COMMISSION OF THE EUROPEAN COMMUNITIES
JOINT RESEARCH CENTRE
Petten Establishment
The Netherlands

High Temperature Materials Programme 1980 - 1983

Technical Description

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HIGH TEMPERATURE MATERIALS

Abstract

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A 'High Temperature Materials Information Centre' collects, disseminates and exchanges information on high temperature materials through the organisation of training courses, conferences, collequia, surveying studies and inquiries.
'Materials and Engineering Studies' are experimental investigations of the interaction between mechanical of materials and their working environments in high temperature service with particular attention to the conditions of coal conversion processes.
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INTRODUCTION

The High Temperature Materials Programme was initated at the Joint Research Centre Petten in 1975 in support of the 'Future Forms of Energy' policy of the C.E.C. Initially the existing state of knowledge and the industrial and research activities in the field were critically surveyed in contact with selected European industrial and academic experts, and from this survey the so-called White-Book - 'Review of the Technological Requirements for High-Temperature Materials R & D' (EUR 5623) resulted. This publication and the continuing contact with the relevant industrial and research organisations formed the basis for the programme operated during the period 1977-1980. The results and achievements arising from the work have been described in the Programme Progress Reports issued every 6 months and in numerous publications.

The new programme now presented to cover the period 1980-1983 had been developed in continuation of the competence acquired and in the light of the response received from European organisations to the actions taken during the previous period. The programme strategy is a combined approach to survey technologichal and industrial requirements in applications ranging from high temperature petrochemistry to thermonuclear fusion in order to identify future needs, to establish and perform research projects in selected key areas and finally to build and maintain multilateral communications with manufacturers, users of these materials and with the research and development sector.

The contacts and inquiries made have shown that the availability of adequate materials for the operating and developing high temperature processes had a critical level of importance. Increasing demand for better processes and higher efficiencies is a logical consequence of the economic constraints caused by the changing pattern of energy resources, and the technical response to this demand necessarily involves higher temperatures and more difficult material requirements. High temperature materials problems are therefore found in a wide range of applications; the most presently the most important group being the various developing coal conversion processes. Furthermore high temperature petrochemistry, nuclear process heat, solar power and even recycling and incineration plant.

Three closely connected projects, a **high temperature materials information centre, materials and engineering studies** and a **data bank** are together able to collect and disseminate information and encourage research in the Community by focussing attention on particular problems.

The actions of 'Meeting Point Petten' which are concerned with the exchange and dissemination of information on high-temperature materials have received general approval. Major conferences organised either by Petten alone or in collaboration with national or international bodies have been invariably succesful; invited groups of experts have attended colloquia on specialised aspects of high-temperature materials science and technology; instructional courses organised by Petten and using outside experts lecturers have been well attended. The current proposals envisage a continuation and extension of these activities into three areas:

- Information exchange and transfer
- Information collection
- Information storage.

The general approach to study the interaction between the mechanical properties of materials designed for high-temperature service and their working environments is continued with particular focus on environments pertaining to coal conversion and utilisation processes. Consequently the programme will consider such materials, atmospheres and stresses as are appropriate to coal gasification, fluidised bed combustion, power turbine and related technologies. Highlights of this project may be summarised as:

- Studies to evaluate the mechanical properties at high temperature of materials relevant to coal conversion processing in corrosive environments designed to provide the separate and conjoint actions of carburising, oxidising and sulphidising gases. The behaviour of materials subjected to static and fluctuating stress states of both uniaxial and mutiaxial nature are envisaged.
- The determination of the corrosion behaviour of these materials in corresponding environments under both stressed and unstressed conditions.
- The assessment of protection against corrosion conferred upon materials by the development of suitable corrosion-resistant scales either by variation of material condition, or by the application of surface coatings.

As far as possible these activities will be integrated with appropriate European cooperative research actions such as for example, the proposed COST 501 exercise, thereby continuing the principle established by participation in the current COST 50 programme on gas turbine materials.

The world-wide generation of data on high-temperature materials by manufacturers, users and research institutes makes it extremely difficult to keep an up-to-date record of validated data and to avoid unnecessary duplication of tests. The need for a data bank of materials properties has been confirmed and steps are proposed to initiate this using a single alloy of current importance, followed by progressive actions leading to a data bank of validated information on a selected range of high-temperature materials.

Projects

Project 1: Information Center

Background

The Petten JRC Establishment has among its tasks a remit to correlate high-temperature materials research activities, to act as a coordinating element and to provide an information service on current research into high-temperature materials carried out in European public institutions and industrial laboratories. In order to expedite these tasks and to promote communication between Petten and the European research organisations, a project entitled Information Centre has been established.

Objectives

The objectives of the project are the provision of an information service to the European high-temperature materials community and the encouragement of cooperative actions. The scope envisaged for the information service ranges from the organisation of small informal discussion meetings for verbal information exchange to the establishment of comprehensive, fully computerised data acquisition, processing and documentation systems; while the promotion of research coordination and cooperation within Europe requires a detailed understanding of the relevant scientific and economic factors governing the HTM research activities of the community as a whole.

Sub-structure of the project

In order to provide the necessary information requirements to meet these objectives, the Information Centre has undertaken three separate activities which are defined under the titles:
- 1.1. Information Exchange and Transfer
- 1.2. Information Collection
- 1.3. Information Storage.

Activity 1.1.: Information Exchange and Transfer

Objective

Building upon the experience gained with the Meeting Point project during the 1977/79 programme period, this activity will further follow the objective to promote on a European basis the exchange of information in matters relating to high-temperature materials through the organisation of conferences, symposia and colloquia. Established knowledge will be transferred through training courses and seminars.

Procedures

Three different types of meeting will be organised:

Conferences (symposia)
- At two yearly intervals an international conference at Petten will be held, which will be principally concerned with the behaviour of alloys in HT environments.

Colloquia (workshops)
- annually at least one colloquium dealing with specific HT areas will be organised. For the spring of 1981 a 'European colloquium on the behaviour of joints in HTM' is planned.

Courses, Seminars.
- an annual scheme 2/3 courses/seminars on relevant areas of materials behaviour will be operated.

Activity 1.2.: Information Collection

Background

The preparation of an HTM White Book under the title 'Review of the Technological Requirements for HTM R & D' (EUR 5623) has provided a starting point for information collection and the definition of future research needs. This compilation will be periodically up-dated.

Objective

The central aspect of this activity is the collection of information concerning:
- HT processes, applications and materials,
- on-going R & D activities,
- future R & D requirements

through the execution of surveys, inquiries and studies.

Procedures

The basis for updating of the compilation is provided by surveys of or inquiries to relevant research institutions. Supplements to the White Book will be commissioned principally to include new areas of material applications in high-temperature technologies e.g. urban waste incineration, process turbines, solar tower and fusion.

In particular, are envisaged:
- Surveys: HT materials in solar tower technology, fuel processing, waste, residual oil and biomasse-conversion
- Inquiries: mechanical properties, advanced ceramics, erosion, substitution, surface protection, alloy manufacturing.

- Studies:
  - Analysis of material requirements for future energy conversion processes in high-temperature technology
  - Potential of coatings for corrosion protection in increased HT applications
  - Innovation transfer in HT technologies, mechanisms and improvements
  - HTM standards, codes of practice (status and needs).

Activity 1.3.: Information storage

Background

The 'Central Information Index' (CII) is conceived as a tool for information processing to provide for the needs of activities 1.1. and 1.2. The principal basis of the CII will be for the time being, a list of European organisations which have direct interests in the field of HTM, — of which there are already approximately 1500. The CII will be gradually developed as a permanent inventory of up-to-date information on HTM-relevant on-going research project and on organizations, active in the HTM field.

Objectives

i) To establish a means of effective treatment of collected scientific and economic information concerning European research activities either current or proposed in order to identify areas where co-operation should be promoted and research be stimulated

ii) to provide a directory of the available research potential inside the European HTM community.

Procedures

Information processing and storage of this nature is conveniently handled using computers. Therefore, appropriate hard- and software solutions are being investigated in order to develop a data-base like system or matrix-oriented information files. 'Matrix oriented information' is information associated with or collected under a specific heading which can be a specific research activity, on a specific organisation or a particular individual. Research activity oriented information is usually being gathered by means of inquiries into specific aspects of European HTM research.

This involves the organisation of all information received from the various institutions into a number of directories, (held in the computer storage) from which information in a specific area may be selectively extracted. A typical set of directories would be:

i) List of research projects
ii) List of research organisations
iii) List of responsibles for research
iv) List of scope descriptors (field of activity).

The main file here is the list of Research Projects, since the other information is only subsequent to this and could be presented in the form of index-lists to the main file. Organization-oriented information requires the establishment of a second main file and also does the individuel-oriented information.

Project 2: Materials and Engineering Studies

Background

In recent years there has been a considerable revival of interest in coal utilisation technology as the result of the increasing awareness throughout the world that supplies of oil and natural gas are rapidly dimishing whereas coal supplies are plentiful. This has prompted considerable effort to develop more advanced coal processing techniques. In most cases the main objective is to convert coal into forms of energy which are more convenient and clean to use; e.g. electricity, gaseous and liquid fuels and chemical feedstocks. As the same time the process must operate in an environmentally acceptable manner and should attain the highest possible conversion efficiency. The result of this is that plant components and construction materials are exposed to very aggressive environments at very high temperatures.

Similar types of corrosive environment are found in a number of high temperature petrochemical processes where the severity of the atmosphere is increasing as a result of the drive to higher operating temperatures for improved efficiency, and also to lower grade fuels for economy. However, the environments found in the fluidised bed combustor and various types of coal gasifier are liable to be even more severe than those met in petrochemical and HTR applications. In fact, for some gasifier applications it appears that high temperature alloys will only survive if they are covered with a special corrosion-resistant coating.

In the different power systems, the gas turbine plays an important part. For instance, low or medium calorific value gas from a gasifier would be burned in a combined cycle plant with a gas turbine followed by a steam turbine to obtain maximum generation efficiency. In the pressurized fluidized-bed combustion process, the combustion gases can be expanded through a turbine.
High pressures are frequently used in high-temperature petrochemical processes not only for the effect on thermodynamics but also for the effect on the kinetics and heat transfer rates of the reaction taking place. High pressure also provides a means of achieving longer residence time in a given vessel volume. The gaseous reaction products are frequently expanded through a turbine, to produce useful power while being depressurised.

