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## High Temperature Reactor Materials: Progress of HTR-M Projects

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|---|---|---|
| D.E. Buckthorpe | NNC Ltd. | , UK |
| M. Davis | NNC Ltd. | , UK |
| R. Couturier | CEA | , France |
| J. van der Laan | NRG | , Netherlands |
| J. Hegeman | NRG | , Netherlands |
| A. Vreeling | NRG | , Netherlands |
| B. Riou | FRA | , France |
| H. Rantala | JRC | , Netherlands |
| P. Ennis | FZJ | , Germany |
| K. Kuehn | FZJ | , Germany |
| G. Haag | FZJ | , Germany |
| A. Buenaventura | EA | , Spain |
| B-C. Friedrich | FRA | , Germany |

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**D. Buckthorpe, M. Davies**
NNC Ltd., UK

**R. Couturier**,
Commissariat à l' Energie Atomique (CEA.), France

**J. van der Laan, J. Hegeman, A. Vreeling**
Nuclear Research and Consultancy Group (NRG), Netherlands

**B. Riou**
Framatome ANP, France

**H. Rantala**
European Commission, Joint Research Centre (JRC), Institute for Energy, Netherlands

**P. Ennis, G. Haag, K. Kuhn**
Forschungszentrum Juelich GmbH (FZJ.ISR), Germany

**A. Buenaventura**
Empresarios Agrupados Internacional S A (EASA.MD), Spain

**B-C. Friedrich**
Framatome ANP (GmbH), Germany

## Abstract

HTR projects have been launched within the 5$^{th}$ Framework Programme of the European Commission to consolidate and advance HTR technology in Europe. This paper considers the mid-term results from the project HTR-M, which looks at the materials requirements for the HTR reactor pressure vessel, the high temperature resistant alloys for the internal structures and turbine, and graphite for the reactor core. The programme of work includes irradiation testing of vessel steel and graphite materials and the main focus of the paper is on the progress of this test work.

## 1. Introduction

The HTR-M project is one of a cluster of projects being performed within the European Union Fifth Framework Programme [1] investigating research and development issues connected with the High Temperature Reactor (HTR). The HTR-M project focuses on the selection and development of materials for the key components of the HTR, namely, the reactor pressure vessel, the high temperature structures (specifically the turbine) and the graphite core. This work started in November 2000 and involves seven partners from five European countries [2]. Additional materials activities (HTR-M1) were started in 2001 to introduce medium term creep tests for the turbine materials and begin a programme of testing on the irradiation properties of graphites. Both projects extend over a period of four years and are being co-ordinated through the direction of a European HTR Technical Network (HTR-TN). This paper gives a brief outline of the HTR-M / M1 programme and presents and discusses some of the mid-term results and ongoing actions, focusing especially on the irradiation tests being performed on vessel steel and graphites.

## 2. HTR M & M1 Projects

The main elements of the work programmes are summarised below:

***Vessel (RPV):***
- Review existing materials used in gas-cooled reactors and previous high temperature reactors
- Set up a materials database on design properties (which will lead to identification of data omissions for future R& D testing)
- Carry out specific tests on welded joints under irradiated and non-irradiated conditions.

Fabrication and the effects of irradiation/environment on the vessel weld behaviour are considered crucial issues in determining the structural viability of the material for future RPV application.

***Turbine (disc & blade):***
- Review existing materials used in gas-cooled reactors and previous high temperature reactors
- Set up a materials database on design properties (which covers a few potential materials for each component)
- Perform specific tests on selected materials (carbon/carbon (C/C) composites, high alloy steels) at temperature and under short and intermediate times in air, vacuum and helium.

The main concern is the ability of the material to withstand the temperatures and conditions in the direct cycle environment. Creep and effects of helium on properties and strength are crucial issues.

