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COMMISSION OF THE EUROPEAN COMMUNITIES

**Multiannual Programme of the Joint Research Centre 1980 - 1983**

# 1981 Annual Status Report

## High-temperature materials

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## High-temperature materials

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# HIGH TEMPERATURE MATERIALS
# 1981

**Research Staff:** **38 persons**

**Budget :** **4,272 Mio ECU**

**Projects:**

1. **High Temperature Materials Information Centre**
2. **Materials and Engineering Studies**
3. **High Temperature Materials Data Bank**

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Commission of the European Communities
Joint Research Centre
Petten Establishment
P.O. Box 2
1755 ZG Petten, The Netherlands

## 1. INTRODUCTION

The High Temperature Materials Programme is executed at the JRC, Petten Establishment and has for the 1980/83 programme period the objective to promote within the European Community the development of high temperature materials required for future energy technologies.

The strategy of this programme is a combined approach to survey technology and industrial requirements in order to identify future needs, to establish and perform research projects in selected key areas (coal conversion and related processes) and finally to build and maintain multilateral communications with manufacturers, users and also with the research and development sector involved in these materials.

The programme contributes to the public service and the "central nature" roles of the JRC by provision of scientific and technical information and expertise and also by acting as a focal point for co-ordination.

# 2. RESULTS

## 2.1 Information Centre

The objectives of this project are the provision of information service functions to the European HTM community and the encouragement of co-operative actions.

In order to meet these objectives, the Information Centre has undertaken three separate activities, i.e.

- Information Exchange and Transfer, organising conferences, symposia, colloquia, seminars and courses,
- Information Collection, executing inquiries, surveys and studies,
- Information Collection, establishing an Inventory on on-going research.

The results obtained are presented in Fig. 1.

## 2.2 Materials and Engineering Studies

The increasing cost of energy and the future need to provide alternatives to oil and natural gas are prompting the development and improvement of many energy conversion and utilisation processes. Examination has shown that satisfactory materials performance is critical for the operation of advanced energy technologies, such as:

- fossil fuel and waste combustion, coal gasification, petrochemistry, biomass conversion, MHD,
- nuclear systems such as AGR, HTR, PNP,
- new energies such as solar power, hydrogen,
- steam & gas turbines, combustion engines, heat exchangers, chemical reactors, cyclones and other cleaning systems.

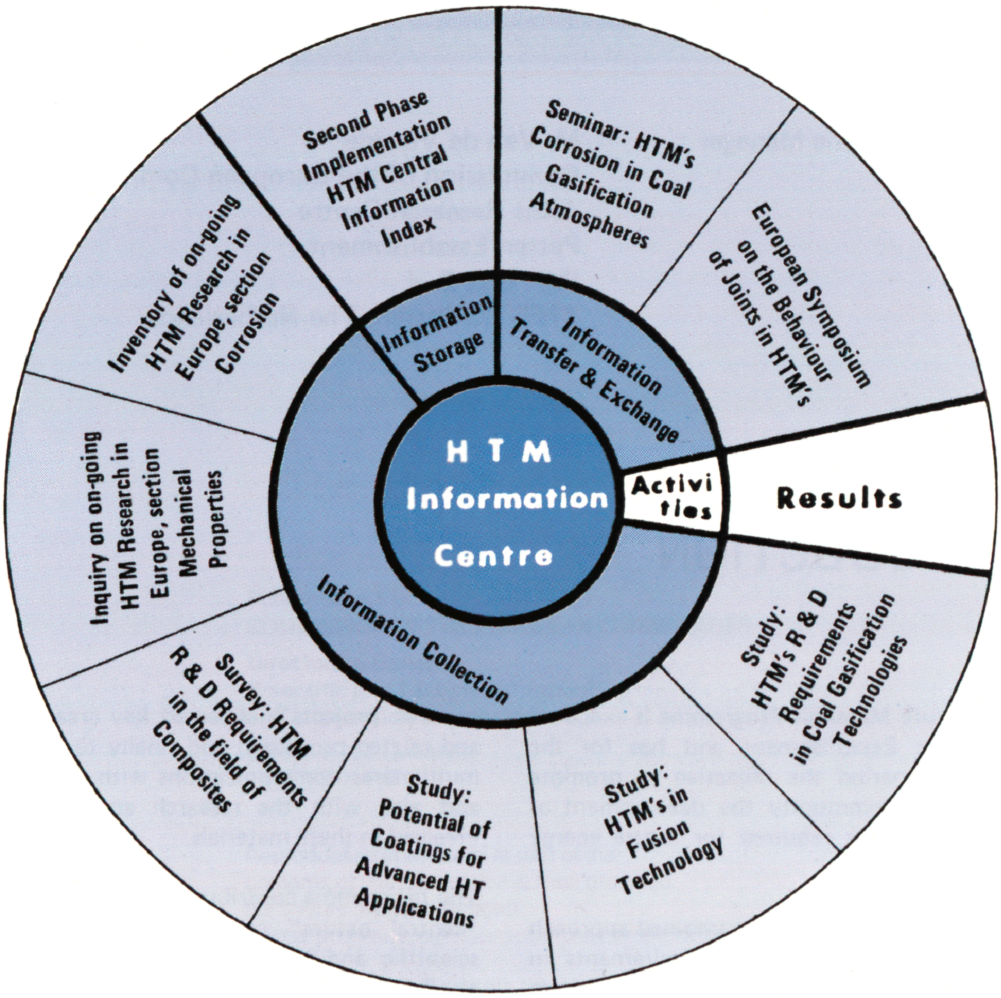



**Fig. 1 Information Centre, Activities and Results.**

The overall technological requirements to be met by constructional materials in these application areas include:

