covers both in-house projects in these fields, as well as participation to internationally organised round robin exercises aimed at the performance characterisation and validation of proposed standards or codes of practice. During the reporting period, two VAMAS RRs were participated to: fracture toughness determination at high temperature, and fractography. Also, a joint action with NPL aimed at the quantification of intrinsic, intralaboratory, and inter-laboratory scatter in uniaxial tensile and creep testing of a hipped silicon nitride was launched.

The third activity consists of the organisation of workshops and meetings dealing with standardisation of ceramics.
In 1993, the first in a series of workshops on test methodology development for high temperature corrosion of ceramics was held with participants from a number of member states.

Standards NDT for Pressure Vessels

Introduction
International programmes addressing the integrity of (nuclear) structural components have been very successful in the areas of inspection (DISC) and in fracture mechanics (FALSER). There is a growing recognition that such structural integrity assessment should not be considered in isolation from each other due to their irrevocable interdependence. Structural assessment is indeed an interdisciplinary process which takes on board not only inspection and fracture mechanics but also materials and engineering evaluation along with risk appraisal. Such contributions can ideally be achieved through the networking of organisations in a mutually beneficial and cost effective manner. NESC professes a framework upon which a variety of structural integrity assessment projects can be supported and brought to fruition.

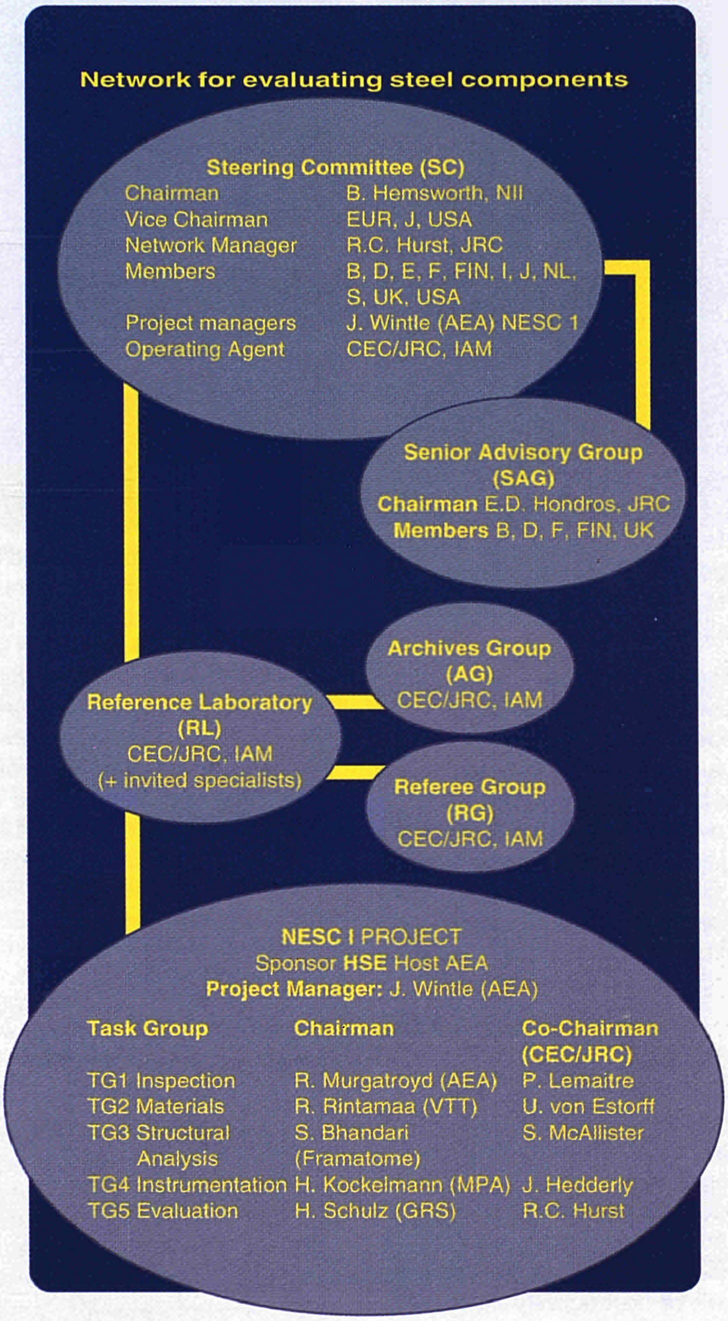
Objectives
The Network objective is to organise and manage an international network of organisations around experimental programmes which will:
- enable information to be exchanged about the entire process of structur integrity assessment
- stimulate the undertaking of specific collaborative studies
- work towards the use of best practice and support the harmonisation of international standards

Organisation and structure

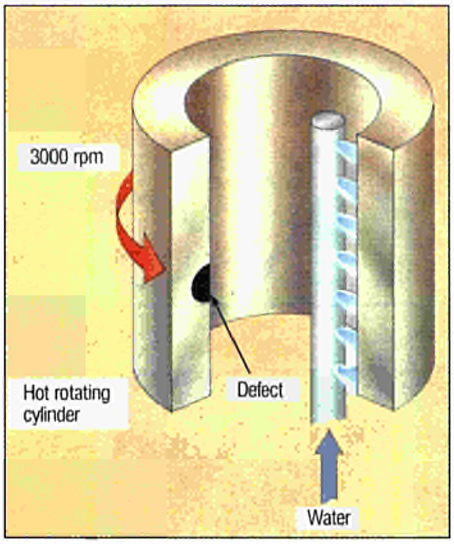
The structure of NESC is centred around the JRC/IAM as Operating Agent and Reference Laboratory with a Steering Committee elected from member organisations emanating principally from EU and EFTA countries (figure on page 77).
The seeds of NESC were sown in the Spring of 1993 and the Network officially launched in September at the Health and Safety Executive offices in Sheffield, U.K. At that time the funding relied mainly on the sponsors, HSE and the CEC, with Network Members offering contributions in kind. Most of the resources to date have been directed towards the support of the first NESC project, NESC 1.

NESC 1 - The Spinning Cylinder Experiment

The aim of the first project executed within the frame work of NESC, is to study the entire process of structural integrity assessment of under clad cracks in PWR vessel steels subjected to conditions



Above: *Network structure*



Above: *Spinning Cylinder Facility*

of pressurised thermal shock (PTS). In the experiment itself, a stainless steel clad, nuclear quality A508B ferritic, thick walled cylinder will be prepared containing both technologically relevant defects as well as defects of such a size that they may reasonably be expected to grow by cleavage during the experiment. Both surface breaking and under clad defects will be introduced.

They will be detected and evaluated by an Inspection Task Group comprising teams practising current in service inspection techniques. The results will be fed to a further Structural Analysis Task Group of structural integrity assessment teams. On the basis of data covering the properties of the forging, the cladding and the heat affected zone provided by the Task Group for Materials, the structural analysts will calculate the potential for cleavage crack growth from these defects under the conditions of the Spinning Cylinder Experiment.