The use of nuclear heat for process purposes involves the environments and materials problems of the petrochemical or coal-gasification industries with added requirements of reactor helium type corrosion in some plant items plus nuclear levels of reliability and safety.

Incineration processes and recycling of materials provide further examples of materials usage in aggressive environments. During the recycling pyrolysis of auto tyres etc, for instance, temperatures of 700-900°C are attained in an environment which has low oxygen level and high activity of carbon and sulphur.

A common feature of the numerous plant components in these various industries is the requirement for them to operate at temperatures in the range 700-1100°C for long periods under stress in aggressive environments which mostly have a restricted oxidation potential. High service temperature and long times promote not only microstructural changes, as for example coarsening or solution of precipitates, but in addition environmentally induced corrosion may cause microstructural changes brought about by diffusion of foreign elements into the bulk and by surface scale formation, indicating the need for an understanding of the kinetics and mechanisms of these materials changes. The superimposition of stress will result in an interaction between deformation/fracture processes and the microstructural changes resulting from the high-temperature corrosive environment makes their systematic evaluation essential in order to achieve further improvement in material usage in the technological applications considered, in terms of safety, reliability and economics of plant.
At present, design techniques for preventing failure of structures operating at high temperature are based upon a number of criteria which place un upper limit on the load and deformation allowed in steady and changing load situations. Assessment of these criteria requires knowledge of material behaviour under conditions typical for the application, but in many cases few data pertaining to the situation exist due to time and cost limitations. As stated above, depending on the stress-temperature, environment, combinations, a given material will exhibit different deformation, corrosion and fracture mechanisms. Empirical adaptation of test data from one set of environmental conditions to another will therefore be reliable only, provided that the extrapolation does not cross boundaries between mechanism fields. Knowledge of damage mechanisms in the types of environment found in the industrial applications and mechanism field boundaries (boundaries between different stress temperature-environment conditions) is thus essential in order for the design to be safe, reliable and not over-conservative. This project aims to provide assistance of this type.

In order to utilise this information, which has been gained from materials tests to provide a source of advice, directly applicable to design engineer or plant operator, an intermediate range of engineering studies must be interposed. This range of studies has been narrowed in this project to research on those parameters which were thought to be critical in linking materials investigations to plant design and operation. The most important of these parameters concerns materials behaviour in component form as distinct from simple test-bar form, and of the many potential stages and geometries presently encountered in high-temperature processes the tubular geometry was chosen as having the highest importance and widest application.

Accordingly the project is organised in the from of two part projects. 'Materials Studies' concentrate on the understanding of the mechanisms and kinetics of corrosion and protection on base material and coatings as well as the deformation modes and fracture mechanisms in creep and fatigue. 'Engineering Studies' investigates the mutiaxial creep behaviour of tubular test pieces under corrosive attack and also includes the specification and feasibility study of an advanced high temperature test facility for tubular components.

Part Project 2.A.: Materials Studies

Objective

The aim of the Materials Studies Part Project is to understand the mechanisms governing the behaviour of selected metallic alloys in certain types of corrosive environments at high temperatures. The work will seek to describe the parameters affecting time-dependent mechanical behaviour and the kinetics of the corrosion which is occuring in order to assist lift-time prediction studies.

Environments

Examination of the atmospheres associated with the different application areas shows that many have low partial pressures of oxygen. In the petrochemical and HTR plants, carburisation is a possibility, the extent depending on the protection offered by a surface scale of the alloy. Generally, in the coal conversion processes, sulphur compounds are also present, sometimes with a dominating influence. For the gas turbines which may be associated with the processes, the oxygen level will depend on the particular application but, in som cases low partial pressures are again found.

The various test environments which will be used in the course of the laboratory studies will be characterised in terms of contents of the reacting species, carbon, oxygen or sulphur rather than by being defined by specific gas compositions. Thus, a methane reformer gas mixture with a ratio H_2O:CH_4 of 3:1 has an activity of carbon a_c=0.3, and partial pressure of oxygen, $p_{O_2}=10^{-20}$ atm., under typical operating conditions.

The group of test environment are given in Table 1.

Table 1.

Type	Gas mixture	Activity of corrodents
Carburising	H_2/CH_4	a_c = 0.3 - 0.8.
Carburising/ oxidising	$H_2/CH_4/CO/CO_2/$ H_2O	a_c = 0.3 - 0.8. $p_{O_2} = 10^{-14} - 10^{-21}$ atm.
Sulphidising	H_2/H_2S	$p_{S_2} = 10^{-4} - 10^{-15}$ atm.
Carburising/ oxidising sulphidising.	$H_2CH_4/CO/CO_2/$ H_2O/H_2S (obtained by adding H_2S to the carburising/oxidising gas mixture)	One set of activities derived from the ranges given above.

In most activities the test atmosphere will be at a pressure slightly above atmosphere, i.e. 1-2 bar absolute, with a gas flow rate of the order of 10 l/h.

During the programme period it is planned that the majority of the effort will be found in carburising and carburising/ oxidising type environments. Sulphur will be added to the environmental range for certain activities as the programme develops.

Materials

To meet the requirements of the industrial applications outlined above it is necessary to consider both iron- and nickel-base alloys. In the former group, certain alloys can be identified which are common to a wide range of applications. These include the alloys which form the core of the project work:

Alloy 800 H (22Cr, 33Ni, Ti, Al)—wrought
ACI type
HP 40 + Nb (25Cr, 35Ni, Nb) —centrifugal casting.
(Werkstoffe 1.4852)

Where appropriate to particular activities certain derivatives of these alloys (e.g. Alloy 802) will be added. Model alloys with precisely controlled composition will also be needed for some areas of investigation. Related alloys (e.g. HK40) which have been in use in the 1977/79 programme will be found necessary for some series of experiments.

Wrought Ni-base alloys are candidates for certain components in coal conversion, N.P.H. and gas-turbine areas and cast Ni-base alloys are essential for gas-turbine blading. A representative selection will be made following further exploration of the materials problems which have to be overcome in these applications.

The use of austenitic iron-base or nickel base alloys in coal-gasification atmospheres may require their protection by a suitable coating. One area of activity will explore features associated with the corrosion behaviour and substrate compatability of some representative surface protection layers.

Material condition

The alloys will be tested in the condition of heat treatment in which they are normally used. Thus for the centrifugally cast austenitic steels specimens will be machined axially from the 15-20 mm wall-thickness tubes and tested in the 'ascast' condition. The wrought steels will be obtained as 15-20 mm dia bar and will be heat treated as recommended, e.g. solution treatment. This heat treatment will normally be carried out in the laboratory in order to obtain maximum consistency between samples. The nickel-base alloys will be obtained as wrought bar or precision castings, depending on the grade finally selected, and will be tested after solution treatment or full solution + aging as recommended for normal application of the particular alloy grade.

Collaboration with COST

In 1978 the JRC became a member of COST (European Cooperation in the field of Science and Technology) and more specifically of COST action 50 on gas turbine materials. Suitable parts of the 1977/79 programme were proposed and accepted as contributions to Round II, the activity which was then in progress. These collaborative contributions from the HTM programme started in October and December 1978, each with a duration of 2.5 years and, having been approved by ACPM, continue as contributions from the 1980/83 programme until their completion in 1981. They are respectively the sub-activities 2.A.3.3. and 2.A.2.4..

COST 50 Round II officially started in mid 77 and hence terminates mid 1980. A further Round III is being organised to act as a bridge until a new action, COST 501, can be established. Following requests from our collaborators, two other pieces of work which relate to these Round II contributions and which fall inside the total committment of effort have been proposed, with a finishing date of 1981. They are the sub-activities 2.A.2.5. and 2.A.3.4.. In addition to their direct relevance to gas turbine technology they are useful for other main aims of the project.

By 1981 it is anticipated that COST 501 on 'High Temperature Materials for Energy Conversion System using Fossil Fuels' will be operational and that a number of activities in this project will be suitable for cooperative work with other participants within this framework.

Activity 2.A.1.: Mechanical properties under static loads in corrosive environments

Objective

To achieve a mechanistic understanding of the behaviour of selected metallic materials under creep deformation conditions in gaseous corrosive environments at elevated temperature and in doing so, to provide engineering data.

Test procedure and conditions

Creep tests, which will usually extend to rupture, will be performed, normally, in single-specimen machines where the strain is measured in-situ, thus providing information for structure/mechanical property relationship and also data for integration into life-time prediction studies. Where necessary for the understanding of the creep behaviour, pre-exposure of test pieces to corrosion, particularly by carburisation, will be carried out, with or without load.

Material

The investigation will be carried out initially on the austenitic steels.

HK 40	(centrifugically cast pipe)
Alloy 800 H	)
Alloy 802	) (wrought bar)

To this group will be added the centrifugally cast steel of the type HP 40 + Nb and perhaps the version with additions of Tungsten. It is planned during the programme period to start testing a selected nickel-base alloy.

The test piece diameter is usually 6 mm. A consistent surface condition will be obtained by grinding.

Environment

a) Air atmosphere

In order to gain the necessary reference data for the casts being used, some creep tests have to be carried out beforehand in air which is chosen as the 'reference environment' in order to position these materials with respect to the literature data.

b) Special environments

These will be selected from the types shown in Project 2, Table 1. Further details are given for each sub-activity.

Temperature

Temperatures for each sub-activity will be selected within the range 800°C-1100°C in 100°C steps with special attention to tests at 1000°C.

Duration

The creep experiments will cover a broad range of testing times, starting with short-term tests of about 50 h and going up to long term tests of about 5000-8000 h. Depending on the test capacity a few tests exceeding 8000 h are planned.

Analysis

Structural studies on tested samples will include the following main subjects:

a) Identification of phases formed by corrosive attack using: chemical analyses, X-ray diffraction, electron probe microanalyses, ESCA.
b) Fracture mechanisms will be studied by: optical microscopy, scanning electron microscopy, transmission electron microscopy.

Sub-structure of activity

The work of the activity is divided into a number of clearly defined areas of investigation or 'sub-activities' which will be studied using the above test procedure. Their titles are:

2.A.1.1. The influence of carburisation on creep properties.
2.A.1.2. The influence of the combined attack of carburising/oxidising atmospheres on creep behaviour.
2.A.1.3. The influence of the combined attack of sulphidising/oxidising/carburising gases on creep properties.