- increased effiency, especially through higher process temperatures,
- improved reliability, thus reducing shut downs,
- safety, both for operators and the local population,
- tolerance of the increasingly corrosive environment arising from changes in process parameters or the use of lower grade fuel.

Some of the requirements can be met by transferring technology from one application to another, others can be met by "fine-tuning" materials and improving their characterisation, but some demand the availability of new materials or new processing techniques. The general approach in the Material & Engineering Studies project is to provide behavioural patterns of materials in specimen and component form for application in the advanced energy technologies. Work to explore the mechanisms by which corrosion proceeds in industrially simulative low oxygen bearing environments is an important part of the programme; the conclusions provide information on expected life and on the steps necessary to develop more resistant alloys. A further line of attack on the problem explores the development of coatings, their application technology, and their succes in preventing corrosion of the constructional materials. When corrosion data suggest that the bare, or coated, alloy has a reasonable life expectancy, information is needed on the effect that exposure has on the mechanical properties.
Data have then to be obtained to charaterise the alloy and components made from it for creep and fatigue behaviour in the environments concerned in order that reliable design information may be derived.

The types of environments being used typify those found in various petrochemical, biomass coversion and HTR processes. They also relate to the effects found in more complex coal conversion atmospheres.
In these applications the special life limiting problems concern carburisation of the material or the effects of interaction between different corroding reactions such as oxidation with carburisation or sulphidation.
Because of its importance to these applications, the range of temperatures used in most of the testprogramme is 700 to 1100°C.

The alloys under test are, for the most part, the heat resistant steels which are typical or candidate structural materials for fossil fuel conversion processes. They fall into two groups with representatives in each of wrought and centrifugally cast products:

25°/o Cr - 20/25°/o Ni
e.g. Type 314, HK 40,

22/25°/o Cr - 33/35°/o Ni
e.g. Alloy 800H, HP40Nb, HP40W.

In addition certain alloys which are used predominantly in gas turbines are being examined either in the above environments or in air.

Nickel base
e.g. Hastelloy X, IN738LC, Waspaloy, PM Astroloy,

Cobalt base
e.g. HA188.

Investigations on dynamic loading (fatigue) and coating of gas turbine alloys have been carried out in cooperation with COST 50 (European Co-operation in the field of Science and Technology - Action 50, Gas Turbine Materials).

## Corrosion without Load

Environmental parameters such as temperature and the chemical activity of carbon (C), oxygen (O) and sulphur (S) are varied systematically and different alloy compositions are used so that a more scienific basis can be formulated for the selection and the continuing development of new materials to satisfy the demands of the service applications.

Intermittent weighing of the specimens during the periodic interruption of tests conducted in autoclaves at temperatures between 800 and 1000°C ensures that the kinetics of the corrosive reactions are monitored.
The mechanisms by which the corrosive degradation proceeds are elucidated by detailed examination of representative specimens using surface and cross-sectional structural analysis techniques, e.g. X-ray diffraction, optical microscopy, electron-microscopy, and electron spectroscopy.

The rate at which carburisation proceeds has been computed from observations made on representative iron, nickel & cobalt base alloys following exposure at various temperatures and activities of carbon ($a_C$). This enables information to be derived about penetration through a component, etc. There are significant effects due to form of the material and any surface working, also due to the composition, both in terms of major alloying elements and minor additions. This information is essential for the estimation of component life-times.
From a knowledge of the alloy class and the exposure conditions the volume of carburised material after a given time can be computed and used to asses the effects on re-welding or on mechanical properties, etc.

When there is sufficient oxygen present to form a surface scale the resulting interaction of carburising and oxidising mechanisms leads to a more complex situation since carburisation is critically dependent on the absence of a stable oxide film. For example, a film of silica forming on the metal surface, or preformed in a seperate exposure reduces the rate of uptake of carbon by a factor greater than 10.

Corrosion studies of the behaviour of materials in purely sulphidising atmospheres have progressed and an understanding of the mechanisms governing such degradation has been developed. Test exposures in both low sulphur activity ($pS_2 \approx 10^{-11}$ atmos.) and high activity ($pS_2 \sim 10^{-6}$ atmos.) $H_2$-$H_2S$ gas mixtures at 825°C have been completed. The amount of sulphidation occuring in a given time is at least two orders of magnitude greater in the higher sulphur activity environment, where much thicker scales of complex morphology and composition are found.

These thick scales readily exfoliated, in marked contrast to the thinner, adherent and relatively protective scales formed during exposure to the low sulphur atmosphere.

