EUROPEAN COMMISSION
5th EURATOM FRAMEWORK PROGRAMME 1998-2002
KEY ACTION : NUCLEAR FISSION

HTR-M

European Project for the development of HTR Technology - Materials for the HTR

CONTRACT N°
FIKI-CT-2000-00032
FIKI CT 2001 00135

Research and Development Programme on HTR Materials

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Dissemination level : PU
Document Number: HTR-M-03/04-D-0.0.64

Proceedings of ICAPP '03
Córdoba, Spain, May 4-7, 2003
Paper 3174

RESEARCH & DEVELOPMENT PROGRAMME ON HTR MATERIALS

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Abstract – *Issues concerned with the material selection and behaviour of the main component reactor materials for the Modular High Temperature Reactor are investigated within a four-year programme of studies and tests. The work considers materials for the reactor vessel, certain high temperature regions including the control rod and turbine and the graphite core and is being performed within a European development initiative supported by the European Union Fifth Framework programme and monitored within the HTR network HTR-TN. This paper reviews the main elements of the programme and presents an overview of some of the key results and conclusions to-date. Future actions with regard to experiments and assessment are also discussed.*

I. INTRODUCTION

HTR projects have been launched within the 5th Framework Programme[1] to consolidate and advance HTR technology in Europe. This paper considers the progress and mid term results from the HTR-M & M1 Projects that look at the materials requirements for key reactor components. The projects involve partnerships between principal industrial and research organisations from different countries of the European Union and include specific experiments on selected materials aimed at providing a materials platform on which to develop the future HTR technological basis within Europe.

II. HTR M & M1 PROJECTS

The main elements of the HTR Materials projects have been reviewed[2] and are summarized in figure 1. The programme has the following objectives.

- Investigation of HTR vessel materials and data in the areas of design analysis, structural integrity analysis and materials properties under irradiated and non-irradiated conditions including testing on welds.
- Materials for the high temperature regions of the HTR under simulated environments for both reactor internals and for the turbine (disc & blades).
- Programme of work on Graphite materials data identifying new graphites suitable for HTR and performing important testing.
- Graphite oxidation and consequences of severe air ingress with core burning including the development of HTR models and data, protective coatings and innovative C-based materials.

The projects therefore consider two main areas of investigation, the review of past experience and assemble available properties, and to perform tests on key materials where necessary information is lacking or scarce.

II.A Technological survey & database for structural integrity

Vessel materials can operate at temperatures as high as 450^{o}C and information on manufacturing, environment (neutron-irradiation fluence, operational temperature, and helium environment) and welding are needed for assurance of integrity and design development. Turbine materials operate at temperatures of 850-900°C and metals and ceramic materials are being considered for the blades and disc and the control rod. Graphite acts as a moderator and structural component and has important safety implications because of structural and property changes that occur when it is irradiated. Information on all these is crucial and design / material property data under HTR relevant conditions are reviewed and compiled for candidate materials of different HTR concepts, in relation to both normal and accident conditions.

II.B Testing under irradiated and non-irradiated conditions

For the vessel tests on Mod 9Cr steel taken from typical welded vessel joints are to be investigated under irradiated and non-irradiated conditions. The test pieces are to be irradiated in the Petten High Flux Reactor (HFR) before mechanical testing. For the high temperature components short term and medium term testing under simulated HTR environments are planned covering fracture, creep and creep / fatigue conditions in air and simulated helium environments. For graphite the tests determine properties and effects of oxidation. Graphites used in previous core designs are no longer manufactured commercially, and currently available grades are limited to a few specific sources of raw materials. Available graphite grades are therefore to be irradiated in the HFR and tested to determine variations in physical and mechanical properties up to specified dose levels. Test work on graphite oxidation on structural and fuel graphite and promising carbon fibre composites from the Fusion programme are also performed.

- **HTR-M Project**
- **Vessel Materials**
  - Properties / Fabrication
  - Irradiation tests
- **High Temperature materials**
  - Internal structures
  - Turbine materials (Short term tests)
- **Graphite**
  - Properties
  - Oxidation tests

- **HTR-M1 Project**
- **High Temperature materials**
  - Turbine blade materials (Intermediate Creep tests)
- **Graphite**
  - Selection & Irradiation Testing
  - coatings

Figure 1 HTR-M & M1 Projects

III. REVIEW AND DATABASE

This activity is performed in all three areas with the objective of assembling a single source of past property information on HTR relevant materials.