Sub-activity 2.A.1.1.: The influence of carburisation on creep properties

Aim

To identify the influence of various test parameters on the creep and stress-rupture behaviour of different austenitic alloys during exposure to a purely carburizing environment and to explore the interactions between structure and mechanical properties.

Particular aspects of the test procedures and conditions

Most of the experiments will be carried out with a carbon activity $a_c = 0.8$. A final decision about the necessity to extend the test programme to a different carbon activity will be made on the basis of results gained on $a_c = 0.8$.

Sub-activity 2.A.1.2.: The influence of the combined attack of carburising/oxidising atmosphere

Aim

To study the consequences on creep deformation and stress-rupture behaviour of attack by a complex atmosphere with both oxidation and carburisation potential and to describe the structural/mechanical properties relationship.

Particular aspects of the test procedure and conditions

The initial environment to be selected will h ave $p_{O_2} = 10^{-20}$ atm. and probably $a_c = 0.3$. The use of other levels of the corrodants will depend on the results obtained here and on information from 'Corrosion without load'. (Activity 2.A.4.). It is expected that testing will start with Alloy 802 and HP 40 + Nb. Other materials will be added as needed to meet the objective of understanding the mechanism of creep/environment interaction.

Sub-activity 2.A.1.3.: The influence of the combined attack of sulphidising / oxidising / carburising gases on creep properties

Aim

To study the influence of sulphur attack under simultaneous oxidation/carburisation on the creep and stress-rupture behaviour of austenitic steels and to establish the structure/mechanical properties interaction.

Particular aspects of the test procedure and conditions

Modifications to the test facilities will be required and information from the corrosion and protection activities will be used to assist a decision about test rig materials. Only one material is expected to be tested in this work and the aims are not expected to be fully realised before the end of 1983.

The environment will be obtained by adding H_2S to a suitable gas mixture used in Sub-activity 2.A.1.2..

Activity 2.A.2.: Mechanical properties under dynamic loads in corrosive environments

Background

A reasonable level of knowledge already exists concerning the behaviour of materials and the mechanisms operating under static loads in oxidising environments. With regard to the varying load situation, though much more frequently encountered in practice, the understanding is much less advanced. Depending on the application, the varying load situation results in purely cycle-dependent or in time-dependent fatigue. The time-dependent processes that may play a role in the latter case are creep deformation, corrosion and possibly strain ageing. The introduction of aggressive corrosive environments complicates the situation even more. The resulting situation is characterized by a multitude of simultaneously operating processes in the material which may or may not mutually interact. Hence the need systematically to evaluate material behaviour and establish the field boundaries within which the various mechanistic processes operate.

Objective

To generate data which may serve as a baseline for design. More importantly, to identify deformation, corrosion and fracture mechanisms, to understand the physical processes involved and to derive quantitative information on material degradation in order to improve phenomenology-based methods for life-time prediction of materials subjected to cycle- and time-dependent fatigue in corrosive environments.

Sub-structure of activity

The work of the activity is divided into a number of clearly defined areas of investigation or 'sub-activities'.

They are:
2.A.2.1. Development of fatigue testing equipment for corrosive environments.
2.A.2.2 HTLCF data generation in corrosive environments.
2.A.2.3. Deformation and fracture mechanisms in HTLCF in corrosive environments.
2.A.2.4. Effect of creep-fatigue interaction on the life of nickel-base superalloys.
2.A.2.5. Crack initiation in high-temperature LCF.

The work of the last two sub-activities forms part of the contribution from the HTM programme to COST 50, as outlined in the Part Project Introduction; sub-activity 2.A.2 4. being linked to Round II and 2.A.2.5. to Round III.

Sub-activity 2.A.2.1.: Development of fatique testing equipment for corrosive environments

Aim

To transform existing universal testing machines into fatigue test units for testing in corrosive environments at elevated temperature.

Procedure

This is a two-phase activity. In the first phase a 100 kN servohydraulic MTS testing machine which is available, will be equipped with an environmental test chamber. It is planned that the test chamber will be composed of an external unit which can be used on its own for tests under vacuum or under inert gases, or in combination with a smaller chamber surrounding the sample for testing under agrressive corrosive gases In the latter case the larger chamber will serve as a safety box. A number of test technique problems exist; specifically temperature and strain measurement under corrosive gas, gas preconditioning, data acquisition, linking up with the gas supply system in the ETL etc..

In the second phase a 100 kN, electromechanical Schenck-Trebel testing machine will be equipped for testing at high temperatures under corrosive gases, taking into account the experience gained in phase 1

One of the machines will also be equipped for the measurement of crack propagation and the technique calibrated for use in corrosive environments.

Sub-activity 2.A.2.2.: HTLCF data generation in corrosion environments

Aim

To characterise the cycle- and time-dependent fatigue behaviour of the materials selected in the reference and corrosive environments.

Work programme

- This sub-activity essentially relates to the generation of a set of baseline data.
- Monotonic and cyclic stress-strain characteristics will be derived to describe the short-time mechanical material behaviour under time-independent deformation conditions.
- Short-time creep tests are included as a part of the study of the material response to creep-fatigue and other time-dependent mechanical deformation.
- Fatigue will be studied primarily through endurance testing on 6 mm. diameter smooth samples whereby the resultant cyclic hardening/softening behaviour will be recorded. Because crack initiation and crack propagation are differently affected by environment, crack-propagation rates will be measured as well as life to fracture

Test procedures and conditions

Material

Work will start on the project core material Alloy 800H. In a later phase tests on material of the type HP 40 + Nb and on a nickel-base alloy will be considered as described in the introduction to Part Project 2A.

Environment

Exploration of these variables will limit the work principally to a single environmental system i.e. a carburising, oxidising environment as described in table 1

Since fatigue data generated in oxidising environments such as air have been published in literature, air is chosen as a reference environment. A limited number of tests in air is therefore planned to complement available data at the high test temperatures envisaged.

Temperature

The test temperatures will be in a range of 600°C-1100/C.

Loading

- Strain- and stress-ranges will be varied inorder to measure the dependence of them of hardening/softening rates and of cyclic life on the one hand, and of the crack-propagation rates on the other band.

- The effects of time-deependent deformation processes will be studied by performing tests at different total strain and stress rates (or test frequencies) in order to generate a cyclic-life versus test-frequency characteristic at selected values of the strain-range and the time-dependency of crack propagation rates respectively. Hold times may be further introduced in order to substantiate these results.
- Generally the conditions will be such that the maximum test duration will be 1500 h.

Sub-activity 2.A.2.3.: Deformation and fracture mechanisms in HTLCF in corrosive environments.

Aim

To understand the deformation and fracture mechanisms which lead to the observed alloy behaviour in order to ensure at least qualitative applicability of the phenomenological conclusions over a range of conditions where the same mechanistic processes are found. Conclusions drawn on the basis of purely phenomenological data are limited in applicability to the test situation used. Identification of deformation and fracture modes and understanding of the mechanisms governing the material behaviour are needed to allow interpretation of these conclusions in the broader context of existing knowledge, thus widening their meaning.

Information gathered from these investigations will allow interpretation of the quantitative mechanical test data (sub-activity 2.A.2.2.) in terms of the mechanisms governing alloy behaviour.

Procedure

The procedure will be to subject a selected set of test samples from sub-activity 2.A.2.2., both after having reached a fraction of their lives and after failure, to a range of structural analysis techniques in order to:

a. identify deformation and fracture modes and their dependence on selected combinations of the test variables mentioned in sub-activity 2.A.2.2., both in the reference and corrosive environments. This will be achieved by SEM inspection of fracture surfaces to define the mode of crack propagation, by optical microscopy to identify crack character and to measure crack densities, and by TEM to study the deformation processes.
b. gain an understanding of the mechanisms causing the observed behaviour. These mechanisms are the final result of mutually interactive processes such as diffusional transport of corrosive elements, precipitation and precipitate ripening, and time-independent and creep deformation. In order to unravel the role played by these processes in the deformation and fracture mechanisms, the information gained at sub point (a) will be complemented by performing selected experiments to study these processes in a more isolated way. In addition the mechanistic understanding reached in activities 2 A.1 (creep) 2 A.4. and 2.A.5. (corrosion without load and under load) is obviously essential to the present activity.

Sub-activity 2.A.2.4.: Effect of creep-fatique interaction on the life of nickel-base superalloys.

Specific background

In this sub-activity, attention is focussed on the question whether time-dependent deformation is of concern for and interacts with low-cyle fatigue in the case of gas-turbine disc alloys which are of concern to COST 50 participants. Moreover it is considered that crack initiation, which may occupy an important fraction of life under conditions of limited plastic strain amplitudes in essentially flawfree components, deserves more attention. Since crack initiation strongly depends on material parameters, its quantitative description is of importance to studies aiming at an improvement of LCF properties through optimisation of the microstructure. (See also sub-activity 2.A.2.5.).

Aim

The aim of this sub-activity therefore is to characterise the HTLCF behaviour in terms of microstructural damage and to assess the results obtained in terms of the mechanisms of failure in order to contribute to improved methods of predicting fracture due to time-dependent LCF.

Test procedure and conditions

Material

The following may be used: Waspaloy and argon-atomized powder metallurgical (PM). Astroloy in commercial and experimental heat-treated conditions respectively.

Environment

The testing sequence adopted is similar to, though somewhat less complex than, that for sub-activity 2.A.2.2. since all data are generated in an air environment.

Temperature

Temperatures are in the range 400°C to 800°C.

Loading

LCF endurance tests are mostly performed in total-strainrate control between plastic strain limits. These limits are in the range 10^{-2} down to approximately 2.10^{-4}. Time-dependent deformation is introduced by lowering the strain rate. Strain rates in the range $10^{-2}s^{-1}$ down to $2.10^{-6}s^{-1}$ are investigated.

Analysis

Mechanistic understanding is achieved through correlation of the mechanical test data with information on deformation and fracture modes, obtained by structural analysis of failed samples, in much the same way as already indicated in sub-activity 2.A.2.3.

Sub-activity 2.A.2.5.: Crack initiation in high-temperature LCF

Aim

To characterise and quantify crack initiation in time-dependent LCF in a nickel-base gas-turbine disc alloy.