The behaviour of high temperature alloys at the lower level of sulphur which is below the threshold for the formation of NiS gives encouragement for the possible use of nickel base alloys in applications where these conditions are found.

Exposure of these materials to a $H_2/CH_4/H_2S$ gas mixture at 1000°C which has a carbon activity of 0.8 and sulphur pressure $pS_2 \sim 10^{-11}$ atmos. resulted in a complex interaction of mechanisms. Examination showed that sulphidation does not take place; only some intermittent sulphide particles could be detected at the original sample surface.

Carburisation is restricted to about a third of that occuring in the sulphur free condition. In addition, more carbides are formed above the original sample surface than in the case for a purely carburising environment which perhaps reduces still further the amount of carbon available to diffuse into the alloy. Thus the presence of a sulphidising species has restricted and changed the carburising behaviour. The lack of corrosion by sulphidation is also remarkable, being due to the effect on corrosion mechanisms exerted by the relative stabilities of carbide and sulphide in the Cr-S-C thermodynamic system. This illustrates clearly the complex situation which exist in various petrochemical and coal conversion environments.

## Corrosion under Load

If the intensity of corrosive attack on metallic materials by aggressive environments is found to be stress dependent, the conclusions derived from corrosion tests without load would need to be confirmed or modified for conditions where the materials are under load. This activity aims to obtain a qualitative understanding of corrosion mechanisms and some quantifications of corrosion kinetics under the action of static and dynamic stresses.

Tests have been conducted on HK40 and Alloy 800H under static (creep) conditions 900 - 1000°C in a gas with a high carbon activity ($a_C = 0.8$). It has been found that stress has the effect of reducing carburisation kinetics but that it is sufficiently small (~ 5°/o) to be ignored in practical situations.

Under carburising/oxidising conditions the effect of load on corrosion could be significantly different, if test piece deformation leads to scale fracture and hence to carburisation which would not occur under stress free conditions.

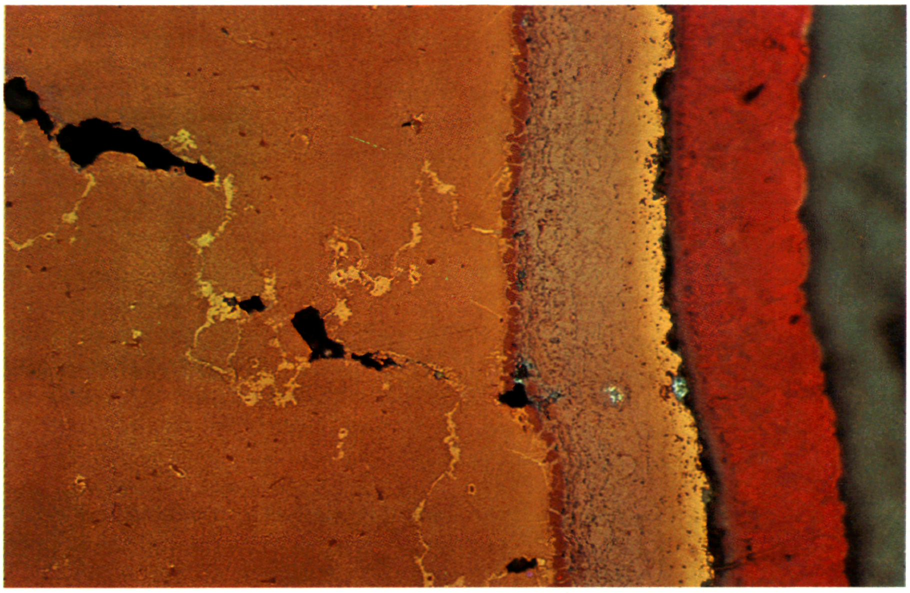
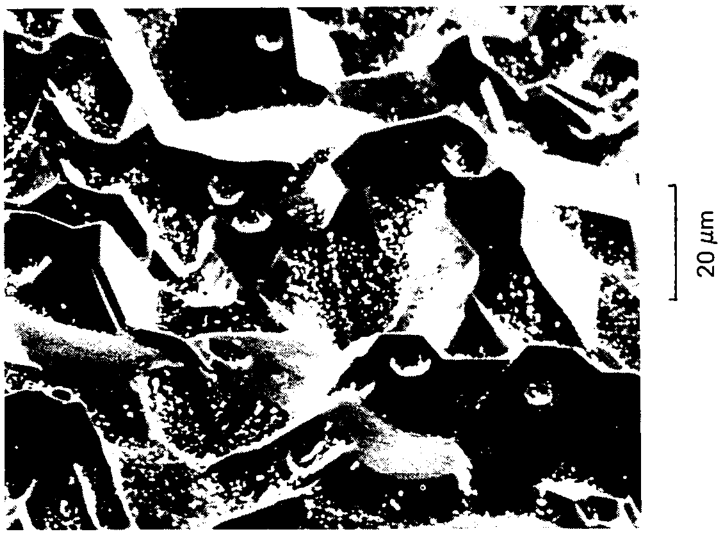