III.A Vessel Steels

Most designs of future plant make use of the either an LWR type steel or modified 9Cr 1Mo. These materials have similar strength levels at temperatures up to 370°C[3].

The 'cold' vessel design concept adopted for the PBMR reactor pressure vessel leads to the choice of Mn-Ni-Mo or C-Mn steel with grades similar to SA 508 Class 2 as prime candidates. Advantage can be taken of PWR vessel technology and design rules.

The 'warm' vessel concept adopted for the GT-MHR reactor pressure vessel leads to the choice of Cr-Mo steels with modified 9Cr 1Mo steel grades similar to ASME Grade 91 as prime candidates.

The HTR-M project aims to support both concepts by a combination of experimental work to supply data on the irradiation and thermal ageing resistance of modified 9Cr 1Mo steel and the compilation of existing data covering all candidate materials.

Modified 9Cr 1Mo steel allows for higher temperatures with only a gradual reduction in design strength at temperatures up to 450°C and above 450°C allowable stresses for all materials fall off rapidly. The main structural integrity concerns are at the vessel welds, at thicker sections, hot spots, the belt line and regions important to functionality. Fracture, fatigue and creep-fatigue (depending on temperature) are the main damage mechanisms and potential environmental effects include the effects of irradiation, thermal ageing and corrosion effects arising from interactions between the pressure vessel steel and impurities in the He coolant.

The effects of irradiation on the toughness of LWR steels have been studied extensively, the major effect being an increase in the ductile brittle transition temperature (DBTT) due to the combined effects of matrix hardening and grain boundary weakening associated mainly with the residual elements P and Cu. For warm vessel options, no data were available for modified 9Cr 1Mo steel irradiated under conditions expected for the HTR. However information at much higher doses (≥1dpa) concludes that there is a strong effect of irradiation temperature in the range 250-450°C. Effects may be small on ΔDBTT at 400-440°C but measurable at 350°C. There was no information on the behaviour of modified 9Cr 1Mo steel welds.

The base materials and weld metals of both cold and warm HTR vessel steels normally have very fine grain sizes. The most likely embrittlement location is therefore the coarse-grained heat affected zone. This is a narrow region adjacent to the fusion boundary where temperatures of 1100-1300°C are reached during welding. Cold vessels operate at temperatures below the embrittlement range so thermal ageing embrittlement (TAE) can occur only during transients involving exposure to higher temperatures for tens or hundreds of hours. Warm vessels operate in the TAE temperature range throughout their lifetime and therefore are of greater concern with regard to vessel integrity.

Design codes usually instruct the designer to take into consideration the effects of corrosion without specifying in detail how this should be done. In practice two measures are usually adopted.

- An allowance for metal losses over its lifetime is added to the minimum design thickness of a pressure vessel.
- Fatigue and creep-fatigue damage calculations made with design data include allowances for data scatter, 'mild' (oxidising) environments and the characteristics of typical pressure vessel surfaces. Additional margins are necessary only where the environment has different and / or more severe effects on fatigue and creep by carburising the exposed surfaces or by producing intergranular oxidation to a significant depth.

For the HTR vessel metal losses will either be by controlled carburisation in cold wall vessels and decarburisation combined with internal oxidation in warm wall vessels. The kinetics of these processes will determine any effective metal loss. However given a beltline wall thickness in the range 140-200 mm metal loss is only likely to become significant if breakaway corrosion or metal dusting can occur which is considered unlikely in HTR Helium.

The vessel materials database includes C-Mn steels (as used in the UK Magnox and AGR types), SA 508 Grade 3 Class 1 (LWR) or its European equivalent, 2¼ Cr 1Mo steel as used on HTTR and modified 9Cr 1Mo. Aspects such as composition, products & parts, test and design data and environment are covered. The materials property database includes code data where available, and raw data for analysis. Modified 9Cr 1Mo steel showed the most uncertainty on data and fabrication experience with respect to HTR characteristics and potentially has the wider applicability hence its selection for the for the irradiation test programme. Significant amounts of data exist for all these steels and the main focus is on comparisons and assembling relevant design data. Management of the database is to be done through a web based system to allow remote partner access and to maintain secure transfer of information between partners.