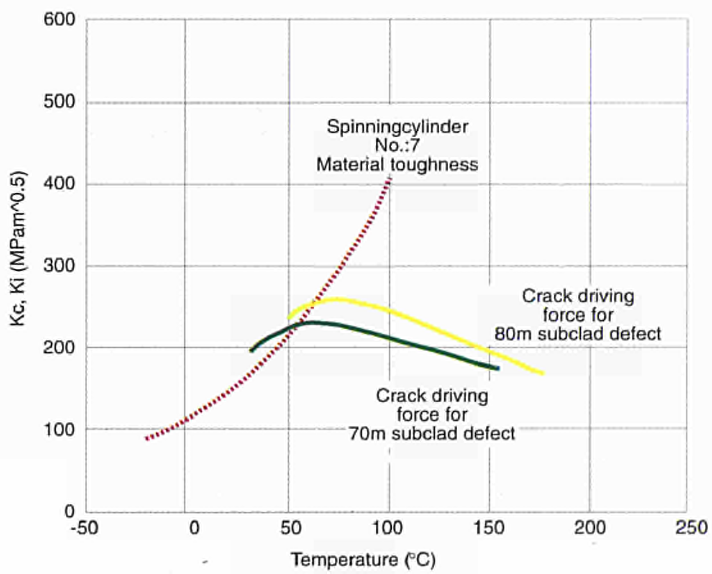
The PTS will be simulated by spinning the cylinder at some 2500 rpm at an initial temperature of 230^{0}C to generate primary hoop stresses followed by a sharp internal water quench (figure above). A special Task Group has been created to address the complex instrumentation requirements of the experiment of which the detection of onset of cracking is paramount. After the test a further inspection will be made to re-size the defects, after which the cylinder will be destructively examined to allow a further post test structural analysis and an evaluation of the pre- and post test inspection results. The results of the project are to be addressed by the Evaluation Task Group who will also provide a co-ordination role throughout the project.

Activities within NESC and NESC 1

By the end of this reporting period, the NESC Steering Committee comprised some 15 Members, including the nominated Officers, and 25 organisations were participating in the various Task Groups, each under nominated Chairmen and Co-chairmen. In addition to the launching of the NESC 1 project, a call for proposals for successive projects had been issued with a view to commencing NESC 2 in late 1994 or early 1995.

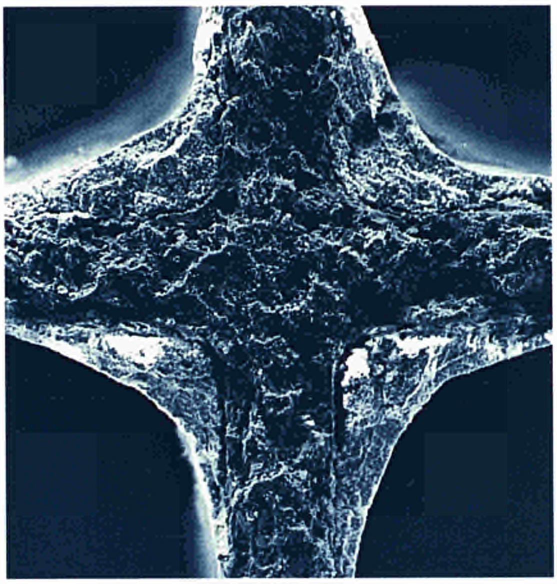
The total effort of the NESC 1 project was concentrated on the selection of a forged cylinder of the required metallurgical properties which would allow a defect to grow in cleavage under the Spinning Cylinder test conditions. This action involved the Materials Task Group in making recommendations regarding thermal treatments of the forging and determining the ensuing relevant mechanical properties. The Structural Analysis Task Group carried out various simplified and finite element calculations based on this data and, by the end of the year 1993, were concluding that the first cylinder selected might be too tough to facilitate the cleavage event and that an alternative cylinder should be favoured (figure below). Meanwhile, the Inspection Task Group had established a nucleus of interested participants and the Instrumentation Task Group has already identified their list of actions. These actions were achieved in a very short time, through Task Group meetings and the co-ordination of the pronounced self interest of the participating organisations.

Below: *Fracture toughness data for cylinder 7*



Ceramic Catalyst Support

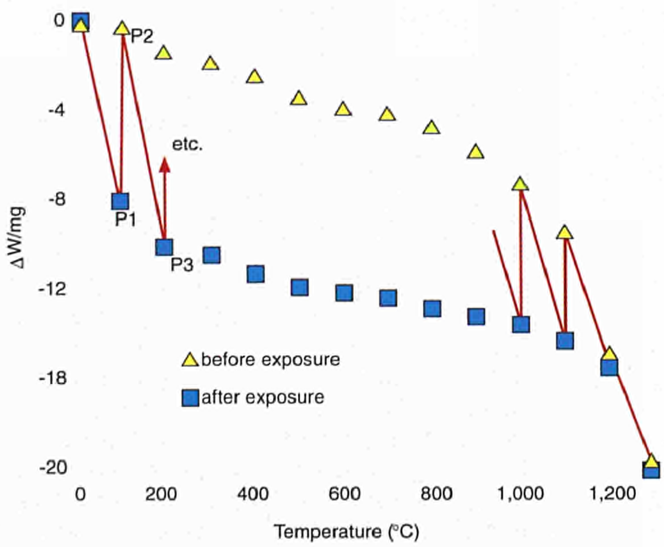
"downstream" —— 100µm



"upstream" —— 100µm

Above: *Migration of washcoat during exposure to SO_2-containing environment in a burner-rig*

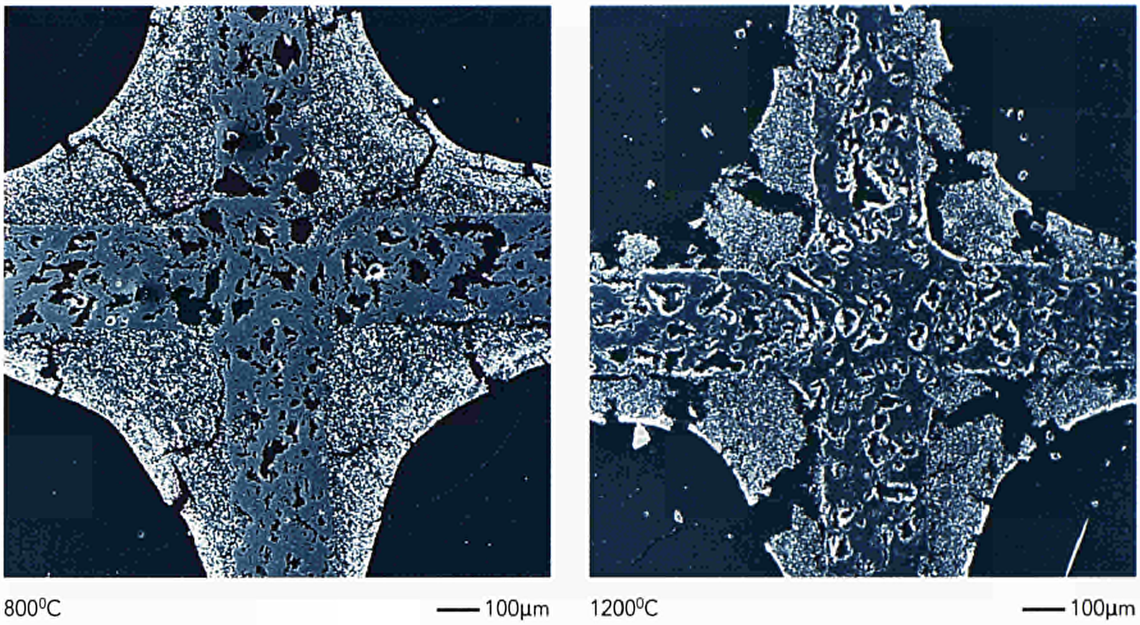
Below: *Hygroscopic behaviour of the γ-alumina based washcoat*



This activity aims to study and evaluate the failure mechanisms of ceramic catalyst support carriers in automobile exhaust systems and is carried out in support of DG XI (Environment), DG III (Internal Market and Industrial Affairs) and DG VII (Transport). The 3-way catalyst system is a very efficient method of reducing environmental pollution in the traffic sector but the long term efficiency is known to be affected by ageing at high mileage.