Test procedure and conditions

Material

A single material, P/M Astroloy will be investigated, basically in the as-HIP'ed and heat-treated conditions, if samples become available through COST 50 round III collaborators, other microstructures obtained via hot working (uniform grain size and necklace-type grain structures) will be included.

Temperature

Tests will be carried out at 550°C and possibly at 650°C.

Loading

Baseline data will be generated in sub-activity 2.A.2.4.. The effect of both strain range and of strain rate on crack initiation will be investigated.

Analysis

Tests will be interrupted at regular intervals to inspect the sample surface for cracks by means of replicas which will be analyzed using optical microscopy, SEM and TEM.

Activity 2.A.3.: Surface protection; scales and coatings

Background

The use of all metallic materials for applications at high temperature in aggressive environments requires the formation and preservation of a protective surface oxide layer or scale to act as a barrier

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 constraints seen in service have to be used. The protective action of these coatings against corrosion is related to the development of their natural scales. Hence, the application of coatings on substrate materials means that we are dealing with a composite material where the substrate will provide the necessary strength and the coating the required resistance to the environmental attack.

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 which can detrimentally affect the protective ability of the coating and the mechanical properties of the substrate.

Objective

To establish an understanding of the chemical and mechanical properties of surface oxides and coatings in order to allow selection of alloy/coating/oxide systems which are capable of providing extended service in high-temperature engineering applications with corrosive environments.

Sub-structure of activity

The work of the activity is divided into a number of clearly defined areas of investigation or 'sub-activities'.
They are:

2.A.3.1. Properties of scales formed on austenitic steels.
2.A.3.2. Protective coatings on austenitic steels.
2.A.3.3. The effect of coatings on the high temperature mechanical properties of some nickel-base alloys.
2.A.3.4. Grain boundary segregation in a nickel-base alloy.

The work of the last two sub-activities forms part of the contribution from the HTM programme to COST 50, as outlined in the Part Project Introduction; sub-activity 2.A.3.3. being linked to Round II and 2.A.3.4. to Round III.

Sub-activity 2.A.3.1.: Properties of scales formed on austenitic steels

Aim

To assess the parameters which affect the adhesive properties of natural scales growing on typical austenitic alloys.

Work programme

The properties of the scales will be assessed in terms of their cracking and spalling modes by defined thermal, and possibly mechanical, loading. They will be related to the oxide growth and to test conditions. The influence of controlled variations of certain alloying elements such as yttrium and/or silicon and of surface pre-treatments will be examined.

Test procedure and conditions

Materials

The following may be selected: Model alloys of IN519 with different Si/Y additions
Alloy 800H
HP 40 + Nb

Environment

Reference testing in air.
Tests in the carburising/oxidising gases shown in table 1.

Temperature

850° - 1050°C.

Duration

Ranges between 50 and 500 h.

Facilities

Tests will be conducted using: thermogravimetry, resonance vibration, acoustic emission, constant strain rate tensile testing.

Analysis

Samples will be analysed by hot-stage microscopy, optical microscopy, XRD, SEM and EPMA.

Sub-activity 2.A.3.2.: Protective coatings on austenitic steels

Aim

To evaluate appropriate coating materials and processes, to assess the protective nature and properties of applied coatings, together with possible effects on the mechanical properties of the substrate material.

Work programme

A literature survey will be carried out on the nature of the various corrosion mechanisms encountered in coal-conversion plants and information obtained from activities 2.A.1. and 2.A.4. in order to select appropriate coating/substrate systems.

A next step will be to obtain samples of representatieve coatings from external collaborating institutes. The structural and compositional stability of the coatings and their interaction with the substrate will be assessed. Furthermore, there will be an evaluation of their resistance to carburizing, carburising/oxidising and finally sulphidising/oxidising/carburising environments. The behaviour in aggressive environments of pre-grown oxide scales on such coatings will form part of the investigation.

Subsequently, it is intended that the effect of creep stress on coating integrity and stability should be explored in the same corrosive environments.

Finally, towards the end of the present programme it is intended to study the behaviour of coated alloys under dynamic load, i.e. low-cycle fatigue and thermal-shock/fatigue.

Test procedure and conditions

Materials

a) Substrate:
Alloy 800H
HP 40 + Nb

b) Coatings:
The present choice would be:
Fe Cr Al (Y) applied by different techniques, e.g. overlays (LP-plasma spraying, ion sputtering, PVD), CVD (pack processes), plus massive (roll bonded) Fe Cr Al Y.

Note: Coatings will be supplied by external collaborators.

Environment

Initially structural stability testing will take place in air. Corrosion testing and also most of the creep testing will be carried out in the range of environments described in table 1.

Temperature

Stability, corrosion and creep tests will be carried out in the temperature range 800-1100°C.

Duration

100 h - 3000 h.

Facilities

- Thermogravimetry and magnetic response measurements.
- Tensile creep testing.
- Thermal-shock/fatigue using fluidised beds.

Analysis

- Full range of structural analysis techniques including optical microscopy, XRD, SEM, EPMA, TEM.

Sub-activity 2.A.3.3.: The effect of coatings on the high-temperature mechanical properties of some nickel-base alloys

Aim

To evaluate the mechanism and kinetics of the degradation of overlay-type MeCrAlY-coatings in comparison to pack-aluminide-type coatings, focussing on the structural changes found at the coating/substrate interface. In particular, to assess any effect on the creep and thermal-fatigue properties of the substrate alloys.

Work programme

The study is based on the evaluation of creep and fatigue properties and related microstructural alterations; in particular on the coating/substrate interaction. An attempt will be made to correlate these features to the coating chemistry and process parameters.

Test pieces in the alloys IN100 and IN738LC have been obtained and were supplied with coatings by the collaborating partners in the COST-project. The majority of the creep testing will be undertaken with continuous extensometry so that the maximum information on the material can be obtained. In addition the degradation of coatings and microstructural changes occuring during isothermal and cyclic oxidation exposures without mechanical stresses will be studied.

For the thermal-fatigue tests a facility will be used in which Glenny-type disc specimens of IN738LC with or without coatings will be cycled between two fluidised beds. The test programme will include the evaluation of the effect of test temperatures, holding time and edge radius.

Test procedure and conditions

Materials

a) Substrate:
IN738LC
IN100

b) Coatings:
Aluminide-type CVD (LDC-2, H1-15, Cr-A etc.)
Overlay coating of Alloy S57 (Co Ni Cr Al Y applied by LP-plasma spraying, ion plating, ion sputtering, laser-technique

Temperature

a) Creep: 750, 850, 950°C
b) Ageing. 750, 850, 900, 950, 1100°C
c) Thermal fatigue: T_{max} in the range 750-1100°C

Duration

a) Creep: stress rupture in 500, 1000, 4000 h.
b) Ageing times: 200, 500, 1000, 3000 h.
c) Thermal fatigue: about 400 cycles maximum.

Loading

Samples will be exposed to isothermal and cyclic oxidation, creep and thermal-fatigue testing.

Analysis

Material analysis will include the following techniques: Optical microscopy, XRD, SEM, EPMA, TEM.

Sub-activity 2.A.3.4.: Grain boundary segregation in a nickel-base alloy

Aim

To investigate the grain-boundary structure and composition of a nickel-base alloy after high temperature ageing treatments and to assess the influence of applied surface coatings on the intergranular composition of the substrate alloy.

Work programme

Grain boundaries are known to play an important part in creep deformation and fracture processes. Clearly the species occupying intergranular sites in these alloys will have a critical influence on failure behaviour and this may be particularly important in coated alloys where corrosion from the surface is inhibited by the coating. This general problem will be studied by taking as example one commonly used nickel-base superalloy which is of concern to COST 50 participants

Test pieces will be strained to fracture under creep conditions in high vacuum in an Auger-coupled hot-straining stage with or without preageing. Fracture surfaces will be analysed in qualitative and quantitative terms by Auger spectroscopy to establish the type and rate of development of segregating species. Morphological and compositional analysis by SEM, microprobe will be used to assess the association of segregants with alloy fracture behaviour. Other analytical facilities used may include optical metallography and transmission electron microscopy

Test procedure and conditions

Material

a) Substrate:
IN738LC

b) Coating:
Some samples will be coated either by a CVD alumide process or by an ion sputtered MeCrAlY.

Environment

Air for ageing.
High vacuum for fracture.

Temperature

a) Preageing: 850, 900°C with or without stress.
b) Fracture: 850°C.

Duration

a) Preageing: up to 400 h.
b) Fracture: 1 - 16 h.

Activity 2.A.4.: Corrosion without load

Objectives

To develop an understanding of the factors governing corrosive attack at high temperatures in simple and complex gaseous environments of low oxygen activity in which the other reactants will be carbon and/or sulphur. Both the kinetics and the mechanisms of corrosive degradation will be studied and the influences of alloy composition, form and metallurgical condition upon corrosion resistance will be established.

General aspects of the test procedure and conditions

Thermodynamics

The interpretation and comparison of the corrosion behaviour of engineering alloys are greatly helped by having a basic undertanding of gas-phase thermodynamic equilibria and metal-environment stability diagrams. The thermochemical data derived and formulated during the 1977/79 programme will continue to provide a valuable insight into the more fundamental aspects of corrosion behaviour.

Kinetics

The kinetics of the corrosion processes will be monitored by conventional gravimetric techniques, both discontinuously, and also continuously in selected instances.

Sample preparation

In assessing the relative corrosion resistance of alloys the surface preparation of the test specimens has to be controlled and evaluated. Normally two types of standardised surface preparation will be investigated; grinding on a 180 grade SiC paper in which a reproducible depth of working is induced in the surface, and an electropolishing treatment with the object of producing work-free surfaces. The size of the specimen to be tested at the start of the work is 10x8x3 mm, although larger specimens will also be used.

Material analysis

The size, distribution and morphology of surface reaction products (carbides, oxides, sulphides) will be monitored by scanning electron microscopy whilst the type and composition of these will be established by X-ray diffraction, electron probe microanalysis and ESCA/AUGER techniques. Magnetic susceptibility measurements may be used to indicate the extent of sub-surface corrosive penetration and these results would be correlated with those of conventional cross-sectional optical microscopy, scanning electron microscopy, electron probe micro analysis and nuclear microprobe analysis. These techniques will enable the extent and mode of corrosion and metal degradation to be established.