III.B High Temperature materials

Investigated materials for the control rod and turbine (blades and discs) include carbon-carbon composites, which offer advantages in strength, ductility and increased thermal resistance over metal alloys, and super-alloys (principally nickel based) used in industrial and other advanced gas turbines.

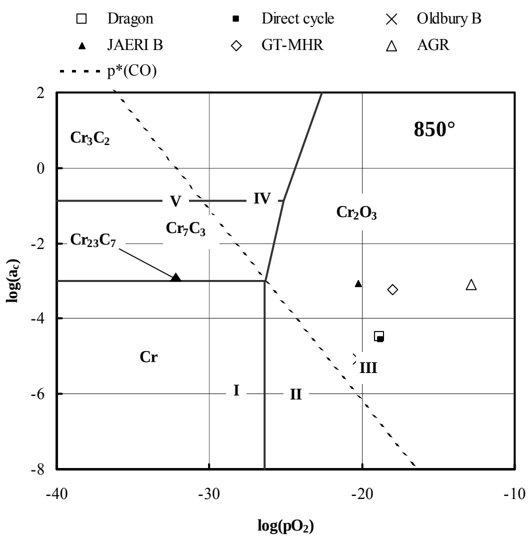
For the turbine, high temperatures and long-term endurance are key issues. The criteria for material selection are typically to be based on a safe operation period of up to 60,000 h, with upper temperature limits in the range 850 to 950^{o}C. The main criteria are creep and the influence of environment. Manufacturing considerations are a major factor for the disc. Candidate alloys must be capable of production of large defect free ingots with good forging properties and proven thermal stability. The need for cooling is a key issue since the review of available materials showed temperature limits for suitable disc materials to be below 750^{o}C.

For the turbine discs and blades susceptibility to corrosion is an important selection criterion. The helium coolant gas used in an HTR contains small levels of impurities (H_2, H_2O, CO, CO_2) at low partial pressures that can interact with the core graphite and metallic components and cause some loss or damage to their properties. Low concentrations of H_2O and H_2 are produced by leakage and / or desorption from both metal surfaces and graphite. CO, CO_2, and CH_4 may be produced by coolant / graphite reactions at high temperatures. Depending on the partial pressure of the impurities the resulting atmosphere can either be oxidising or carburising for the materials selected. The following table gives some information on He impurity levels from various sources

Description	Pressures in Pa				
	H_2	CO	H_2O	CO_2	CH_4
Dragon Normal maximum	1.5	0.8	0.015	0.08	0.15
Realistic Direct Cycle	3	2	0.5	-	-
Oldbury B boiler leak	50	50	5	-	5
JAERI Type B	20	10	0.1	0.2	0.5
GT-MHR normal	<35	<42	<14	<1	-
AGR	800	$4x10^4$	$1.7x10^4$	$4x10^6$	800

Table 1 Typical Gas Reactor Impurity levels

Damage may be either at the surface (formation of surface films involving oxides or carbides) or diffuse to significant depths into the metallic matrices (internal oxidation or carburisation or decarburisation) resulting in loss of mechanical properties over a significant thickness of the material leading to either loss of strength or local embrittlement and favour the initiation of cracks. Phase diagrams are used to determine the neutrality of the chemical activity and to determine whether compatibility issues are likely to have a strong impact on materials selection. For Ni based alloys phase diagrams are constructed based on Cr. Typical zones for carburisation and de-carburisation and formation of protective oxidation are identified at different temperatures and used to aid materials selection.



Regions I and II: Severe decarburisation
Region III: Protective oxidation plus slight carburisation
Region IV: Carburisation
Region V: Severe carburisation

Figure 2. Phase diagrams for Cr at 850°C in atmospheres containing H_2, H_2O, CO_2 and CO

Helium atmospheres from decomposition of methane under extremely low oxygen partial pressures are heavily carburising and can cause a significant shortening of the material creep life and accelerated creep crack growth rates. The presence of alloying elements such as cobalt (which is in most of the currently available turbine disc and blade materials) is difficult to avoid. The main issues are the potential for plate out and lift off of particles and their activation and prevention of them flowing through the core. The review and database work identified a few potential materials to be considered for the experiments.