## References


[1] J Martin-Bermejo, M. Hugon, G. Van Goethem, "Research activities on High Temperature Gas-cooled Reactors (HTRs) in the 5th EURATOM RTD, Framework Programme", Paper No.1560, SMiRT 16, Washington DC, USA, August 12-17, 2001

[2] D. Buckthorpe, R. Couturier, B. van der Schaaf, B. Riou, H. Rantala, R.Moormann, F.Alonso, B-C. Friedrich, Investigation of High Temperature Reactor (HTR) Materials, NEA/OECD, 2nd Information Exchange meeting on High Temperature Engineering, Paris, on 10-12 October 2001

***Graphite core:***

- Review of existing graphite materials irradiation data and formulation of a database of available information.
- Identify which of the currently manufactured graphites would be suitable for a HTR
- Study of irradiation testing of a few selected graphites.
- Graphite oxidation and the investigation of the consequences of severe air ingress with core burning, including the development of models and data, protective coatings and C-based materials.

The graphite programme is an important first step in establishing suitable materials for future HTR's given that almost all the graphites previously irradiated are no longer manufactured. Note that of the test results that do exist, most use irradiation temperatures <550°C and so are not representative of HTR temperatures

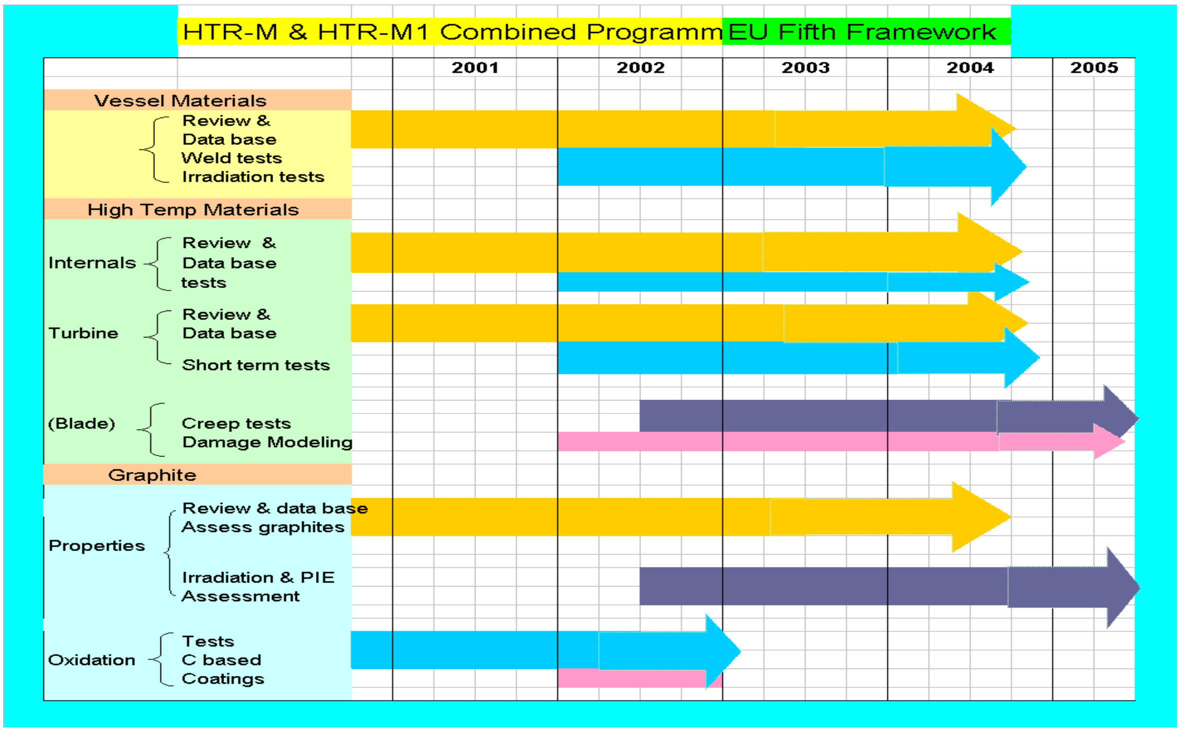



**Figure. 1 Description of Overall HTR-M & M1 Materials Programme**

The projects therefore consider two main areas of investigation: the review of past experience and assembly of available properties (including available design information) plus tests on key materials where necessary information is considered lacking or scarce.