**Fig. 2 Deformation of the S57 coating by subcoat cracking.**

a) Section through Sulphide Scale


b) Morphology of Scale - Gas/Scale Surface




c) Morphology of Scale - Scale/Alloy Surface

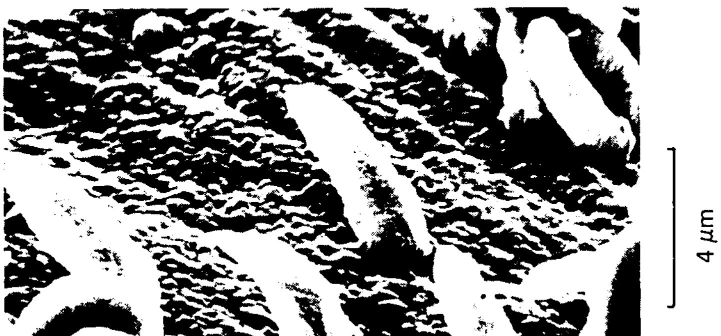



Fig. 3 Example of Scale Morphologies (Inner and Outer Surfaces), formed on 28 Cr-40Ni-4W Alloy exposed to Low Sulphur Activity Environment ($pS_2 \sim 10^{-11}$ atmos.) for 50 hours.

## Mechanical Properties under Dynamic Loads

The materials in many industrial components are subjected to periodic stress cycles, either imposed at constant temperature by changes in the mechanical loading or caused by temperature cycling.

A reasonable level of knowledge already exists concerning the behaviour of many materials under static loads in oxidising environments. However, with regard to the varying load situation which results in fatigue, the level of understanding is much lower. The introduction of aggressive environments complicates the situation even more and the resulting situation is characterised by a multitude of simultaneously operating processes in the material which may or may not mutually interact.
The initial work for the exploration of the interaction between fatigue and time dependent processing (e.g. creep) to be expected in High Temperature Low Cycle Fatigue (HTLCF), has been orientated towards the behaviour of gas turbine disc forgings for which an air environment is appropriate. Because of its application area this investigation, which is now concluded, has been integrated into the COST 50 activities.

The alloys were Waspaloy, tested in the range 500 to 650$^{o}$C and P/M Astroloy, tested between 400 and 730$^{o}$C, the latter alloy being examined during the past year. Test temperatures and frequencies correspond with those found in service. The applied plastic amplitudes extended from conditions commonly used for short duration laboratory testing down to the very low levels which are more directly applicable to the operating conditions in gas turbines.

The raw data have been analysed in terms of cycling hardening/softening curves, endurance curves and cycling stress-strain curves to yield a full phenomenological description of the tensile and LCF behaviour of the alloys over a wide temperature and strain-rate range.
The observed material behaviour is the result of interactions between the cycling deformation process and several time dependent phenomena. The time dependent processes of interest were identified either on the basis of the phenomenological results or were deduced from an analysis, on a microstructural scale, of the deformation and fracture processes.
The relevant time dependent processes are ageing reactions, time dependent deformation (creep), dynamic strain-ageing and oxidation.

Over the largest part of the temperature/strain-rate field the deformation is controlled by dislocations gliding in planar slip bands. The confinement of dislocation glide to these bands becomes less accentuated at high temperatures and low strain-rates. At 730$^{o}$C for very small strain -amplitudes (i.e. long living tests) the deformation changes to a homogeneous mode producing a dislocation cell structure, as commonly found in creep. In the same area of high temperature, long life testing, grain boundary strenghtening by precipitation of $M_{23}C_6$ carbides was also observed. Conversely, dynamic strain ageing was associated with conditions more representative of "pure" fatigue. Oxidation was found to be effective over the entire field of investigation and to be a major life determining factor. The change from vacuum to air environment could reduce life by 2 orders of magnitude.

## Surface Protection - Scales and Coatings

The use of all metallic materials for application at high temperatures in aggressive environments requires the formation and preservation of a protective surface oxide layer or scale to act as a barrier to corrosion.
In several engineering applications the strength requirements involve the use of materials which do not develop a protective oxide scale. Hence coatings able to withstand the conditions seen in service have to be used. The protective action of these coatings against corrosion is related to the development of their natural scales.

Common to both modes of surface protection is the need to preserve the oxide scale under the operating conditions. In addition to chemical reactions there are also mechanical constraints through oxide growth stresses, thermal cycling and/or superimposed mechanical loading which can cause fracture or spalling of the scale, so making it ineffective as a corrosion barrier.
In the case of applied coatings, reactions may also occur between coating and substrate alloy which can detrimentally effect the protective ability of the coating and also the mechanical properties of the composite material.

The scope of this activity is to examine the behaviour of a number of oxide scales and protective coatings on high-temperature steels and nickel base alloys, to allow the selection of alloy/oxide systems which are capable of providing extended service in coal conversion and other engineering applications.

During 1981 work commenced on the corrosion behaviour of a potential cladding alloy, FeCralloy (16Cr 5Al 0.5Y). In comparison to work previously conducted in an austenitic steel, IN519 with addition of yttrium, it was found that the oxidation kinetics of FeCralloy indicated a much slower growing $Al_2O_3$ scale with better protective properties. The resistance of this thin scale to spalling during severe cyclic temperature exposure was also much improved.