This failure by ageing has been attributed to corrosion as well as mechanical or thermal fatigue properties of the ceramic carrier (cordierite, $2MgO.2Al_2O_3.5SiO_2$).

Preliminary isothermal studies of corrosion in air containing $1\%SO_2$, to simulate the worst condition when a sulphur containing fuel is used, showed that the cordierite was virtually unaffected by the environment. Similarly, the complete catalyst material (cordierite + washcoat of Al_2O_3/CeO_2 with Pt/Rh) was found to be resistant to the environment at elevated temperatures representing 'overheating' conditions. At 'normal' operating conditions (600^0C), however, $Ce_2(SO_4)_3$ was formed in $1\%SO_2$ which can lead to so-called 'hot corrosion' conditions in which a surface liquid can accelerate corrosive degradation of materials. Indeed, migration of the washcoat layer downstream was observed after exposures in a burner-rig (figure above).



Above: *Damage to washcoat induced by thermal cycling in air to indicated temperature*

Under thermal cycling conditions in air, no degradation of the cordierite structure was observed at temperatures up to 1200⁰C and there was no significant mass change. The catalyst system with washcoat, however, exhibited two notable effects.

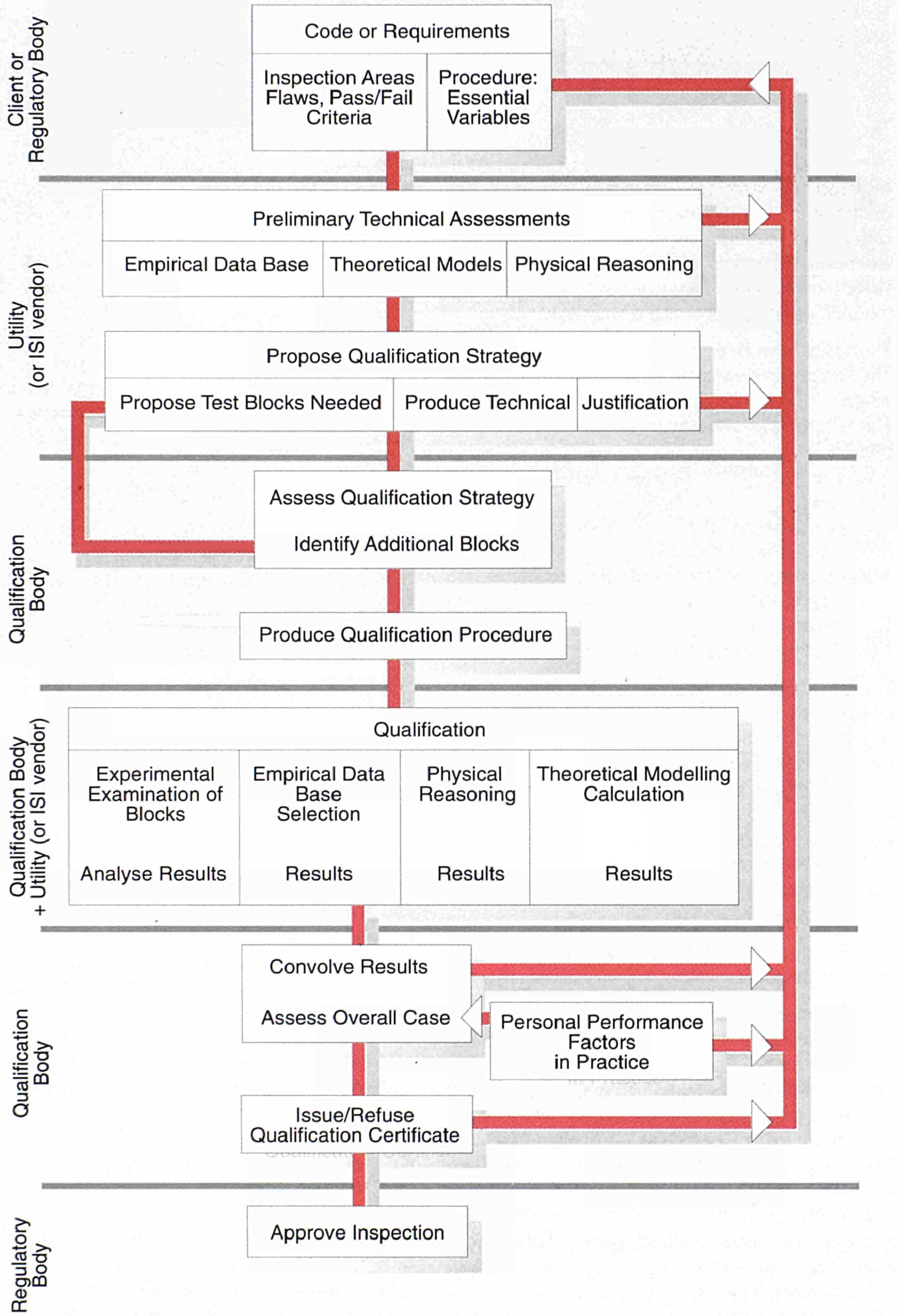
Firstly, there was a reversible "de-hydration" of the γ-alumina washcoat layer (figure on page 79 below) superimposed on a progressive weight loss up to 1100⁰C after which the de-hydration was permanent.

Secondly, 500 cycles to temperatures above 800⁰C show cracking and spallation damage to the washcoat caused by shrinkage of the γ-alumina (figure above). Clearly such damage can be a significant factor in the long term stability and efficiency of the catalyst.

Performance Demonstration of NDE Techniques

The main objective of the European Network for Inspection qualification (ENIQ) is to coordinate and manage 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 NDE techniques and procedures used and on qualification of In Service Inspection (ISI) through performance demonstration and technical justification. The ultimate goal will be the support of international codes and standard bodies by making available the state-of-the-art results, technical tools, expertise and performance/capabilities demonstration exercises that can be sponsored and managed at a European level. As a consequence ENIQ could help in establishing a common European opinion on inspection qualification in general.

The organisation of this network is similar to that of the successful Programme for Inspection of Steel Components (PISC) , closed in December 1993. The Joint Research Center of the European Commission fulfills the role of Operating Agent with the help of national institutions of excellence as



Above: *European Qualification Methodology* required. A Steering Committee provides guidance and recommendations to different task groups, executing specific tasks. The Steering Committee consists of utilities, vendors and research institutions. It is expected that the input of the utilities will become more important.

The tasks have been subdivided in three groups. The first group deals with gathering of information, which can be of interest for inspection qualification. The following priorities were determined (September 1993):

1.1 The correlation between real and realistic flaws
1.2 An inventory at European level of available assemblies and blocks
1.3 Application of human factors to inspection qualification
1.4 Reliability study
1.5 Structural integrity significance of flaws

The information gathered within the first group of tasks can be used within the second group of tasks where qualification schemes are studied. Three tasks are identified:

2.1 qualification of in-service-inspection (ISI) procedures
2.2 development of qualification procedures for specific reactor components
2.3 development of a simulator for ultrasonic inspections

Task 2.1 deals with general qualification schemes for ISI procedures.