## 3. Technological survey & database for structural integrity

The need for reliable material property information is a key issue in the development of any innovative reactor technology. For the HTR its especially important in view of the strong impact the environment will have on behaviour.

***Vessel:***

For the reactor pressure vessel assurance of safety is of paramount importance. HTR vessel materials will operate at temperatures higher than 450$^{o}$C and information on materials behaviour during manufacture, in the operating environment (neutron-irradiation fluence, operational temperature, and helium environment) and welding are needed for assurance of integrity. Two vessel options are considered in the technological survey:

The ‘cold’ vessel design concept adopted for the PBMR reactor pressure vessel uses a Mn-Ni-Mo or C-Mn type steel with grades similar to SA 508 Class 2. The advantage for these steels is the large experience that can be taken from 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 9Cr1Mo steel grades similar to ASME Grade 91 being a prime candidate. For the GT-MHR the operating conditions are around 400 – 460°C with potential temperature excursions up to 570°C.

Thermal and mechanical loads, in combination with environmental conditions can enhance degradation and can have a significant effect on reliability. The main safety and 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 degradation mechanisms (irradiation, thermal ageing, temper-embrittlement and corrosion) the main environmental considerations. The effects of irradiation on the toughness of LWR steels have been studied extensively elsewhere. The major effect is an increase in the ductile brittle transition temperature (DBTT) due to the combined effects of matrix hardening and grain boundary weakening. This is often associated with the residual elements phosphorus (P) and copper (Cu). For LWR this threat is generally considered to be significant when irradiation causes a change from ductile to brittle fracture at temperatures within the operational envelope. For the warm vessel option, no data are available for modified 9Cr1Mo steel irradiated under conditions expected for the HTR. Information has however been obtained at much higher doses (≥1dpa) and these show 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 is no information on the behaviour of modified 9Cr 1Mo steel welds.

On thermal ageing embrittlement (TAE) the base materials and weld metals of both cold and warm HTR vessel steels normally have very fine grain sizes and therefore the most likely embrittlement location is the coarse-grained heat affected zone. This is a narrow region adjacent to the fusion boundary where temperatures of 1100 to 1300°C are reached during welding. Cold vessels operate at temperatures below the embrittlement range (350-550°C) so thermal ageing embrittlement is only possible during transients involving exposure to higher temperatures for tens or hundreds of hours. Warm vessels on the other hand operate in the TAE temperature range throughout their lifetime and therefore TAE is of greater concern with regard to vessel integrity.

For corrosion, the main concerns are on metal loss/ carburisation/ decarburisation due to actions of the impurities in the helium coolant. For the HTR vessel, metal losses will be either by controlled carburisation in cold wall vessels, and decarburisation combined with internal oxidation in warm wall vessels. The kinetics of these processes determine the effective metal loss.

For the developing database materials such as 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 9Cr1Mo are considered. The potential for 12Cr steels will also be investigated. Aspects such as composition, manufacturing information, test and design data plus operating environment are included in the database. The materials property database includes code data where available, and raw data for analysis. Modified 9Cr1Mo steel has the most uncertainty on data and fabrication experience with respect to HTR characteristics and potentially has the wider applicability. Hence this material was selected 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 via a web based system to allow remote access by all the partners and to maintain secure transfer of information between the different partners and countries.