Sub-structure of activity

The work of the activity is divided into a number of clearly defined areas of investigation or 'sub-activities' which will be studied using the above test procedure. Their titles are:

2.A.4.1. The corrosion behaviour of materials exposed to carburising environments.
2.A.4.2. Inhibitive treatments and their effect upon materials behaviour in carburising atmospheres.
2.A.4.3. The corrosion behaviour of materials in mixed carburising/oxidising environments.
2.A.4.4. The effect of sulphur upon the corrosion behaviour of materials in binary and complex component environments.
2.A.4.5. The in-plant exposure of test specimens for commercial processes of relevance.

Sub-activity 2.A.4.1.: The corrosion behaviour of materials exposed to carburising environments

Aim

To identify the chemical reactions that occur and the mechanisms responsible for the degradation of alloys in carburising atmospheres. To determine the kinetics by which carburisation proceeds and to establish the rate-controlling step. The role of different alloying additions will be identified with a view to providing guidelines for the use of alloys resistant to this type of attack.

Test procedure and conditions

Material

Commercially available cast and wrought versions of the various 20-25Cr/20-35Ni alloys referred to in the introduction to part project 2A will form the basis of the test programme. Several 'model' alloys, which are analogues of the commercial materials will also be studied.

Environment

The corrosive environments will all be based upon the H_2-CH_4 binary system varying the carbon activity (a_c) between 0.3 and 0.8 (Table 1).

Temperature

Temperatures will be within the range 800-1100° C, the exact values being selected on the basis of the initial test results.

Duration

Exposure periods will vary from 10 to 1000 hours or more.

Sub-activity 2.A.4.2.: Inhibitive treatments and their effect upon materials behaviour in carburising atmospheres

Aim

To identify and evaluate techniques by which corrosion in carburising environments can be reduced. To establish the mechanisms by which inhibition occurs and also to quantify the improvement in materials performance obtained therefrom. The inhibitive treatments will be approached from two standpoints, firstly by modifications to the gas chemistry and, secondly, by modifications to the alloy content.

Test procedure and conditions

Material

Commercially available cast and wrought alloys will be selected from those studies in sub-activity 2.A.4.1. Several 'model' alloys based upon the commercial materials will be manufactured with variations in minor elements such as Si, Al and S.

Environment

The corrosive environments will be based upon the H_2-CH_4 system but, in addition, the effect of introducing very low concentrations of sulphur in the form of H_2S (5 to 100ppm) will be studied. Pre-oxidation in low-oxygen-activity environments as a means of producing oxides, e.g. SiO_2 and Al_2O_3, which will remain stable in these H_2-CH_4 environments, will also receive attention.

Temperature

Test temperatures will be varied within the range 800-1100° C.

Duration

Exposure periods are likely to be lengthy, i.e. several thousand hours, in order to establish any long-term benefit from the inhibitive treatments.

Sub-activity 2.A.4.3.: The corrosion behaviour of materials in mixed carburising/oxidising environments

Aim

To identify the factors involved and the mechanisms responsible for materials degradation in carburising/oxidising environments. The ability of protective oxide films to form in simultaneously carburising atmospheres and hence to modify the rates of carburisation will be studied. The role of different alloying additions will be identified with a view to providing guidelines for the better use of alloys resistant to this type of attack.

Test procedure and conditions

Material

The alloys will be selected from those used in Sub-activity 2.A.4.1. The presence of minor elements with a high affinity for oxygen, will be an important aspect of these studies.

Environment

The corrosive test environments will be multi-component, i.e. $H_2/CO/CO_2/CH_4/H_2O$ with H_2 and CO being the major constituents. The carbon activity and oxygen partial pressure will be varied independently between $a_c = 0.3$ and 0.8 and $p_{O_2} = 10^{-14}$ and 10^{-21} bar. (Table 1).

Temperature

Temperatures will be concentraded within the region of 800-900° C.

Duration

Exposure periods will vary up 1000 hours or more.

Sub-activity 2.A.4.4.: The effect of sulphur upon the corrosion behaviour of materials in binary and complex component environments

Aim

To evaluate the role played by sulphur in the degradation of materials in single reactant (S) and multi-reactant (S-O-C) environments. The ability of oxide scales to prevent internal degradation by sulphur will be examined and the relative merits of Cr_2O_3 and Al_2O_3 scales in providing resistance to this type of attack analysed.

Test procedure and conditions

Materials

Commercially available cast and wrought versions of the various 20-25Cr/20-35Ni alloys and also Ni base alloys referred to in the introduction to part project 2A together with 'model' alloys, will form the basis of the test programme. The controlled addition of minor elements will be important in these investigations.

Environment

Initially small amounts of H_2S ($\leqslant 1\%$) will be used in a H_2 carrier gas or will be injected into the mixed carburising/oxidising environments used in sub-activity 2.A.4.3.. This will result in low partial pressures of sulphur, of the order, 10^{-10} bar.

Temperature

Test temperatures will be concentrated in the region of 800-900°C but lower or higher temperatures may be investigated.

Duration

Exposure periods ranging from several hours up to perhaps 1000 hours will be covered.

Sub-activity 2.A.4.5.: The in-plant exposure of test specimens for commercial processes of relevances

Aim

To relate the corrosion behaviour of test samples exposed to laboratory environments and to the on-line process environments of relevant pilot and commercial plant. In this way to ensure a strong and active liaison with the petrochemical and coal-processing industries.

Test procedure and conditions

Laboratory-prepared specimens of identical metallurgical and surface condition to those exposed within the corrosion test facilities will be exposed within pilot and commercial plants of collaborating European industries.

The gas chemistries, operating tempertures and pressures and intended exposure periods cannot yet be defined. These will be governed by the industries concerned but accurate information regarding these test variables must be available at the time of exposure. Exposure periods are likely to be of the order of thousands of hours. Close comparison of the kinetic and mechanistic data obtained from laboratory studies and field trials will be an essential part of these investigations using the full range of material analysis techniques. The effect of system pressure on kinetics will be assessed in comparison with the results obtained at relatively low pressures in the laboratory studies.

Activity 2.A.5.: Corrosion under load

Background

Under realistic conditions the structural materials are always under a load which may be low or high. Conclusions arising from corrosion tests without load therefore need to be confirmed or modified for conditions where the materials are under load. For selected materials which are under test in activities 2.A.1., 2.A.2., 2.A.3., 2.A.4. and 2.B.1. a comparative study should show the influence of a load (static or dynamic) on the kinetics and the mechanism of corrosive degradation.

Objective

To obtain a qualitative understanding of corrosion mechanisms and quantification of corrosion kinetics under the action of static and dynamic stresses both where there is no effective surface scale protection and even where a scale remains protective.

Test procedures

Loading

The mechanical tests under corrosive environments will normally be executed in the frame of activities 2.A.1., 2.A.2., 2.A.3. and 2.B.1. In addition, a relatively small number of tests may be necessary with either special materials (eg. models or samples given prior deformation), special test conditions, or special preparation (eg. electropolished).

Reference samples prepared by the same machining procedure as those for mechanical testing will be exposed simultaneously for comparison with the samples used in activity 2.A.4.

Analysis

The selected samples from the various activities will be subjected to study using the techniques found to be necessary for an understanding of the processes involved. The techniques available include:
Optical metallography
X-ray diffraction
Scanning electron microscopy
Electron probe microanalysis
Transmission electron microscopy
ESCA/AUGER.

In addition chemical analysis and/or nuclear microprobe analysis will be obtained where necessary.

Sub-structure of activity

The work of the activity is divided into a number of clearly defined areas of investigation or 'sub-activities' which will be studied using the above procedure. Their titles are:

2.A.5.1. The influence of static stress on the carburisation of high temperature alloys.
2.A.5.2. The influence of static stress on the carburising/oxidising behaviour of high temperature alloys.
2.A.5.3. Influence of a dynamic load on corrosion behaviour.

Sub-activity 2.A.5.1.: The influence of static stress on the carburisation of high temperature alloys

Aim

To study the influence of creep deformation over a range of stresses on the mechanism and kinetics of corrosion of different high-temperature alloys in carburising atmospheres, to explore the structural-corrosive interactions and make comparison with the behaviour in the absence of a load.

Test conditions

Materials

First investigations will be carried out on: HK40, Alloy 800H and Alloy 802.

This group will be expanded by other materials referred to in the introduction to part project 2A, depending on the progress of the activity.

Environment

A H_2/CH_4 environment with carbon activity, $a_c = 0.8$ is envisaged.

Temperature

Test temperature will be 1000°C for most experiments.

Duration

Tests will be performed in the range of 20-200 h exposure time.

Sub-activity 2.A.5.2.: The influence of static stress on the carburising/oxidising behaviour of high temperature alloys

Aim

To study the influence of creep deformation over a range of stresses on the mechanism and kinetics of corrosion of different high-temperature alloys in carburising/oxidising atmospheres, to explore the structural-corrosive interactions and make comparison with the behaviour in the absence of a load.

Test conditions

Materials

To be selected from those under examination in sub-activity 2.A.5.1.

Environment

The oxidising-carburising atmosphere will be selected on the basis of experience in activities 2.A.1. and 2.A.4.

Temperature

Test temperature 900/950°C is foreseen.

Duration

Tests will be performed in the range of 50-1000 h exposure time.

Sub-activity 2.A.5.3.: Influence of a dynamic load on corrosion behaviour

Aim

To study the influence of a cyclic load on the mechanism and the kinetics of corrosion in a carburising/oxidising environment at high temperature. To compare the results with those obtained under static load and in the absence of load.

Test conditions

Materials

Essentially the same materials as those tested in activity 2.A.2. will be investigated, i.e. initially Alloy 800H.

Environment

Carbon activity and oxygen partial pressure values will be determined for the carburising/oxidising environment described in Project 2, Table 1 on the basis of data and mechanistic understanding achieved in activities 2.A.1., 2.A.2. and 2.A.4.

Temperature

Test temperature will be in the range 800 - 1000°C.

Loading

Initially the loading conditions will be chosen to permit investigation of the corrosion behaviour in the presence of an effective protective surface layer. The variables that matter are strain range and test frequency. Test limits and precise test values will be selected when more information from activities 2.A.1., 2.A.2. and 2.A.4. is available.