For the reactor internals material selection is based on ability to resist the effects of irradiation at the highest gas temperatures. Such materials (e.g. AISI type 316 steel, Alloy 800H (control rod) and Hastalloy XR) have to cope with high thermal strains arising from power changes and load following requirements. AISI type 316 steel is normally used for components operating at temperatures up to 550-600°C and the last two, which have been extensively investigated for the HTTR, for higher temperatures (750°C). For the control rod, which compensates for fuel burn-up and power variation reactivity effects and is used to control the reactor operation under normal operation modes and to shutdown the reactor system, available metallic materials (such as alloy 800H) are considered to be at their operating limit, and new carbon based materials are being considered as alternatives. Such materials are expected to give potential for improved reactivity control during shutdown and for allowing the normal operating temperatures of future reactors to be increased .

A synthesis of possible materials was carried out to aid selection and to provide information for a build-up of irradiated and non-irradiated properties for assessment and database purposes. The findings show that properties are largely an-isotropic and are severely degraded by neutron irradiation, the level of degradation being dependent on irradiation condition and temperature. From the references studied limited irradiation induced properties were available although some general trends in behaviour were seen in properties such as thermal conductivity and thermal resistivity. Also the high cost and limited supply of suitably manufactured material for such an application meant that future testing would need to be very selective and specific to the component needs.

- Issues
- *Segmented containing absorber material*
- *Gaps between tubes and absorber rings need to be maintained under irradiation induced swelling*
- *Seismic considerations*
- *Less restriction on reactor control procedures*
- *High strength & Heat resistant (1300°C)*
- *Good properties under irradiation expected*
- *Less brittle than graphite*

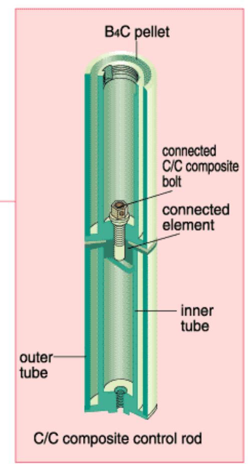


Figure 3 Control Rod Issues

III.C Graphite

The graphite core is a key component that affects safety and operability of the reactor. It provides structural support, coolant channels, moderation, and shielding while operating in a high temperature helium environment. Its performance is critically dependent on the graphite properties, which are irradiation dependent. The most important considerations are component integrity and changes in core geometry, both of which are affected by the dimensional change. Many of the graphites used in previous core designs are no longer available and there has been a serious decline in ability to manufacture nuclear grade graphite in large quantities, the main questions concern the availability of the coke and manufacturing procedure. Today's HTGR projects - HTTR (Japan) and HTR-10 (China) - use a Japanese graphite (IG-110) which, with its high strength, is suitable for exchangeable core components where low fast neutron fluences and hence low total doses are applicable.

In the past, only a small number of irradiation programmes were aimed at determining the irradiation behaviour of graphites at high temperatures, i.e. >600°C. Two of the most significant were for the European Dragon HTR programme, and the German HTR programme. Unfortunately most of the data obtained by other countries are confidential and cannot therefore be published in open literature. The work on the review and collection of graphite properties considers the accessible information on the IAEA database, internal information and published information at seminars and conferences. The IAEA database was established to help the development of International programmes on graphite-moderated reactors, assist safety authorities in assessment of safety aspects and serve as a source of scientific information for nuclear and non-nuclear technology. The data relevant to the irradiation temperature and neutron fluence domains for new HTRs will largely come from the new tests and built up for each graphite grade and sample orientation, and contain the appropriate details of the graphite i.e. grade, manufacturer, coke source, grain size and manufacturing method.

For the HTR-M programme three main graphite manufacturers were approached to see which graphites could be offered for the next generations of reactors. Five graphites were selected and identified for the programme, two iso-molded and two extruded graphites, manufactured from pitch coke and petroleum coke. A range of grain sizes (1mm down to 10 µm) was also selected. IG100 (HTTR graphite) was also chosen (iso-moulded graphite made using a petroleum coke) since this has previously been irradiated at high temperature, although only to a small / medium fluence. Nevertheless, the available data will provide a useful comparison with the data obtained from the new graphites. The selected graphites, test conditions and programme are discussed in more detail in section 4.