Furthermore, preliminary tests showed that the $Al_2O_3$ scale which formed under conditions of very low $pO_2$ gave excellent protection against carburisation. A more comprehensive programme has now been established in collaboration with a number of other research institutes to explore the corrosion behaviour of this material and other coatings in coal conversion atmospheres. Assessments of coating/substrate interaction have been carried out on the cast nickel-base alloys IN738 and

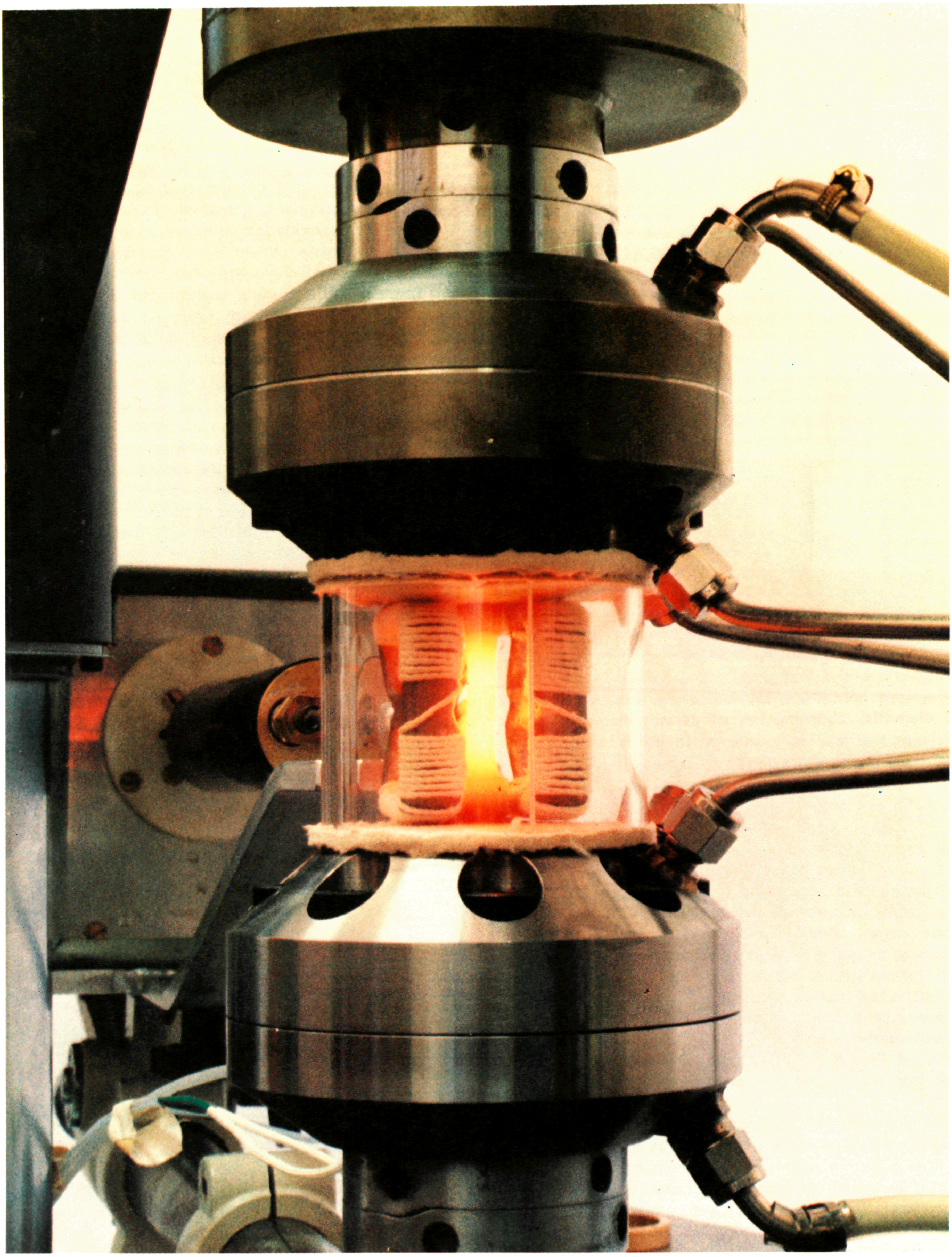


**Fig. 4** **MTS machine with HF heated sample.**

IN100 following reports of a degradation in strenght properties as a result of the formation of intermetallic phases at the interface between the coating and the substrate blading alloy. These investigations, which are now complete were integrated into the COST 50 activities.

Conventional aluminide coatings, applied by pack diffusion processes (e.g. LCD-2) and new overlay coatings deposited by techniques which are still under development (e.g. Low Pressure Plasma Spray) have been explored. The overlay coatings were in a NiCoCrAlY alloy identified as S57.

Investigations of the structural stability of various aluminide coatings shows that their properties and structure depend significantly on the coating process parameters and the composition of the substrate alloy.
Interaction between coating and substrate occurs during exposure in the range 750 - 1100°C, leading at all except the highest temperatures, to the formation of a subcoat region containing varying amounts of platelike precipitates. The exact details of the interaction and of the platelike intermetallic compounds appeared to depend on the coating, composition temperature and time of exposure.