The sepecific effect of cyclic stress on corrosion mechanism and kinetics in the absence of a protective layer will be studied by varying the same parameters.

Part Project 2.B.: Engineering Studies

Objective

The objective of the Engineering Studies part project is to provide the required link between research on materials and engineering applications by a limited number of experiments on the long-time elevated temperature properties of welded tubular components. It also includes the specification and feasibility study of an advanced high temperature test facility for tubular components.

Activity 2.B.1.: Creep of tubular components in corrosive environments

Background

At ambient temperatures, sufficient knowledge and expertise is available on stress analysis and materials deformation behaviour to allow designs of tubular components to proceed from a combination of available materials data with standard analysis methods and design codes. However, at high temperatures, the time-dependent damage accumulation mechanisms, through which materials degrade, do not lend themselves easily to analysis or design codes. As a consequence the evolution of high-temperature design data from materials data and mechanisms can be assisted by undertaking tubular component testing

Critical to the development of stress analysis competence is the appreciation of the different behaviour of materials under non-uniaxial stress. In process plant materials, complex stresses are invariably present and we must be able to determine behaviour under multiaxial stresses from our knowledge of uniaxial behaviour

Another feature of high-temperature plant which does not easily lend itself to simple tests and subsequent extrapolation to service behaviour concerns the behaviour of weldments. It is well known that uniaxially tested welded specimens do not necessarily provide reliable data. The geometry of a tubular test specimen is ideal for weldment testing as the weldment can easily be subjected to a multiaxial stress field and extrapolation to design, service and performance behaviour is facilitated by the fact that circumferentially welded tubes and pipes are common features in high-temperature industrial plant

The additional influence of environment has to be superimposed. The tubular component is also capable of being operated in conditions simultative of the plant environment with different atmospheres inside and outside the tube.

Consequently, tubular test samples, which represent the geometry of many structural components in high-temperature plants, are chosen to allow the investigation of the influence of multiaxial stress and environment on high-temperature behaviour of materials and welded structures

Objective

The aim of this activity is to achieve a mechanistic understanding of the behaviour of selected alloys and weldments under multiaxial creep conditions in corrosive environments for comparison with uniaxial behaviour.

Sub-structure of activity

To achieve this objective the activity is divided into three sub-activities as follows:

2.B.1.1.	The influence of multiaxial stress on the creep behaviour of plain tubular alloys at high temperatures.
2.B.1.2.	The behaviour of weldments under multiaxial stresses at high temperatures
2.B.1.3.	The effect of corrosive environment on the behaviour of alloys and weldments subjected to multiaxial stresses

Sub-activity 2.B.1.1.: The influence of multiaxial stress on the creep behaviour of plain tubular alloys at high temperatures.

Aim

To study the behaviour of metallic tubes under multiaxial stress in order to understand the mechanisms of creep deformation, to relate these to uniaxial behaviour and to test their use for component life predictions.

Test procedure and conditions

Materials

The project core material, Alloy 800H will be used in the initial stages of the programme. It is readily available in dimensions suitable for tube testing (eg. 1"). At a later stage when facilities are available for testing larger tubes, materials of the type HP 40 + Nb will also be studied.

Environment

The tube internal environment will be either high pressure argon or air and the external environment ambient pressure argon.

Temperature

Temperatures in the range 800-1100° C consistent with those chosen for other activities.

Duration

Initial experiments are likely to be less than 500 h but later tests may be aimed for several thousand hours.

Loading

Conventional uniaxial creep testing of tubes will be followed by testing under internal gaseous pressure, variation in biaxial stress ratio being later achieved by combining the pressure and axial load.

Test facilities

Uniaxial creep testing will be mostly achieved using Mand 20kN conventional creep machines. Pressure testing will be initially carried out under external contract and the combined-stress testing will be carried out using an MTS 50kN and a Mand 100kN creep machine modified for the addition of internal pressure and housed in specially constructed concrete safety cells.

Analysis

Metallurgical aspects of deformation will be studied post test using conventional metallography, scanning and electron microscopy. Analysis of stress-rupture and deformation strain data will not only relate to uniaxial data but will also require to be extended to contribute towards the development of constitutive equations governing material degradation of direct relevance to the designer or plant operator.

Sub-activity 2.B.1.2.: The behaviour of weldments under multiaxial stresses at high temperatures.

Aim

To examine the effect of superimposed multiaxial stress on the behaviour of welded joints in order to understand the deformation response and to provide data on which predictive models may be based and which can contribute to a better description of weldment reliability and lifetime.

Test procedure and conditions

Materials

Fully circumferential, part circumferential, and part longitudinal welds will be examined on the 1" Alloy 800H tubing. These welds will be made by conventional techniques, in particular those used for joining and repair of components of this type in commercial plant such as tungsten inert gas.

Test conditions

The test environments, temperatures, duration, loading and facilities will be identical to those described in 2,B.1.1.

Analysis

The metallurgical analysis will concentrate on only the weldment and the heat-affected zone and the microstructure resulting from multiaxial creep will be compared to the as-welded microstructure to elucidate information on microstructural evolution. The effect of varying biaxial stress rations on the resistance of the weldment to failure and deformation will be assisted in order to reach at least an empirical description of the multiaxial creep response which can be of considerable relevance for industrial plant components.

Sub-activity 2.B.1.3.: The effect of corrosive environments on the behaviour of alloys and weldments subjected to multiaxial stresses at high temperatures.

Aim

To determine the influence of a simulated process environment on the mechanisms of multiaxial creep deformation of tubular materials, with or without welds, and thereby also to quantify the corrosion influence so that it may be incorporable into component life-time prediction and design codes.

Test procedure and conditions

Materials

These will be identical to those of sections 2.B.1.1. and 2.B.1.2.

Environment

hough some tests will be carried out initially on tubes under uniaxial creep where the external environment will be approximately ambient-pressure active gas, all pressure tests will utilise high-pressure active gas inside the tubes acting also as the stress-raising medium, with inert gas at ambient pressure surrounding the tube acting as a safety gas. The initial active gas environment will be carburising using a mixture of methane and hydrogen in such a proportion to give a carbon activity of 0,8 at the pressure and temperature of the test. Later, lower carbon activities consistent with other investigations will also be used and by the end of the programme more complex environments should be utilised containing both carburising and oxidising components.

Testing temperatures, duration and loading will be in the same ranges as given in the previous sections.

Test facilities

All tests using active gas of a potentially explosive or toxic nature will be conducted in specially constructed rigs. Tests using slightly above ambient pressure active gas will be carried out using work tubes around the creep specimens and the safety control circuit will be a local control cabinet (L.C.C.) of standard design used for all other projects. High pressure tests with active gas will take place inside a metallic safety-box filled with inert gas, the total experiment being housed in concrete safety cells in a controlled area. For internal pressure tests and axial loading the safety boxes will be mounted on the 50kN MTS and 100kN Mand creep machines.

Analysis

The analysis requirements will be exactly the same as in sections 2.B.1.1. and 2.B.1.2. but which the additional requirement for a more chemical approach to micro-structural evaluation and a further compounding of the already existing complex analysis required for the prediction of failure and deformation behaviour.

Activity 2.B.2.: Study of a high temperature test facility for tubular components.

Background

The research activity described here is restricted solely to a specification and feasibility study.
Following a survey of all high-temperature materials and component testing facilities in Europe carried out by the J.R.C. Petten staff and a survey of the needs of European industry which was carried out under a J.R.C. contract with the Battelle Institute, Frankfurt, it was concluded that a tubular component testing facility would cover the most important area in terms of future needs. The technical specification task was awarded as a study contract to the National Engineering Laboratory (N.E.L.) East Kilbride, U.K. and, at the beginning of the 80-83 multiannual programme, was nearing completion.

Objective

To prepare the specification of a high temperature test facility for tubular components and the corresponding test programme.

Activities

The conclusion of the study contract awarded to N.E.L. which was initiated during the previous multi-annual programme forms the first main action.

It is planned to discuss the specification of the conceptual test facility and programme with European experts in order to evaluate the interest and importance of such a facility for European industry, the suitability of the technical specification proposed and the feasibility of the facility.

Project 3: Data Bank

Background

Recent progress on the establishment of information networks such as originally launched by the USA (Lockheed, SDC, BRS) is now allowing Community-wide user access to any type of data base through, e.g., EURONET. The demand of the European materials users for efficient and rapid access to high temperature materials data therefore can in future be satisfied, if a data base on high temperature materials is created and linked to the system.

The High Temperature Materials Programme has investigated the European interest for such a data information system during the Multiannual Programme 1977-1979 in order to identify alternatives for the dissemination of information in the form of factual data. From the results of a contractor study it could be extrapolated that about 400 European institutions are potential users of the system and that the main interest of the prospected 800-1000 personal users relates to application oriented, evaluated property data as opposed to exclusively bibliographic information.

The investigation showed that this interest area is at present not satisfied by any generally available information system existing in Europe. It therefore appeared appropriate to use the research capacity of the Petten High Temperature Materials Programme as a basis for the establishment of a property data bank meeting the European needs.

The project defined under the 1980-1983 programme foresees the set-up of a data bank on evaluated materials data with a restricted scope of alloys, properties, temperatures and environments.

Objectives

The project long-term objective is to set up all essential functions for a pilot system of a data bank containing evaluated data on mechanical properties and corrosion resistance of HT materials used in energy conversion plant. The goal of the operational data bank is the provision of:

- Service functions to users
- An instrument for the identification of areas where research in data generation requires stimulation.

Materials of primary concern are those which are used in the R & D project of the HTM programme such as austenitic iron-base alloys like Alloy 800.

In detail the practical objectives to be achieved during the 1980-1983 period can be grouped in two phases:

a) Preliminary demonstration phase

- Identification of data sources
- Initiation of data collection and formatting action
- Publication of a 'Data Bank Demonstration Handbook'
- Testing user' reaction
- Design of the implementation phase (co-operation with JRC Ispra computer services)

b) Implementation phase

- Utilisation of JRC Ispra, depart. A, facilities and assistance (software (ADABAS) and hardware) for data input, storage and retrieval function
- System testing
- Demonstration of system operation
- Operation
- Establishment of data evaluation functions.