For nuclear applications, the graphite has to be as free as possible from impurities. Most of the impurities present will become activated during the operating life of the reactor, which will give rise to operational problems, as well as decommissioning and final disposal problems. Most impurities, however, are volatile and so disappear during graphitisation. To remove as much of the remainder as possible, halogens are added generally during graphitisation to aid the conversion of metal impurities and boron particularly, to their more volatile halides (extremely low boron levels are important from a reactor physics point of view, as it is a very strong neutron absorber).

IV. MATERIALS TESTING

IV.A Vessel Steel

The test programme for the vessel steel concentrates on qualification of modified 9Cr 1 Mo. For this purpose an irradiation test of thick welded joints is being prepared. The tests involve the use of the Lyra rig, (see figure 4) giving accurate control of the temperatures and use of a helium atmosphere. Heaters in the rig are used to correct the temperature levels obtained from gamma heating.

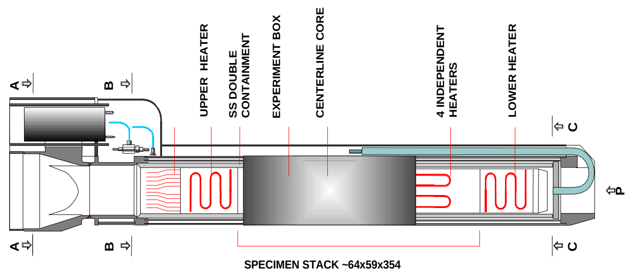


Figure 4 Lyra test rig used for Irradiation of vessel material within the HFR

Post irradiation tests include tensile, creep, Charpy and CT toughness tests. The fabrication and characterisation of the welded specimens involves tests on 40 and 150mm thick plates. The thinner plate specimens provide a means for trials using different weld bead lengths and different weld parameters before producing the final weld specimens with the thicker plate. The conditions used will simulate an upper bound to the expected accumulated dose at the end of life (between 10-20mdpa). The maximum dose rate per cycle in HFR at the available positions is 5mdpa, which suggests up to two to four cycles. This relatively low dose irradiation takes place in the Pool Side Facility of the high flux reactor (in contrast to the graphite irradiation which is an in core irradiation). It is possible to irradiate to higher levels, which will require more cycles and a longer irradiation time. For the tests the expected lowest temperature conditions are being considered, as only one test temperature is possible. Higher temperatures act to anneal out any irradiation damage effects.

Work is in progress to define the tests for post-irradiation and reference properties and final test selections and boundary conditions. The procurement of the material and the fabrication of the thick section welded test pieces is near completion and inspection and cutting plans for the range of different test conditions are being drawn up (typically see figure 5). Characterisation tests are to start shortly following machining with the irradiation of the specimens expected to start mid to late 2003.

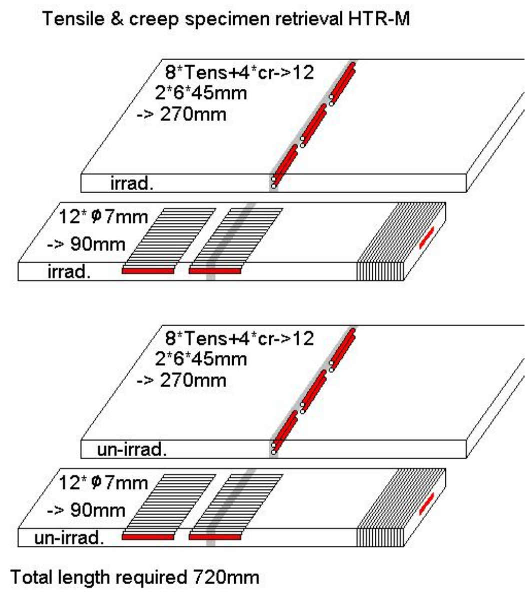


Figure 5 typical cutting plans for vessel tensile & creep specimens

IV.B High Temperature materials

Experiments on these materials will involve short and intermediate term tests. High temperature short-term mechanical / creep tests are planned for turbine disc and blade materials in simulated environments. For the blade material 2 grades are being examined (IN792 DS & CM 247 LC DS), for the disc 1 grade (Udimet 720). Udimet 720 is considered to have a potential for operation up to 700°C and to be the best available for this application using established manufacturing techniques. Manufacturing considerations are a key factor and producing large ingots without porosity and segregation is a major challenge for the size of disc being considered. The tests on the disc material will be carried out at 700 and 750°C. For the turbine blade materials the current view is to avoid single crystal materials for the blades and test two grades of material directionally solidified materials (Cr and Al formers) under tensile and creep conditions, at temperatures up to 850°C. Procurement of suitable quantities of the materials is being made, and characterization tests on the disc material have started. The duration of the intermediate creep tests is expected to be around 3000h and will be performed on one of the turbine material grades and will include aged and notched samples.