Task 2.2 has reached the furthest state of development. The objective of this task is to set out guidelines and options in the form of a "Recommended Practice" for the inspection qualification of each component to facilitate the adoption of the optimum route for any particular set of circumstances. The contents of the Recommended Practice are summarised hereafter:

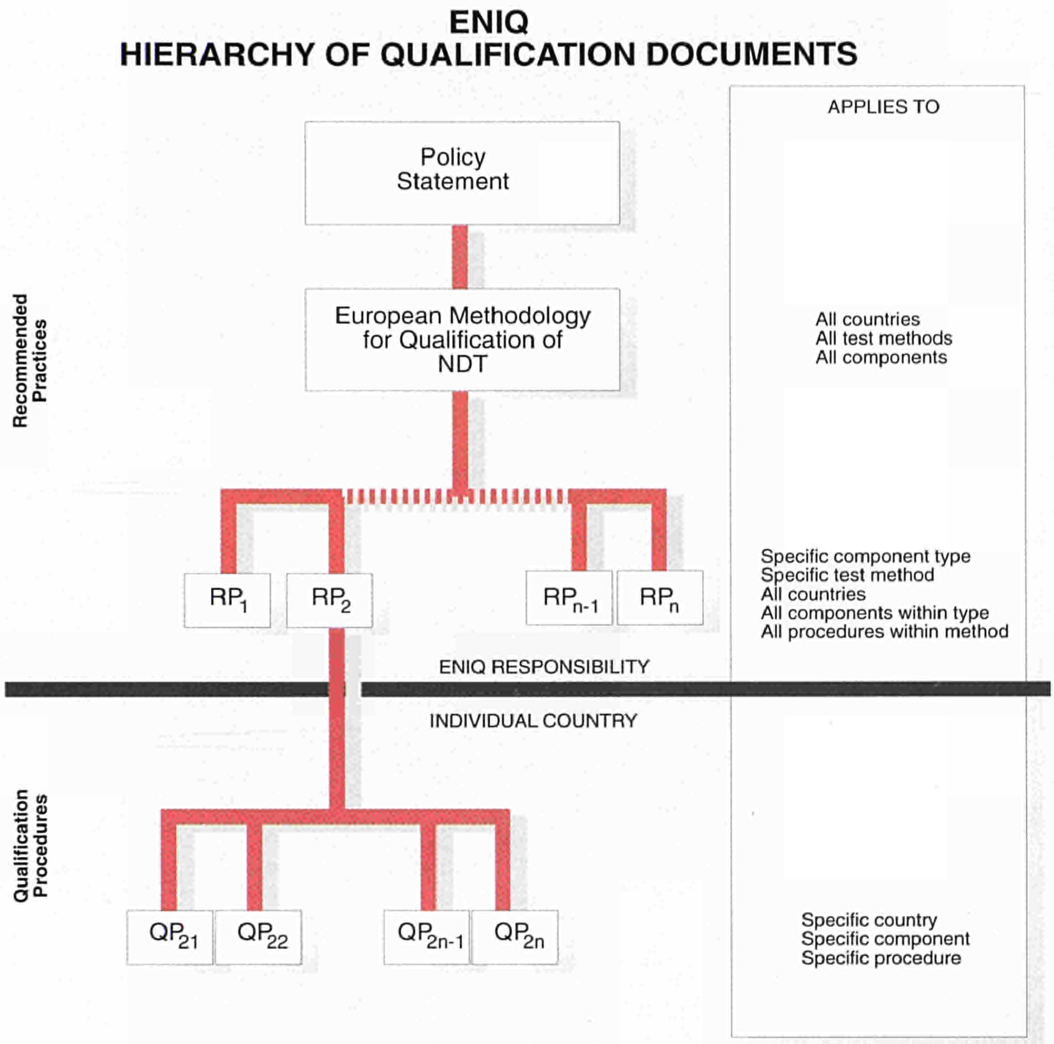
- qualification dossier
- overall approach qualification (role of blind trials)
- technical justification:
  * physical reasoning
  * field experience
  * previous qualifications (where available)
  * relevant round robin trials
  * mathematical models (where available)
  * laboratory studies (where relevant)
- certificates: duration
- conduct:
  * time available
  * conditions
  * practice and training

The Recommended Practice will consist of the appropriate mix of test piece trials and Technical Justification comprising experimental evidence, theoretical arguments, field experience and mathematical modelling to make a case for effective NDT.
Five components were selected for pilot studies:

nozzle-to-shell weld
austenitic piping
ferritic piping
steam generator piping
sub-cladding zone

These Recommended Practices will be developed along the lines of a general policy document, describing a European methodology for qualification of NDT.
The document is of a very general nature dealing with a number of important issues such as the items for agreement between the different parties involved in qualification (utility , qualification body, vendor and regulatory body), the objectives of qualification, the NDT qualification methods, the different qualification approaches, the qualification certificates and the conduct of qualification.
The figure on page 81 gives a schematic overview of the European methodology. This document was discussed extensively at the different meetings of ENIQ and there is a growing consensus of opinion on it.
It is expected that it will be accepted soon by most involved.

The figure on page 83 shows the hierarchy of the qualification documents.
The contribution of ENIQ will hence be to reproduce Recommended Practices which the individual countries can use to develop their own national qualification procedures.
In this way it is hoped to harmonise the different national qualification approaches.



Above: *Hierarchy of qualification documents*

The third group of tasks is concerned with application of knowledge acquired within the two other groups of tasks. The priorities as determined by the Steering Committee in September 1993 are given below:

3.1 Management scheme at a European level of resources available
3.2 Development of accreditation criteria
3.3 Cooperation with Russia in view of developing qualification schemes
3.4 Reporting on cooperation with Russia (hardware installation)
3.5 Reporting on cooperation with Central Europe

In 1994, emphasis will be put on the development of these Recommended Practices for the selected components.

The basic parameters for inspection qualification of the selected components will thus be established.

Furthermore a catalogue of defects will be prepared for each component and Handbooks of Evidence on relevant full scale trials, experimental evidence, theoretical modelling and returns from field experience will be written.

All this information will then be used in the Recommended Practice.

Materials and Inspection Evaluation

The European Action Group on RPV Materials Irradiation Effects and Studies (AMES) was set up officially in 1993 after a first meetings in 1992. Indeed with the maturing and rationalisation of nuclear technology worldwide there is a perceived increase in cooperative activity in this area.

Information about the objectives, the major tasks, the Steering Committee members and the officers of AMES can be found in table below.

The Operating Agent of AMES is the IAM (JRC of CEC).

Table below: *General information about AMES*

Objectives

- Validation and establishment of safe limits for irradiation effects, mitigation and amelioration measures (annealing, etc.);
- Formulation of a microstructurally based model capable of predicting the effect of irradiation, annealing and re-irradiation;
- Validation of the application of novel techniques, including reconstituting specimens, miniature and in-situ mechanical test procedures and also advanced microstructural techniques to RPV condition assessment for long term use;
- Act as a European Review Group for this subject area;
- Maintenance of a European capability for RPV condition assessment and remedial action;
- Participation in collaborative programmes with organisations in the former Soviet Union and Eastern Europe;
- Advice to regulatory bodies and provision of a base for development of common European Standards.

Major Tasks

The network will include the following range of activities on material studies and expertise:

- Review the capabilities within its member organisations together with the existing knowledge base from previous work programmes.
- Studies on components other than the Reactor Pressure Vessel e.g. core internals.
- Assess the availability of irradiated and unirradiated materials that might be made available for work programmes as well as material that might be recovered from operating or decommissioned reactors.
- Studies on model alloys to improve the understanding of the underlying effects for irradiation damage, thermal ageing and annealing.
- Annealing validation and re-irradiation studies on materials of current interest for LWR (Light Water Reactor) systems in Europe and the former Soviet Union.
- Development of microstructural models of irradiation damage, thermal ageing and annealing.
- Studies on new materials other than those used in the old power plants.
- Studies on irradiation and thermal degradation of materials for a new generation of reactors.
- Survey of national Regulatory Requirements and identification of existing, planned and required Standards at European level relevant to material damage and mitigation methods

These tasks will be allocated to the following groups:

Task I: Evaluation of mitigation methods of irradiation damage

Task 2: Survey of national regulatory requirements

Task 3: Identification of existing, planned and required standards at a European level (relevant to irradiation damage and mitigation methods)

Task 4: Harmonisation of rules for defining RPV material condition (with the aim of reevaluation safety margins where possible)

Steering Committee

AEA Technology and NE (UK); TRACTEBEL and CEN/SCK (B); MPA Stuttgart and SIEMENS (D); CEA and EDF (F); VTT (FIN); TECNATOM (E); SKI (S), ECN (NL); JRC/IAM, DGXI of CEC, DG XVII.