Sub-structure of the project

In order to achieve the objectives of the preliminary demonstration phase and the implementation phase, a number of parallel and interlinking activities are required. They are executed in collaboration with JRC Ispra, utilizing the available hardware and software facilities for operation and development. The data acquisition and data input preparation activities require contribution by external contractors so that the development of procedural routes and verification routines is a vital need.

Therefore defined objectives of the two phases can only be realized with the available resources, if the project structure permits the successive concentration of the effort on the completion of functionally coherent work units. Accordingly a project strategy is chosen which successively achieves a number of data bank 'sub-totals' which are called 'modules'. Each module is not only a part of the data bank but also a fully operable unit in itself. Although the four defined modules have different goals, their tasks to be accomplished are to a certain extent overlapping. Table 2 gives a survey of the four modules and their relation in function and time.

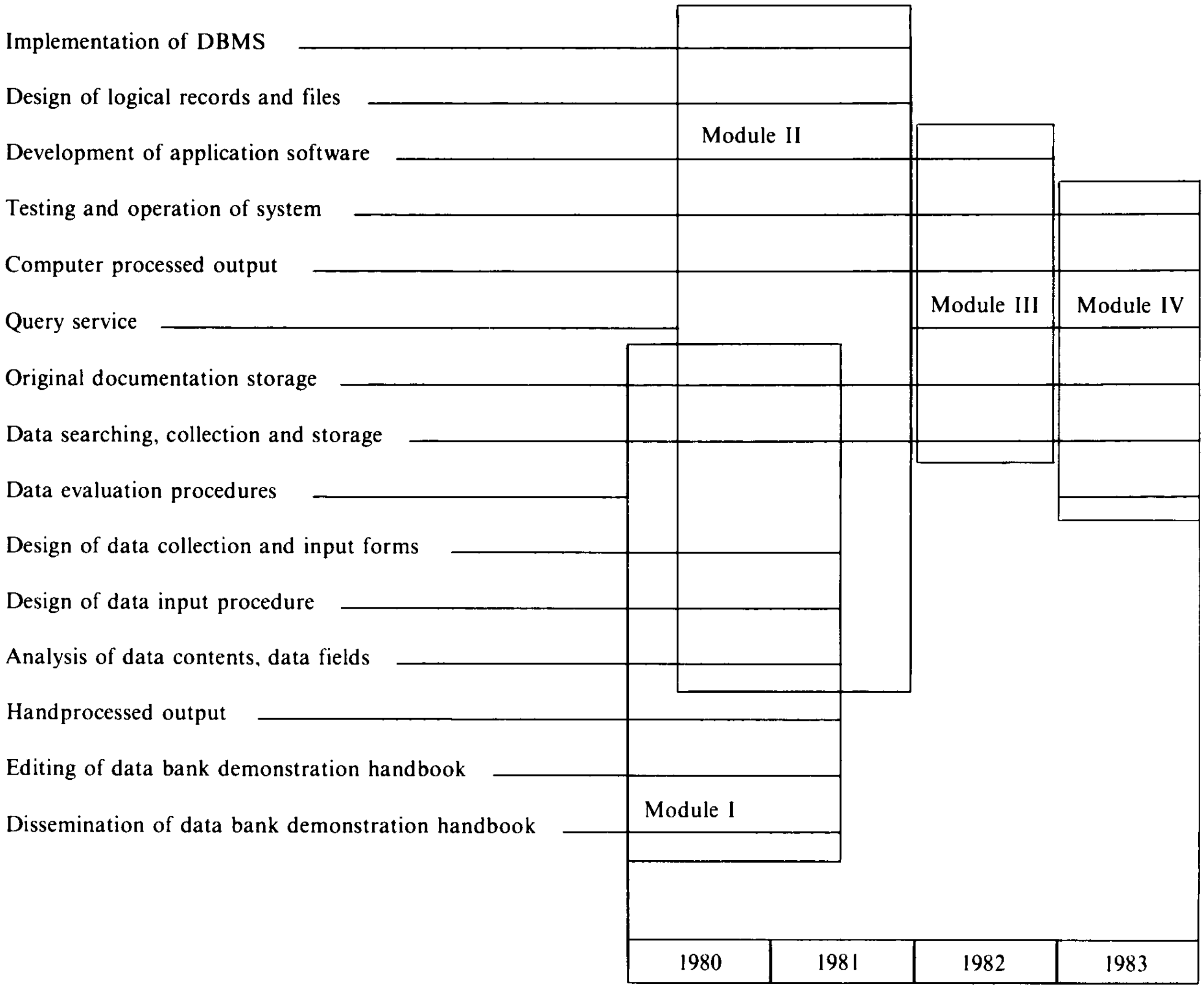


Table 2: Data Bank Project, Function and Time Relation of Modules

Module I: Functions required to assemble a set of data sheets compiled as a 'Data Bank Demonstration Handbook'

Management Objective

To set up and test the essential functions of a data bank information system (DIS), with special emphasis on the input procedure.

Definition of Goals

Publication of a set of data sheets compiled as a 'Data Bank Demonstration Handbook'. Possible form: EUR-report. Intended publication date: Mid 81.

Scope of Data Content

Main emphasis on the following range (handbook):

- Material: Alloy 800 varieties
- Temperature: 600-1000°C
- Environment: C-O-H
- Tests: Tensile, creep, fatigue.

The scope of data collection will be wider. Restriction to data not older than 10 years.

Data Sources and Methods of Collection

Data sources:

- Petten HTM programme experimental data
- Proceedings 'Petten International Conference Alloy 800'
- Literature surveyed and available at Petten
- Data offered by initially (easily) accessible partners.

Methods of collection:

- Initially (design phase) by own staff
- Later under contract.

Selection and Transfer of Data to Data Input Form

Data are selected and transfered into standard data input forms with segments for:

- Data and test conditions
- Materials characterization
- Information on data source and other.

Data input forms are such that computer input is enabled. Verification procedures are applied e.g. common sense check.

Data Storage and Retrieval

. Storage on data input forms only
. Manual retrieval of the data from file of data input forms;
. Storage of prototype data dictionary (initially as list of data content).

Output

Data assembled to tables, plots and lists for the selected parameters. Data sheets, lists and tables, together with data dictionary, edited as data bank demonstration handbook.

Data Dissemination

The data bank demonstration handbook is the sole route of data dissemination.
Distribution:
. Standard distribution
. All data sources
. All authors made reference to
. Relevant advisors and advisory bodies
. Participants to relevant meetings, workshops inquiries etc.

Management, Marketing and User Support

. An (external) management or steering committee should develop during this module
. Goals of marketing and promotion: future modules (III, IV) by means of data bank demonstration handbook.
. Feedback from users by means of interviews e.g. on data sheets resulting in user's support to develop future modules.

Remarks

The data bank demonstration handbook is not developed as a normal handbook but as a simulation of the prospected data bank output.
Its construction therefore is based upon all DB procedures and formalisms already available either in hand or computer processing depending on progress within module II.

Module II: Computer functions required to generate a handbook type output from a developed data input

Management Objective

To design and implement the essential functions of the computerized data bank.

Definition of Goals

Design of the DB and development of all computer functions required to operate a computer processed publication as handbook type output.

Scope of Data Content

See module I; but quantitative extension of data scope.

Data Sources and Methods of Collection

Same as module I, extension:
. Negotiations for data acquisition from sources of unpublished data.

Selection and Transfer of Data to Data Input Form

Data input sheets of module I, same procedure as module I. Cross linking with module I to assure full comptability with computer requirements. Data are converted into the form necessary for storage into the computerized data bank. Computerized input functions to check faulty preparations.

Data Storage and Retrieval

Data Bank with data bank management system. Storage of the data, data dictionary, statistical items and information required to run DB administration and promotion.

Output

Module I type output supplemented by inverted lists and outputs for data bank administration. Data delivery by printer, plotter and terminal display envisaged.

Data Dissemination

- Concept of data dissemination like during module I
- Commercial channels for sale
- Further channels to be decided.

Management, Marketing and User Support

. Functions of module I run simultaneously
. DB project execution by Data Bank Administration and Data Bank Design
. Inquiry followed by improvement of system.

Remarks

Module II concentrates on the development of the computerized data processing with limited effort related to perfection of retrieval and output functions.

Module III: Implementation of retrieval functions to handle complex queries

Management Objective

To set up all functions for the handling of queries. To continue and extend regular publication of data sheet compilations.

Definition of Goals

Implementation of retrieval functions to handle complex specific queries. Set up of a query service.

Definition of Goals

Implementation of retrieval functions to handle complex specific queries Set up of a query service.

Scope of Data Content

Qantitative and qualitative extension with respect to modules I en II.
Options are:
. Various other HT alloys
. Ceramic materials
. High temperature test methods and standards

Data Sources and Methods of Collection

As module II but characterized by a gradual extension of data collected from non published sources resulting in continuous extension of data network.

Selection and Transfer of Data to Data Input Form

See module II.

Data Storage and Retrieval

Data bank with special retrieval programmes for selective output in order to tackle complex queries.

Output

Selective outputs of variable formats. Analyst/machine dialogue to handle queries. Data delivery embraces multi user mode by terminal display. No direct access for users planned at this stage.

Data Dissemination

. Handling of incoming queries viá letter, telex or telephone
. Regular publications of single data sheets and handbook compilations.

Management, Marketing and User Support

. Set up of committees to work out recommendations on principles and methods for data evaluation
. Marketing and promotion by means of cross referencing with other data banks and information systems
. Distribution of a user's manual and conditions of DB access.

Module IV: Development of data evaluation procedures

Management Objective

To introduce and operate a data evaluation procedure and provide information on evaluated data.

Definition of Goals

Publication of data sheets on evaluated data.

Scope of Data Content

To be defined e.g :
. Evaluated data
. Data on corrosion tests.

Data Sources and Methods of Collection

See Module III.

Selection and Transfer of Data to Data Input Form

See module II.

Data Storage and Retrieval

Like Module III but including retrieval programmes to support data evaluation procedures.
. Separate files for evaluated data.

Output

See module III. First publication of data sheets on evaluated data possible.