Oxide formation and spallation is the major coating degradation process occuring at an alloy surface during exposure in air. The type and morphology of the oxide scale depends primarily on the coating type (and substrate alloy) and - to a lesser degree - also on the exposure conditions (atmospheres, temperature, etc.). Preferential consumption of certain elements in the coating by scale formation was found to lead to further changes in metallic and carbide phase stability.

Overlay coating structure was much less dependent on the substrate composition but was strongly influenced by process parameters. Taking LPPS (the only process examined which resulted in satisfactory coatings) the structure, after deposition followed by the quality heat treatment appropriate for the substrate alloy, had a homogenous intermetallic matrix containing a dispersion of small hard particles. There was a narrow zone of interdiffusion between coating and substrate. The changes in this structure with exposure time and oxidation followed similar paths to the aluminide coatings but with certain important differences which suggest that the overlay coating affected zones might be more ductile and more stable.

The presence of the coating appeared to have no significant influence on the 850°C creep behaviour of either alloy. In addition, ageing of specimens at 850°C prior to testing to promote increased coating-substrate chemical interaction did not induce a significant reduction in creep life or show differences between coated and uncoated alloy systems. Penetration of these reaction phases into the alloy was marginally enhanced in grain boundaries.

All test pieces failed by propagation of cracks from the substrate alloy surface, following the development of cavities and small internal cracks on grain boundaries.
In the coated specimens, unstable crack growth usually began in the subcoat region, on a small number of grain boundaries. The complete absence of similar damage on the majority of subcoat boundaries suggests that cracking is related to boundary orientation, rather than to chemical interaction between coating and substrate.
In uncoated specimens, the onset of crack propagation was assisted by oxidation from the surface, along grain boundaries. Cracks appear to propagate only in the very last stages of creep life when non uniform straining (necking) is becoming pronounced. The high ductility of the coatings, both overlay and aluminide at 850°C is demonstrated by their ability to deform and accomodate the high local strains associated with major crack formation in the substrate alloy.

Work by a COST 50 collaborator indicated that the aluminide coating tested (LDC-2) becomes brittle at temperatures below 800°C. Creep tests were therefore conducted on the S57 overlay coating at temperatures down to 700°C without an indication of brittle behaviour being obtained. In order to assess the potential effect of the interdiffusion zone platelike phases on mechanical properties (other than creep-rupture) of coated IN738LC, thermal fatigue testing was carried out by cycling tapered disc-shaped (Glenny) test pieces between two fluidised beds held at different temperatures (950°C and 40°C). On the basis of visual observation, it appears that the application of LDC-2 and S57 LPPS coatings does not significantly influence the thermal fatigue damage, though with increasing holding time (3, 6, 12 min.) the onset of cracking seems to become noticeably enhanced.

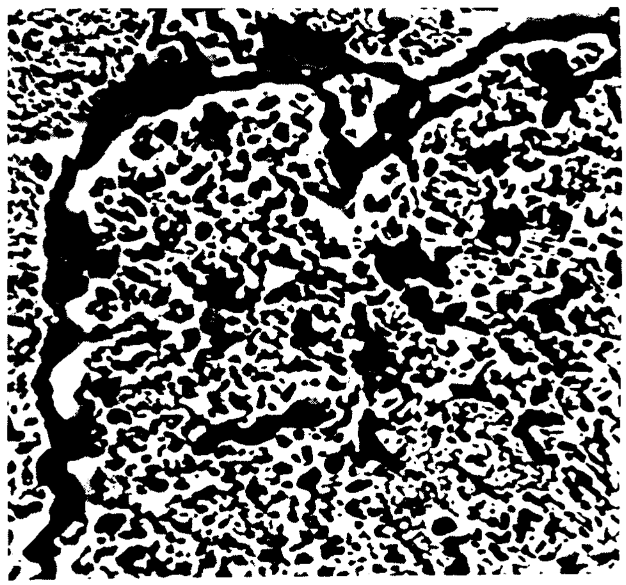


**Fig. 5 Inter- and intragranular creep damage in HK30. Material conditions: T = 1000°C $\sigma = 9$ N/mm$^{-2}$.**

## Creep of Tubular Components in Corrosive Environments

The link between basic research into the mechanisms of stress-environment interactions and the design information required in high temperature plant is further explored through simple sub-component tests on tubular Alloy 800H specimens. The development towards the eventual goal of combining complex multiaxial creep stress with environmental degradation proceeds along a route encompassing:

a) uniaxial testing of tubes in air;
b) internal pressure testing of tubes using argon;
c) uniaxial testing of tubes containing corrosive gas;
d) internal pressure testing using corrosive gas and finally;
e) combined internal pressure-axial loading with corrosive gas.

The tests performed for comparison with uniaxial properties obtained in air or inert gas have shown a considerably reduced ductility for the tubular specimens under both internal pressure and uniaxial stress. The stress rupture, however, appears only to be reduced during long term tests of around 1500 hours. The first uniaxial tests using a low pressure carburising environment were in principle used for endfeature design purposes but the type and extent of carburisation through the tube wall compared well with predictions from corrosion experiments.