The test matrix and proposals for the test conditions will as far as possible bound the temperature and transient cases experienced by the turbine. Four environments will be used to cover fully carburised, decarburised, reference heat-treated and delivery condition. The setting up of the test rig and associated programme are underway and tests are expected to start mid 2003.

Figure 6 Carburising test rig

IV C Graphite

The irradiation experiment will use a test rig that allows distinct temperature levels to be obtained. Irradiation temperatures are higher than 10 years ago and there are three temperatures of major interest (600°C, 750°C and 900°C). A temperature of 750°C, was considered to be most relevant for new designs. The rig design will require the stacking of up to 150 samples, 8mm diameter (either 6 or 12mm in height), covering two main directions (15 samples per grade per direction). Pre and post irradiation measurements will include dimensional changes, Young's modulus, CTE and thermal conductivity. A schematic of the measurement facility for Young's Modulus is shown in figure 7. A companion paper in this conference, discusses issues concerning the derivation of thermal conductivity with respect to graphite[4].

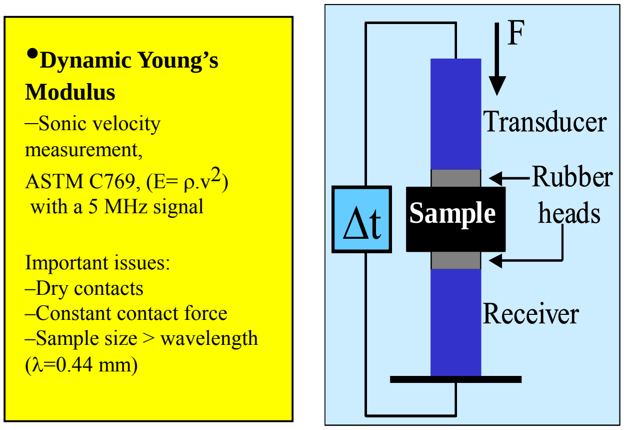


Figure 7 Youngs' Modulus Graphite measurement

The current programme uses currently available graphites since the development of a new material would take 3-5 years. The graphites proposed by the manufacturers are as follows.

Grades suggested by UCAR:	Pitch coke	Pet coke
Extruded	**PPEA**	PCEA
Iso-molded		**PCIB-SFG**
Grades suggested by SGL:	**Pitch coke**	**Pet coke**
Extruded	NGB-10	**NBG-20**
Iso-molded		**NBG-25**
Grades suggested by Toyo Tanso:	**Pitch coke**	**Pet coke**
Extruded		
Iso-molded	IG-430	**IG-110**

Table 3 Graphites proposed for irradiation tests

Although five (major) grades of graphite have been selected for full testing in the experiment (see table 3), it was decided that it would be useful to have an early indication of the irradiation behaviour of some of the other proposed graphites. These could be included as 'piggy-back' samples in the irradiation capsule. The (minor) graphites selected are UCAR grade PPEA, SGL grade NBG-20 and Toyo Tanso grade IG-430.

The irradiation test will provide a number of data points by using the flux (buckling) distribution available in the HFR capsule. A third order polynomial can describe the behaviour and a sufficient number of points should be obtained from the test to describe the distribution.

Ideally the test conditions for the experiment would be those that give data for candidate graphites up the peak end of life fluence. However this requires a significant effort over a long time scale, typically 10 years. The target flux in the current four-year programme will be as high as possible. The current available locations in the HFR suggest an irradiation for 1-1.5 year to EDN fluence ~6-7 10^{25} m^{-2} (approx. 8 dpa). This is not expected to yield properties up to turn round behaviour but will provide information on whether the graphite is useful or not. The current frame is for a first sorting and as the experiment progresses possibly replacing those that are unsuitable with alternatives. For the highest flux position in HFR a total of 3 years will be needed for the irradiation.