Funding

- Various sources: national programmes, regulatory bodies, utilities, CEC programmes and JRC/IAM support.
- Contribution in kind of all task members.

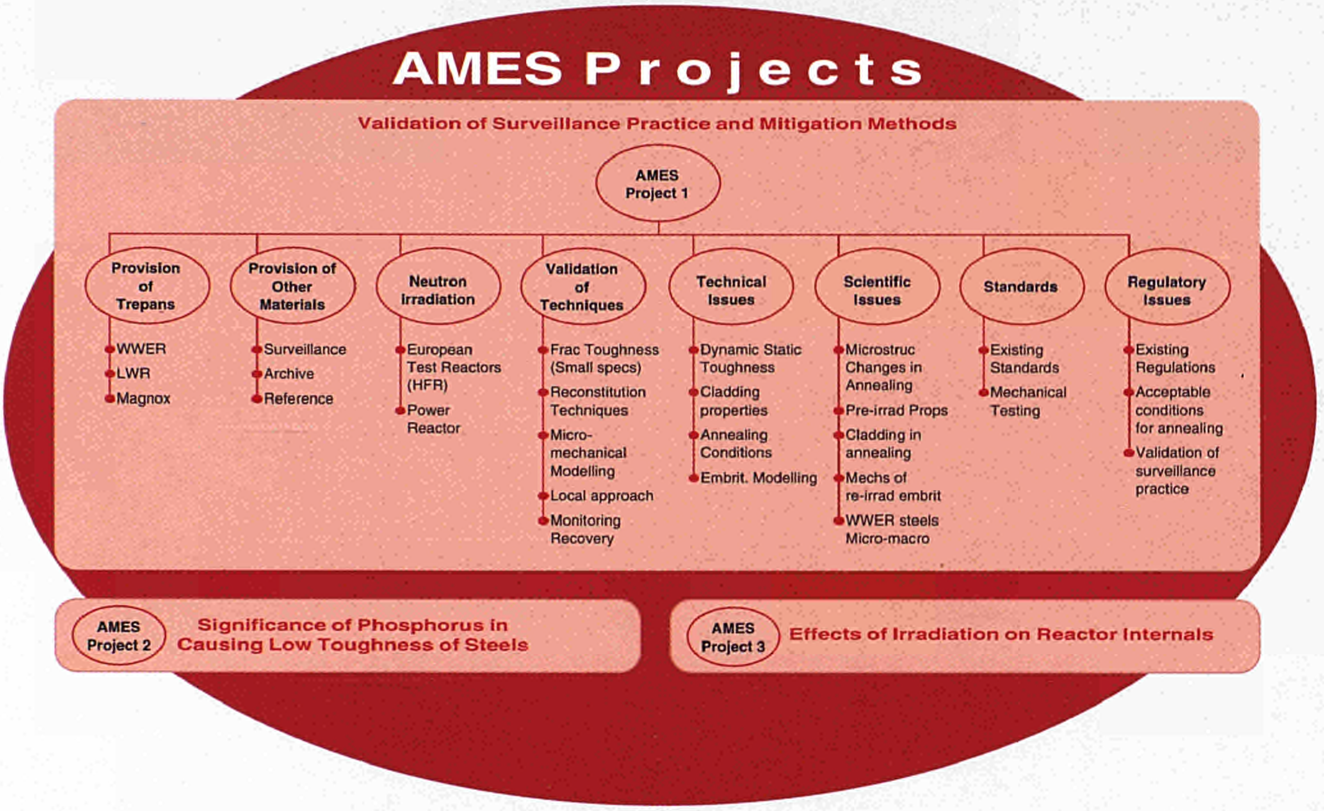
Operating Agent

JRC, Institute for Advanced Materials of CEC in principle with the help of national institutions of excellence as required by the tasks: VTT, MPA Stuttgart, TRACTEBEL, CEA/CEREM.

Officers

• Chairman of the Steering Committee:		M. Davies	UK
• Network Scientific Advisor:		C. English	AEA, UK
• Network Manager:		S. Crutzen	CEC JRC/IAM
• Network Secretary:		U. von Estorff	CEC JRC/IAM
• Task Chairmen:	Task 1	K. Törrönen	VTT, FIN
	Task 2	R. Gerard	TRACTEBEL, B
	Task 3	J. Föhl	MPA, D
	Task 4	P. Petrequin	CEA/CEREM. F

Table below: *AMES projects*



There have been several Steering Committee meetings in 1993 both for identifying projects of common interest and for organizing the first actions. The outcome is given in the table above.

AMES1: "Validation of Surveillance Practice and Mitigation Methods"

The irradiation response of current WWER-440 reactor pressure vessels (RPVs) has not yet been thoroughly studied. Only miniature specimens have been taken from representative vessels; larger trepans taken from Novovoronetz1, or to be taken within the TACIS programme from Novovoronetz2, do not represent the majority of operating WWER-440 plants. This is due to differences in size of the vessel, operating temperature and the lack of proper operation history documentation. Similarly, the annealing response has been studied only with miniature specimens.

The knowledge of the actual irradiation embrittlement, annealing and reirradiation response is of utmost importance for the safety of still operating VVER-440 units, and also for the credibility of nuclear power in general.

A similar situation is true for Western-type reactors. Decommissioned vessels studied so far have not been truly representative of the majority of operating power reactors. There is, however, a clear need to develop generic techniques to be used also in the Western-type reactors with regard to sampling of vessel material for evaluating the annealing response. These techniques should include miniaturised mechanical testing and microstructural evaluation. There is European interest to demonstrate these techniques with available decommissioned vessels, i.e. both WWER-440-type and Western PWRs (possibly also with Magnox-type) vessels.

Four units of the Greifswald NPP, representing WWER-440 reactors, and e.g. ChoozA, representing a Western PWR, have been decommissioned and which could be available for extensive investigations.

AMES2 will deal with the "Significance of Phosphorus in Causing Low Toughness of Steels".

AMES3 will deal with the "Effects of Irradiation on Reactor Internals".

5. Exploratory Research

Boron Neutron Capture Therapy (BNCT)

The development at the High Flux Reactor of BNCT as a radiotherapy modality for treating malignant glioma, continues steadily towards the initiation of clinical trials. Progress has included:

Free-beam measurements

Further measurements were made to assess that the beam parameters (flux intensity and neutron energy spectrum) do not change or are not significantly affected by reactor changes. The results of the latest measurements, indicate that the beam parameters vary by no more than 1-2%, (table below).

Phantom measurements

More phantom irradiations were performed, including assistance by a group from the Nuclear Institute at Rez, Czechia, using their phantom of the upper-torso of a human, which included internally, an actual human skeleton.

Cell culture experiments

Further irradiations were performed to observe the effect of the boron capture reaction at depth inside a phantom. Participating groups included JRC and ECN Petten, University of Bremen and the Netherlands Cancer Institute in Amsterdam.