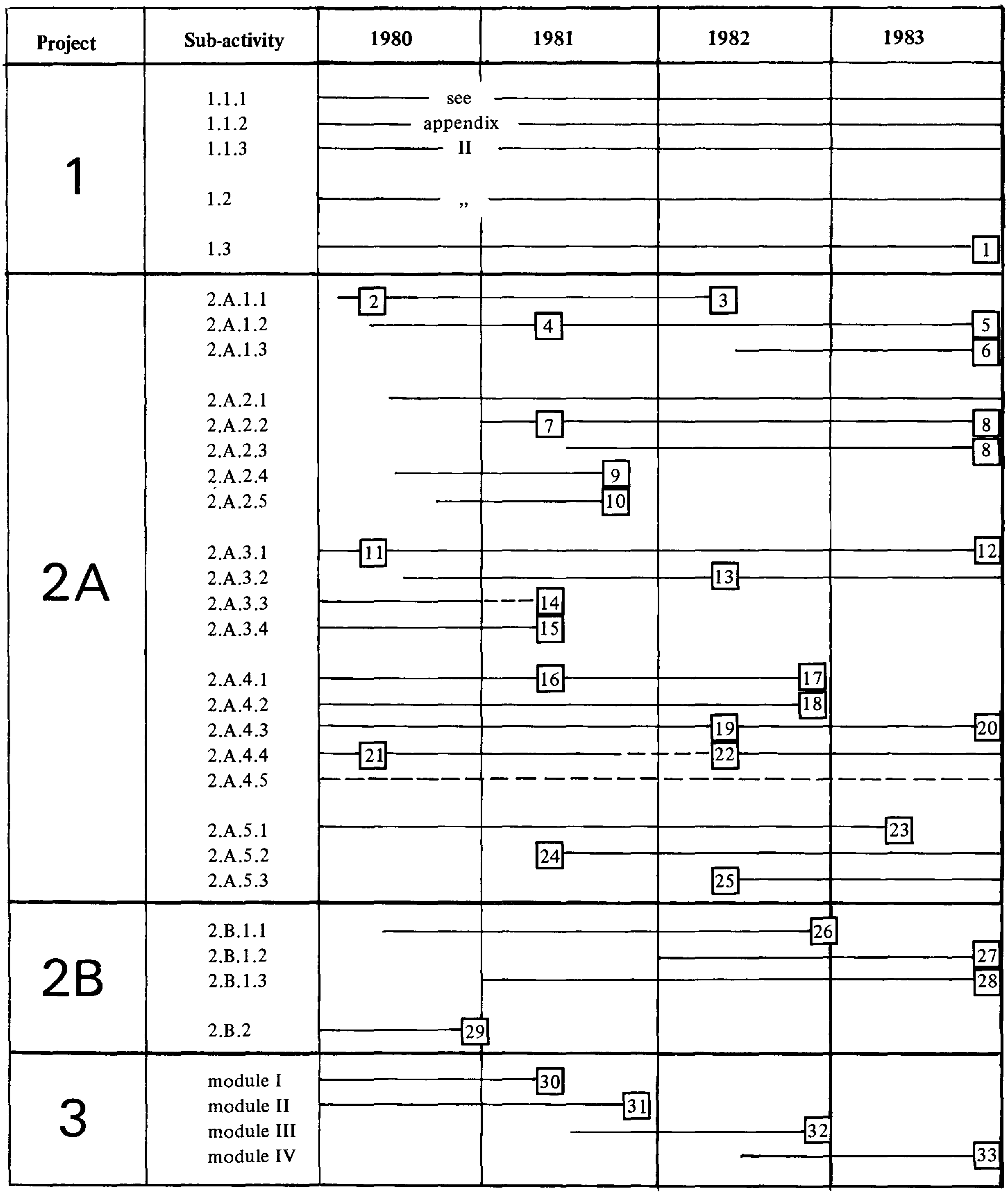
Data Dissemination

See module III.

Management, Marketing and User Support

Working groups with external experts for data evaluation editing their reports as special publications. Exchange of information with other systems.
User courses on data bank use and inquiry service.

Appendix I **General Time Schedules**



——— action
— — — planned, conditional
☐ milestones, explanation in following table

Explanation of Milestones

1. Publication of directory on ongoing HTM research.
2. Creep experiments in carburising atmosphere completed for 2 alloys (HK 30, Alloy 800H).
3. The influence of structure on creep behaviour under carburising conditions evaluated for selected cast and wrought austenitic alloys.
4. Successful test result obtained in carburising/ oxidising atmosphere.
5. The consequences of attack by complex carburising/ oxidising atmospheres on the creep deformation and stress rupture behaviour of selected cast and wrought austenitic alloys evaluated.
6. Test procedure established for tests in sulphur bearing complex gas.
7. LCF testing commences in carburising/oxidising atmosphere.
8. The cycle-and time dependent fatigue behaviour of selected alloys characterised in carburising/ oxidising atmospheres.
9. The HTLCF behaviour of two forged nickel base superalloys characterised and mechanism of failure assessed.
10. Crack initiation in time dependent LCF characterised and quantified in a forged nickel base superalloy.
11. Air oxidation tests on the first series of IN519 +. Si/Y completed.
12. The parameters affecting the adhesive properties of natural scales growing on typical austenitic alloys assessed for carburising/oxidising environments.
13. Literature study and preparatory stages complete; Experimental testing started.
14. The mechanism and kinetics of degradation of overlaytype MeCrAlY-coatings evaluated in comparison to pack-aluminide-type coatings for coating/substrate structural changes and effects on creep and thermal fatigue properties.
15. The grain boundary structure and composition investigated for a cast nickel base superalloy.
16. Effect of alloy composition in carburising atmospheres explored for a range of austenitic alloys.
17. The mechanism and kinetics of carburisation corrosion assessed for selected austenitic alloys.
18. The effect of inhibitive treatment on corrosion in carburising environments evaluated.
19. Effect of alloy composition in carburising/ oxidising atmospheres explored for a range of austenitic alloys.
20. The mechanisms and factors responsible for materials degradation in complex carburising/oxidising environments assessed.
21. Screening tests on a range of alloys completed for low sulphur partial pressures.
22. The behaviour of selected iron and nickel base alloys in pure sulphidation corrosion conditions explored.
23. The influence of creep deformation on the mechanism and kinetics of carburisation corrosion explored.
24. Studies commence on the influence of static stress on carburisation/oxidation corrosion.
25. Studies commence in the influence of dynamic loads on carburising oxidising corrosion.
26. Test results available on combined pressure, end- and thermal loads with inert environments for plain tubes; correlation with uniaxial creep data and comparison with theoretical analysis for life prediction.
27. Test results available on welded tubes, inert environment.
28. Test results available for combined loads with process gas environments and correlation with uniaxial and corrosion data.
29. Specification of a high temperature test facility for tubular components available.
30. Data Bank Demonstration Handbook available.
31. Generation of data plots and data tables by computer processing developed.
32. Retrieval functions for handling of queries developed.
33. Data bank service for handling of queries available; Development of data evaluation procedures.

Appendix II Information Centre Actions 1980/81

Actions		Timing	1980 s	1980 o	1980 n	1980 d	1981 j	1981 f	1981 m	1981 a	1981 m	1981 j	1981 j	1981 a	1981 s	1981 o	1981 n	1981 d
Information Exchange and Transfer	Courses/Seminars	Analysis of HTM (C)	—	—	—													
		Statistical Planning and Evaluation of Experiments (C)			—													
		Topography and Chemistry of Surfaces (S)									—	—						
		Protection of Surfaces against Gaseous Corrosion (S)															—	—
		Corrosion in Aggressive Gases (S)														—	—	
	Conferences/Symposia/Colloquia	European Symposium on Corrosion/Mechanical Stress (Symp.)																
		HTM for Thermochemical products of H_2 - Ispra/Petten (Coll.)	—	—														
		COST 50: Corrosion and Coatings Group (Coll.)		—														
		Advanced Ceramics for Mechanical Applications (Symp.)								—	—							
		Feasibility of a European Component Test Facility (Coll.)							—	—								
		Welds in Aggressive Environments (Symp.)								—	—	—						
Information Collection	Inquiries	Mechanical Properties in Aggressive Environments			—	—	—	—	—	—	—							
		Advanced Ceramics for Mechanical Applications				—	—	—	—	—	—	—	—					
		Production of HT Alloys Parts							—	—	—	—	—	—	—			
	Surveys	HTM for Advanced Solar Power Plants		—	—	—	—	—	—	—								
		HTM for Thermal Conversion of Urban Waste, Biomasses, etc.			—	—	—	—	—	—	—	—						
		HTM in Coal Conversion	—	—	—													
		Materials for Diesel Engines	—	—														
		Composites for Advanced HT Applications							—	—	—	—	—	—	—			
		Process and other Industrial Gas Turbines					—	—	—	—	—	—						
	Studies	System Analysis	—	—														
		Potential of Coatings for Coal Conversion and Utilization		—	—	—	—	—	—	—	—	—	—					
		HTM in Fusion Technology	—	—	—	—	—	—	—	—	—							
		Materials for HT Energy Conversion Processes			—	—	—	—	—	—	—	—						

Appendix III

Responsibilities

Programme Manager: M. Van de Voorde

Assistant Programme Manager: H. Kröckel

		Project Leader	Activity Leader
Project 1. Information Centre		M. Merz	
Activity 1.1.	Information exchange and transfer		M. Merz
Activity 1.2.	Information collection		M. Merz, B. Bathe
Activity 1.3.	Information storage		B. Bathe
Project 2. Materials and Engineering Studies			
Part Project 2.A. Materials Studies		J.B. Marriott, G. Kemeny	
Activity 2.A.1.	Mechanical properties under static loads in corrosive environments		V. Guttmann
Activity 2.A.2.	Mechanical properties under dynamic loads in corrosive environments		J. Bressers
	Activity 2.A.3.		
Activity 2.A.3.	Surface protection; scales and coatings		E. Lang
Activity 2.A.4.	Corrosion without load		J. Norton
Activity 2.A.5.	Corrosion under load		G. Kemeny
Part Project 2.B. Engineering Studies		H. Kröckel	
Activity 2.B.1.	Creep of tubular components in corrosive environments		R. Hurst
Activity 2.B.2.	Study of a high temperature test facility for tubular components		R. Hurst
Project 3. Data Bank		H. Kröckel	R. Krefeld