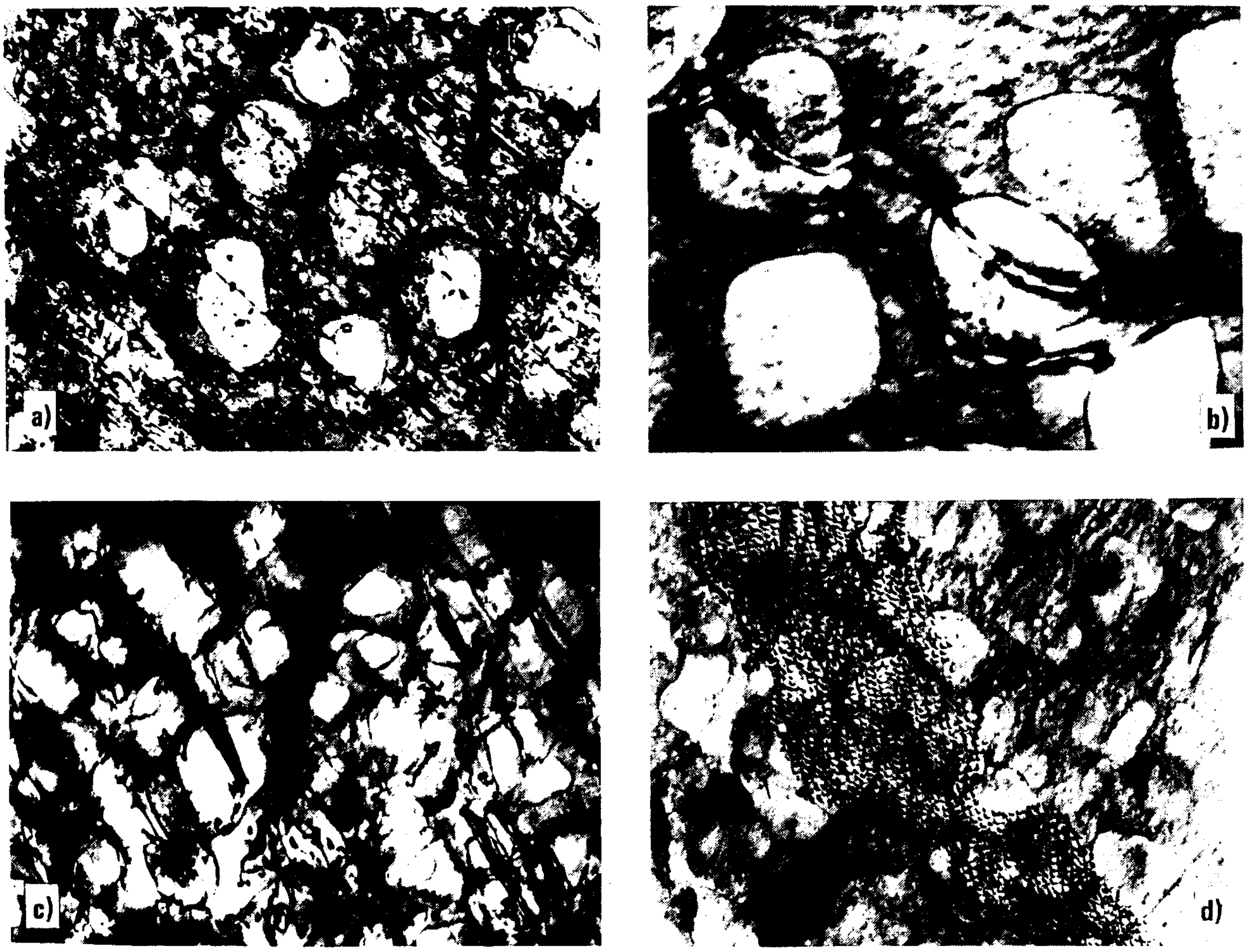



**Fig. 6 TEM micrographs illustrating dislocation mechanisms.**

a) dislocations moving in glide band and shearing of $\gamma'$precipitates at $\dot{\epsilon}_t = 5 . 10^{-5}s^{-1}$, $\epsilon_p = 0.67$ °/o. magn. 100.000x.
b) cutting mechanism of $\gamma'$precipitates by pairwise motion of dislocations at T = 823 K, $\dot{\epsilon}_t = 5 . 10^{-5}s^{-1}$, $\epsilon_p = 0.15$ °/o. magn. 130.000x.
c) cutting mechanism of $\gamma'$precipitates by single dislocation motion at T = 1003 K, $\dot{\epsilon}_t = 5 . 10^{-5}s^{-1}$, $\epsilon_p = 0.67$ °/o. magn. 100.000x
d) dislocation formation at T = 1003 K, $\dot{\epsilon}_t = 5 . 10^{-5}s^{-1}$, $\epsilon_p = 0.03$ °/o. magn. 60.000x

**Fig. 7 First experimental rig and concrete cells for tubular component testing.**

The termination of the design phase and commencement of construction of a facility for the multiaxial stress tests using corrosive gas as the pressurising medium formed the highest priority of the years' work. Nearly all infrastructural items such as four concrete test cells, control room, electrical power, cooling water, compressed air, ventilation and gas supply from a gas store have been supplied and installed. A prototype pressurising system is under construction for the internal pressure testing only of tubes in the first cell. The first high temperature creep tests on the alloy 800H tubes are planned during the next half year and tests with corrosive gas will follow once all the intricate safety procedures have been satisfied and the necessary systems installed.