Healthy tissue tolerance studies

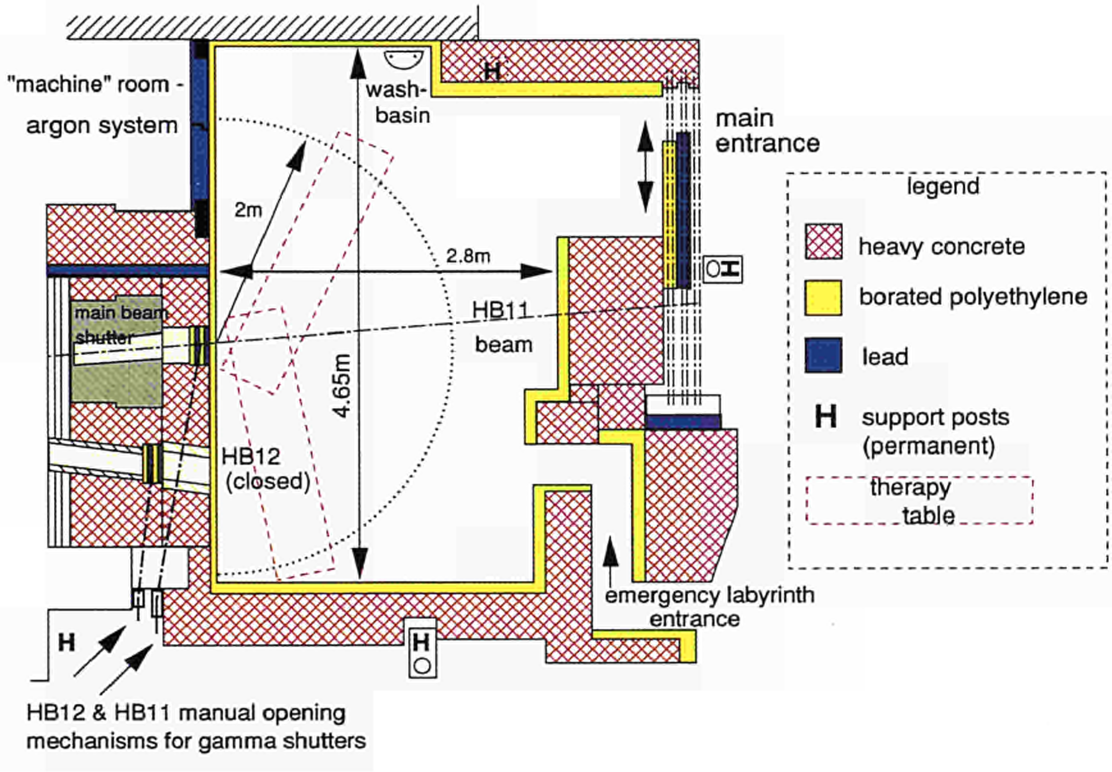
A critical component in the whole project is knowledge of the safe dose to be given to the healthy tissue as a function of boron dose and irradiation time. Consequently, 47 canine experiments have been performed. The final irradiation took place in June. The full range of radiation effects has been observed, varying from severe neurological changes to no radiation damage at all. The experiment has therefore been highly successful, whilst performing only a limited number of irradiations. Following a further observation period, the tolerance dose for 2 levels of boron concentration will be confirmed. In other words, the recommended starting dose for patient treatment can be specified.

On-line dosimetry

The on-line beam monitoring system, utilising a twinned system of GM tubes and fission chambers, has been extensively tested. Measurements can detect: reactor power changes of 0.5%; neutron: gamma ratio changes of less than 2%; activation of argon increases the gamma signal by 0.3%; and scattered neutrons from the patient (phantom) increases the fission count rate by 1.2%.

Table below: *Comparison of Free Beam Measurements at the HB11/BNCT facility*

Foil and reaction	ratio: - reaction rate: reference reaction rate (4/92)					
	7/91	**4/92**	**5/92**	**7/92**	**10/92**	**6/93**
$^{197}Au(n,\gamma)$	1.04	1.00	0.95	0.95	0.95	0.95
$^{115}In(n,\gamma)$+ Cd	0.96	1.00	0.94	0.94	0.98	0.99
$^{45}Sc(n,\gamma)$+ Cd	0.99	1.00	0.95	0.97	0.94	0.97
$^{197}Au(n,\gamma)$+ Cd	1.01	1.00	0.95	0.96	0.94	0.96
$^{186}W(n,\gamma)$+ Cd	0.96	1.00	0.94	0.95	0.95	0.95
$^{238}U(n,\gamma)$+ Cd	0.98	1.00	0.95	0.95	0.94	0.94
$^{139}La(n,\gamma)$+ Cd	0.98	1.00	0.95	0.96	0.94	0.95
$^{55}Mn(n,\gamma)$+ Cd	1.00	1.00	0.92	0.96	0.95	0.94
$^{63}Cu(n,\gamma)$+ Cd	1.01	1.00	0.94	0.95	0.94	0.95
$^{115}In(n,n')$+ Cd	1.22	1.00	0.87	0.92	0.93	0.99
$^{58}Ni(n,p)$+ Cd	2.63	1.00	0.93	0.94	1.03	0.92
$^{27}Al(n,\alpha)$+ Cd	—	1.00	0.88	1.03	0.95	0.93



Above: *Cross-section through the new, enlarged irradiation room at the HFR BNCT facility*

Treatment planning

The treatment planning scheme, based on the Monte Carlo code MCNP, has been further improved, including pre- and post-graphical routines, but substantial changes to improve the calculational time are still required.
As such, a collaborative effort between JRC, the Netherlands Cancer Institute and INEL has been started.

Patient treatment room

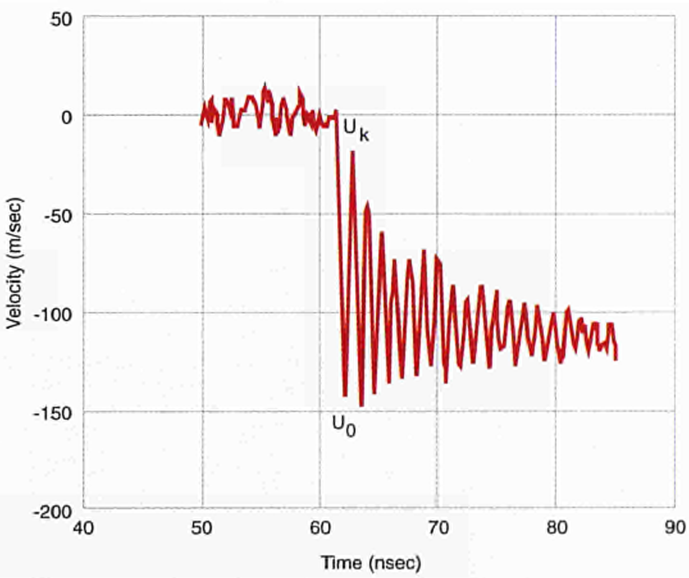
Work began after the summer on constructing the new irradiation room. The new set-up has dimensions 4.65 x 2.8 x 2.2 m^3, (figure above).
The room comprises of over 70 tonnes of shielding material. The ceiling consists of 2 layers of solid steel beams, each 100 mm thick, 1 layer of lead bricks, 50 mm thick and 2 layers of borated polyethylene, each 50 mm thick. The inside surfaces of the room will be lined with 1 or 2 layers of borated polyethylene plates. The new set-up, including a medical observation area, will be ready early next year. Soon thereafter, it is expected that clinical trials can begin.