Irradiation will start in the first half of 2003 with first PIE in the fall of 2004 with a view to completing and continuing the test within the context of the 6th Framework.

The graphite oxidation work involves two principal tasks relevant to safety analysis and licensing of HTRs for normal operation.

- The improvement of experimental database for advanced graphite oxidation models.
- Experimental investigation of innovative C-based materials with respect to their application on HTRs.

This experimental work uses the thermo-gravimetric facility THERA and the induction furnace facility INDEX. Graphite burning under severe air ingress accidents, is usually assessed using computer models, based (up to now) on isothermally measured kinetic equations that consider in-pore diffusion and chemical reaction mechanisms. The data requirements for such models are burn off dependent chemical (regime 1) reactivities and in-pore diffusion coefficients. THERA was used for the regime 1 (see figure 8) and INDEX for regime II where higher flow rates are needed. Long-term experiments at low temperature in air were done in an annealing furnace.

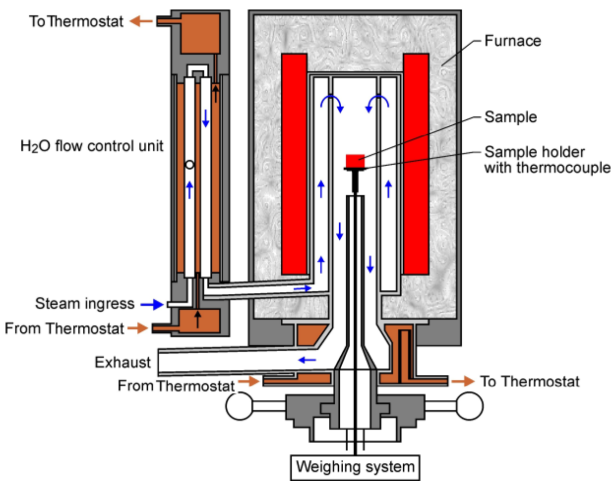


Figure 8 Schematic of THERA test facility

The second series of experiments looked at the performance of CFC materials under accident typical temperatures (about 1000 ^{o}C) in steam and in air. Oxidised gas flowed through the inner bore hole of the tube with flow rates sufficiently high to suppress the influence of boundary layer mass transfer on kinetics. Oxidation effects were measured by mass spectrometric analysis of the product gases and by weight loss of the sample. Experiments were also performed to determine properties of CFC materials irradiated and non-irradiated (up to 1dpa). These include measurements of strength, dimensional change, thermal diffusivity and heat capacity. The main findings from this graphite oxidation work are reported separately in a companion paper [5] in this session.

Sample	Description	Density [kg/m^3]
A3-3	**HTR fuel element matrix graphite consists to 90 % of a filler graphite and 10 % of a coked resin binder**	**1735**
NB31	**3D-CFC**	**1920**
NS31	**3D-CFC infiltrated with 8 – 10 % liquid silicon**	**2120**
AO5	**2D-CFC**	**1870**
V483T5	**fine grain nuclear graphite**	**1810**
RG	**well graphitised standard HTR graphite with a low ash content**	**~1750**

Table 2 Graphite Corrosion Tests – Investigated materials in steam

V. SUMMARY AND CONCLUSIONS

This paper reviews research activities on the HTR-Materials projects in support of the modular HTR technology development within Europe. Overall the projects are progressing well with review and database investigations maturing and planning and preparations for the experiments well advanced. The graphite oxidation experiments are concluding and results are available. The irradiation-testing phase of the work on vessel steel and graphites is expected to start shortly and post irradiation examination of specimens planned for the fall of 2004. The high temperature short and medium testing phase will occupy much of 2003 and 2004 with reporting planned for the end of next year.

V1. ACKNOWLEDGEMENTS

The projects HTR-M & HTR-M1 are being co-sponsored under the Fifth Framework programme of the European Atomic Energy Community ("Euratom"), contracts FIKI-CT-2000-0032 and FIKI-CT-2001-00135. The information provided herein is the sole responsibility of the authors and does not reflect the Community's opinion. The Community is not responsible for any use that might be made of the data appearing in this publication.

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