### Study of a High Temperature Test Facility for Tubular Components

The large tubular component testing programme and facility proposed last year was used as the starting point for the elaboration of a specification for a smaller prototype sub-component testing facility as requested by the programme adivsory committee (ACPM). In this specification account has been taken of the existing programme of tubular sub-component testing (outlined above). The proposal covers the extension of this work to the investigation of the deformation behaviour of tubular sub-components under the action of a thermal gradient. A conceptual design of the required experimental rigs and a small programme for up to three of these test rigs has been derived which is considered most suited for maximising the output of information when carried out in conjunction with computational methods for predicting deformation behaviour during creep. Sufficient information is now available to incorporate the project specification in the next multiannual programme proposal of the High Temperature Materials Programme.

## 2.3 Data Bank

The programme pursues in the third project the implementation of a pilot data bank on mechanical and corrosive properties of materials relevant to energy conversion technologies. This data bank is planned to become a tool for research management and service to materials' users and producers. Such functions are now facilitated by the establishment of information networks like EURONET, which permit Community wide access to any type of base.

The programme has investigated the European interest for a data information system on high temperature materials and concluded that there is an interest for application-oriented, evaluated property data, which is at present not satisfied by any generally available information system existing in Europe. The project started work relating to HTM data with a restricted scope of alloys and properties. Materials and properties of primary concern are those investigated in the HTM programme, in particular Alloy 800H and tensile, creep and fatigue properties at $600^{\circ}C$ - $1000^{\circ}C$ in environments of C-O-H composition.

The data bank is developed in collaboration with the computing centre of the Ispra Establishment where the project finds support in terms of an available data base management system (ADABAS) and computer hardware.

Transfer of data between Ispra and Petten takes place by postal transfer of the data collection sheets and magnetic tapes. Direct transfer in both directions is established via the Euronet system.The preparation of a Data Bank Demonstration Book (DBDB) had been planned as a hand-processed action preceding the data bank operation. However, due to the rapid development of the computer based bank it has become possible to assemble it from genuine data bank output, albeit with some delay to the first plan.

The book will consist of two parts. The first part describes the character and quantity of data stored in the Data Bank at the time of publishing the DBDB. The second part demonstrates Data Bank processing features and show plots and tables with comments and explanations. The present data bank structure comprises the following seven files:

- test results
- specimens
- materials
- test conditions and methods
- source of data
- field definitions
- synonyms dictionary.

The data content is rapidly growing. Preliminary search procedures permit the retrieval and presentation of data in a simple format as shown below. The perfection of the output procedures is the main goal of the next period.

```
TYPE COMMAND
    find [all record(s) in file] test results with proof stress not = 0 and
    strain = 0.2.

ACCEPTED
    171 RECORDS FOUND HOLD WITH S01

TYPE COMMAND
    find [all records in file] test results with = (s01) coupled [to file]
    materials with element = carbon and concentration < 0.3.

ACCEPTED
    18 RECORDS FOUND HOLD WITH S02

TYPE COMMAND
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    proof stress, element, concentration.

ACCEPTED
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         000390213  2130000E+03                 .199999E-01
         000390214  1600000E+03                 .289999E-01
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**Fig. 8 Illustration of a search using preliminary procedures (only 3 of 18 results are shown).**

# 3. CONCLUSIONS

The HTM programme contributes to developments in the new energy conversion field through the provision of improved knowledge on materials science and technology. Its activities were executed in close contact with universities, industries and governmental bodies. The concerted European action (COST 50) provided the frame within which the gas turbine research work was carried out.

The Information Centre project was successful in supplying information on materials R & D and developing close contacts with national and industrial energy programmes making use of high temperature materials.

Dependable materials performance is critical for the operation of advanced energy technologies. The programme contributes to the satisfaction of the requirements by studying behavioural patterns of materials in specimen and component form which involve the use of the unique facility for high temperature testing in toxic and explosive atmospheres which represent those found in the industrial applications.

The results obtained have increased the understanding of materials behaviour, assist in the selection of high temperature materials for plant design and contribute to the development of new materials.

Engineering creep research on tubular Alloy 800H subcomponents has generated results for the (isolated) influences of axial load (uniaxial stress), industrial pressure (imposed multiaxial stress) and corrosive gaseous environment (structure degradation). The construction of the facility which is needed to combine these factors in more complex - and rather hazardous - tests is well advanced and will permit the first experiments to be made in 1982.

The data bank development has proceeded to a status which enables the first experimental output operations to be performed. Plans are made to apply these to the preparation of a "Data Bank Demonstration Book" which is based on the available data bank content compiled from Alloy 800 literature.

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