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

A laser spallation experiment has been developed to measure the strength of adhesion of a thin film to a substrate at high temperatures, using a laser pulse induced spallation method and a Laser-Doppler-Interferometry device (VISAR). The technique involves impinging a high-energy, pulsed laser beam on the back side of the substrate which is coated by an absorbing material. The incident laser radiation is converted rapidly to thermal energy and the explosive evaporation of the absorbing material sends a compressive shock wave through the substrate toward the film-substrate interface. This is, in turn, reflected from the free film surface giving rise to a tensile wave leading to the film removal. The free surface velocity of the coating is monitored during the experiment by the VISAR.
From the velocity measurements one can determine the maximum stress at bond separation through the relation : $\sigma_s = \frac{1}{2} \rho_0 c (u_0 - u_k)$ where, σ_s is stress on the interface at the instant of rupture, ρ0 mass density of the film, c velocity of propagation of the stress wave, u_0 and u_k initial jump-off and first pullback velocities as shown in the figure, and c velocity of propagation of the stress wave in the coating material. The latter is also derived from the velocity profile and the coating thickness. The measured bond strengths are reproducible and do not show dependence on shock amplitude for identically prepared specimens. Finally it is shown by experiment and mathematical simulation alike that de-bonding occurs rapidly enough (in less than 10 nsec) that separation through crack growth cannot occur. Thus atomic bond rupture is the mechanism of separation in this experiment and the measured spall stress is the measure of the strength of adhesion where atomic bonding is the mechanism of film deposition as, for instance, in the case of chemical vapour deposition.
During 1993, this project made significant progress towards its ultimate goal, measurement of bond strength at high temperatures, based on an extensive programme of experimental and analytical/numerical work including

a. the VISAR development and its successful performance in monitoring a vibrating spall surface with an area of about 1 mm^2 with time resolution of 1 nsec.
b. selection of the optimum energy absorbing material, measurement of overall absorptivity at various laser flux levels optimization of thickness
c. measurement of the pressure pulses generated by different flux levels using a microelectronics device based on the piezo electric phenomenon
d. determination of the threshold flux level that causes spallation
e. computation of the value of the tensile stress experienced at the interface, based on the results of c and d, by mathematical simulation of the stress wave propagation through dissimilar materials. This leads to cross checking of the bond strength levels measured by the VISAR and those estimated based on steps c through e.
f. finally the microelectronics device and VISAR results enable us to calibrate our fully non-linear physical/numerical model for the generation of the primary shock and its propagation across the various substrate/coating materials. This task is expected to be completed in 1994.

Below: *Coating surface velocity profile, measured by Laser Doppler Interferometry, of an Al_2O_3/SiC system during a spallation experiment*



Engineering of Fibre-Matrix Interfaces

The liquid infiltration of woven fibres with ceramic or ceramic precursor materials offers a low cost, versatile processing route for the fabrication of ceramic composite components. However, current processing routes using polymer precursors or ceramic dispersions introduce a level of intrinsic porosity following burn-out or drying which limits ultimate composite properties.

Infiltration of powders under electrophoretic potential may generate more uniform and higher green densities and reduce macrodefect populations in the densified ceramic. Targetted to support the on-going activity to develop a C-fibre/Si_3N_4 composite, a new exploratory research project to evaluate this novel processing route will investigate the fabrication of appropriate oxide and non-oxide powders and their deposition as coatings or by infiltration into fibre weaves, from aqueous and nonaqueous solvents.

Commercial Si_3N_4 powders have been coated with ca. 10 nm coatings of Al_2O_3, Y_2O_3 and Al_2O_3/Y_2O_3 mixed coatings, by in-situ precipitation from water/urea solutions of Al and Y sulphates respectively.

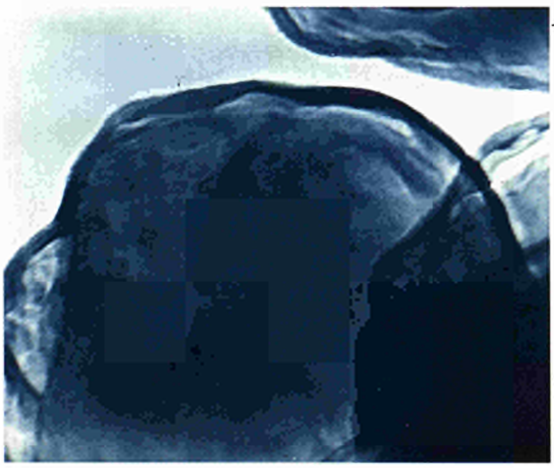
TEM shows these coatings to be continuous, adherent and uniformly deposited on the particle surfaces (figure below left). Aqueous 1.5 vol% suspensions of these powders have been stabilised with commercial dispersants and their dynamic mobility profiles determined for the pH range 4.0 to 10.0 . The general profile for the single coated particles matches well values for pure Al_2O_3 and Y_2O_3 powders under the same conditions.

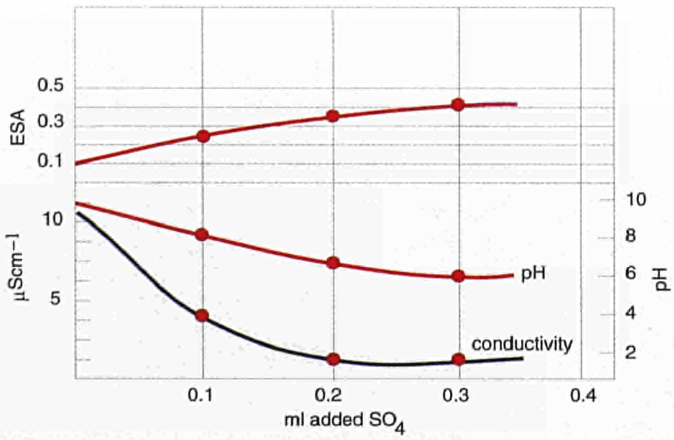
The surface of the mixed coating shows an adsorption/desorption behaviour which will be further investigated.

Electrophoretic studies have started with a survey of candidate dispersion conditions, using standard powders and taking into account, pH/zeta potential, concentration and dispersant additives. Optimised 5 vol%.aqueous dispersions of both Si_3N_4 and Al_2O_3 were used for depositions on to C rod, plate and woven fibre mat electrodes, varying the applied potential within the 3.5 volt gas evolution maximum and the cell/electrode geometry. Coatings were, in general, non-uniform and cracked heavily on drying. From the poor quality of coatings deposited from water dispersions, attention has turned to the use of non-aqueous solvents. Dispersions in ethanol are stabilised with citric acid (for Si_3N_4) or with sulphuric acid (for Al_2O_3). Figure below right shows the comparative gain in particle surface charge and the reduction of solvent conductivity and concomitant change of pH from 10 to ca 6.5 with addition of H_2SO_4. Early coatings show considerable promise.

Below left: *TEM micrograph showing coated layer of alumina and yttria on Si_3N_4 powder particles*

Below right: *Gain in Al_2O_3 particle surface charge and decrease in pH and solvent conductivity with SO_4 surface adsorption*





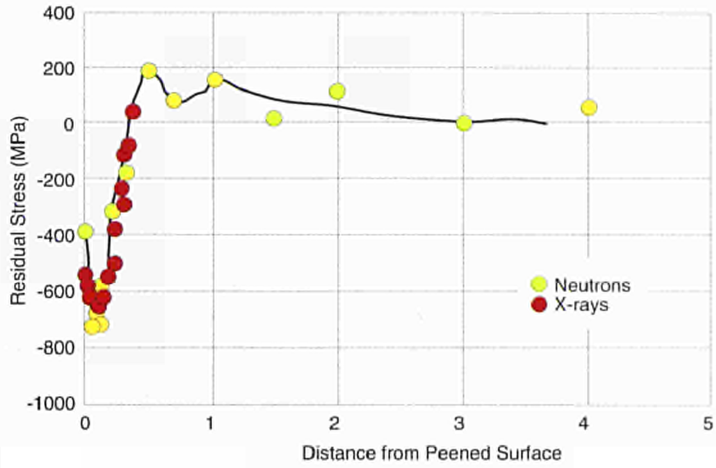
Measurement of Localized Stress Fields by Neutron Scattering

The objective of the project is to explore the potential of the neutron scattering technique for measuring of stress field gradients near surface and interface layers in advanced materials. The activity started in April 1994, following the conclusion of agreements with ECN with regard to the conditions of collaboration between both Institutes, and with Imperial College, London, for providing expertise. The equipment used for the measurements is the diffractometer HB4 in the High Flux Reactor (HFR) at JRC Petten.

First efforts focussed on: improving the alignment of the existing diffractometer HB4, establishing an improved alignment procedure, and the training of IAM staff and transfer of expertise from ECN to IAM staff with respect to the operation of HB4. Stress measurements concentrated on shot peened steel blades and on joints between silicon nitride and steel or niobium. Results on the shot peened steel plates show that the measured residual stress profiles compare well with X-ray diffraction results, (figure above).

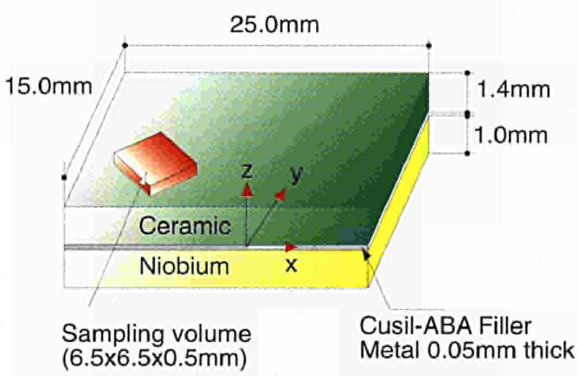
The advantage of the neutron diffraction technique, however, is that it reveals the stress distribution through the entire thickness of the sample, enabling conclusions on aspects as, for example, the depth of changeover from compressive to tensile stresses, which is information not readily accessible by means of X-ray measurements.

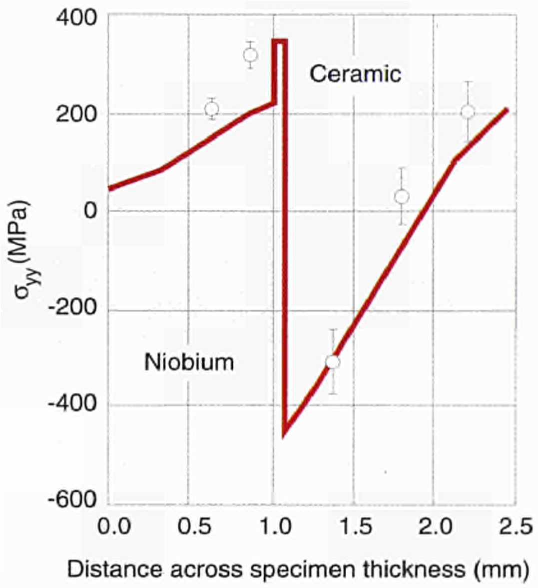
The first series of measurements on the silicon nitride-steel joints yielded disappointing results in that diffracted peaks at certain positions in the ceramic were lost, stress levels converted from the



Above: *Comparing the residual stresses measured by means of neutron diffraction and X-rays in a shot peened steel plate*

Below: *Silicon Nitride/Niobium bonded specimen and residual stresses parallel to the bonded interface as measured by Neutron Scattering and computed (solid line) using 3D finite element modelling*





measured lattice spacings were far too high and the error on the stress levels is too large. By optimizing the choice of the diffracting plane, the scanning angle, the counting time and the sampling volume, the situation has now evolved to the point where realistic levels of the stress with acceptable error margins have recently been measured near the interface of a silicon nitride bonded to niobium. Measured stress levels are compared to calculated values in figure on page 92 below.

Still further improvements in terms of resolution are required, for example by reducing the noise level. This involves identifying the source of the background radiation prior to any decision with respect to shielding of the diffractometer.

The next measurement campaigns are scheduled to focus on exploring the feasibility of the neutron diffraction technique for measuring stresses near the coating-substrate interface of coated materials. A range of coated steel and nickel-base alloy specimens for residual stress measurements has been obtained from the Advanced Coating Centre (steel with alumina coatings) and from MTU (Munich). The latter delivery includes thermal barrier coatings, wear resistant coatings, abradable coatings etc. made according to industrial standard practices.

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IV Meetings/ Conferences

Meetings/Conferences

Date	Venue	Subject Area	Type of Meeting	Co-organiser/Co-Sponsor
4.5	Petten	Thermal Protection Systems	Workshop	ESA Noordwijk
17.5	Petten	HTMTC Alignment WP2	Workshop	HTMTC UK
5/6.6	Ispra	Vanadium Alloys as Structural Material in ITER	Workshop	
14/16.6	Petten	Materials for Coal Gasification	Int. Workshop	EPRI, USA; KEMA, NL
15/20.8	Stuttgart	SMIRT 12 Section G	Conference	Univ. Karlruhe
22/25.8	Stuttgart	SMIRT 12 Section 2	Conference	Univ. Karlsruhe
27.9/1.10	Stresa	Fusion Reactor Materials	Int. Conference	
25/29.10	Ispra	Inspection Qualification	Eurocourse	
29.10	Petten	HTMTC Alignment WP2	Workshop	HTMTC
1/2.12	Moscow	Inservice Inspection Reliability	Workshop	DG XI
13/14.12	Ispra	Characterization of Coatings by NDE	Workshop	

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 Techniques
RWE	Rheinisch Westphälische Elektrizitätswerke
SAS	Standard Acoustic Source
SEM	Scanning Electron Microscopy
SEP	Société Europèene 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

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

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 H.T. alloys: V. Guttmann, H. Fattori

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, L. Lamain, G.P. Tartaglia, G. Sordon

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

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, P. Lemaitre, F. Vila Aparicio, U. von Estorff

Physical properties of coatings: W. Gissler

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

Application of NDE to Materials and Thin Coatings: S. Crutzen, R.F. Denis, F. Lakestani, J.F. Coste

RESEARCH AREA: SURFACE MODIFICATION TECHNOLOGY

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

Wear and corrosion resistant coatings: E. Bullock,

Surface/bulk structural properties modification: F. Lanza

Marangoni phenomena in materials processing: G. Tsotridis, R.A. Brash

Sub-microstructural engineering: S. Pickering,

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

Coatings characterization: E. Lang

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. Cundy

SPECIFIC PROGRAMME: FUSION MATERIALS

Assessment of Research Area Progress and Achievement: P. Schiller

Low activation material: P. Fenici

Thermal fatigue: P. Fenici

Blanket studies: P. Fenici

Fusion materials data bank: P. Schiller

SUPPLEMENTARY PROGRAMME

Operation of the High Flux Reactor: J. Ahlf

S/T SUPPORT TO SERVICES OF THE COMMISSION

Standards for advanced ceramics (DG III): M. Steen, D. Baxter

Standards NDT for pressure vessels (DG III): S. Crutzen,

Ceramic catalyst support (DG XI/VII): R.J. Fordham

Performance demonstration of NDE techniques (DG XVII): S. Crutzen

Materials and inspection Evaluation (DG XI): S. Crutzen, U. von Estorff

EXPLORATORY RESEARCH

Boron neutron capture therapy: R.L. Moss

Adhesion in films and coatings by a laser pulse induced spallation technique: A.G. Youtsos

Engineering of fibre-matrix interface: E. Bullock

Measurement of localised stress fields by neutron scattering: J. Bressers, A.G. Youtsos

Editing and Coordination: M. Cundy

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Abstract

The Annual Report 1993 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.

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