***Turbine (disc & blade):***

For the turbine, high temperatures (850-900°C) and long-term endurance (60,000h) are key issues. The main concerns are creep and the influence of the environment. Manufacturing considerations are especially important for the disc and blades. Candidate alloys for the disc must have good forging properties and proven thermal stability. For the blades cast material is not sufficient, and so directionally solidified or single crystal alloys have to be considered. The review suggests that the need for cooling is an important issue for both disc and blades. For current disc materials temperature limits are in the region of 750$^o$C, suggesting that a cooled disc could be necessary for the design.

The impact of corrosion is also an important selection criterion for the turbine disc and blade materials. The helium coolant gas used in an HTR contains small levels of impurities ($H_2$, $H_2O$, CO, $CO_2$) at low partial pressures, these can interact with the core graphite and metallic components and cause some degradation of 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 oxidizing or carburising for the selected materials.

Damage may be either at the surface or may diffuse to significant depths in the metallic matrices (internal oxidation or carburisation or decarburisation) resulting in a loss of mechanical properties over a significant thickness of the material. As with the RPV, 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 were constructed based on Cr levels. Typical zones for carburisation and de-carburisation and formation of protective oxidation were identified at different temperatures and used to aid materials selection.

Impurities in helium atmospheres, arising 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 going through the core.

The review and data base work identified some potential materials to be considered for the experiments. Investigation of turbine disc materials revealed Udimet 720 to be the best currently available candidate that would not require significant manufacturing development. For the perceived endurance this is thought to have a temperature ceiling of around 700-750$^{o}$C and is likely to require cooling. Two DS grades of material were selected for the blade tests (CM 247 LC as an Al-oxide former and IN 792 as a Cr-oxide former). Because of corrosion considerations, the current view is that any blades may have to be coated for long life. The choice of coating remains to be made and tested in a future Framework Programme. Adoption of a cobalt free material was not considered a priority.

For the reactor internals, material selection is based on the 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$^{o}$C and the other two, which have been extensively investigated for the HTTR, for higher temperatures (750$^{o}$C). For the control rod, which compensates for fuel burn-up and power variation reactivity effects and is also used to control the reactor operation under fast normal operating modes and to shutdown the reactor system, available metallic materials (such as alloy 800H) are considered to be at their operating limit. 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 carbon based materials was carried out to aid selection and to provide information on irradiated and non-irradiated properties for assessment and data base purposes. The findings show that the properties are largely an-isotropic and are severely degraded by neutron irradiation, the level of degradation being dependent on level of irradiation and temperature. From the references studied, only limited information on 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 was prohibitive.

***Graphite core:***

Graphite behaviour has important safety implications because of structural and property changes that occur when it is irradiated. Information on behaviour is crucial and design / material property data under HTR relevant conditions is needed for candidate materials for both normal and accident conditions. The most important considerations are component integrity and changes in core geometry, both of which are affected by the fast neutron induced dimensional change that graphite exhibits. Many of the graphites used in previous core designs are no longer available and there has been a serious decline in the ability to manufacture nuclear grade graphite in large quantities. Today's HTGR projects - HTTR (Japan) and HTR-10 (China) - use a Japanese graphite (IG-110) which has a high strength and is suitable for exchangeable core components where low fast neutron fluences and low total doses are applicable.

In the past, only a small number of irradiation programmes have looked at the effects of irradiation on the 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 e.g. are confidential and cannot therefore be published in the open literature. This review and collection of graphite properties considers the published information plus available internal information within the project partners. The data relevant to the irradiation temperature and neutron fluence domains for new HTR's will largely come from the new proposed tests, and will need to be built up for each graphite grade. The database of information will contain 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:- three iso-moulded and two extruded graphites, manufactured from pitch coke and petroleum coke with a range of grain sizes (1mm down to 10 μm). One of these, IG110 (an iso-moulded graphite made using a petroleum coke and used in HTTR and HTR-10) was selected since it has previously been irradiated at high temperature, although only to a small/medium fluence. Nevertheless, the data will provide a useful comparison with the data obtained from the other selected graphites. The selected graphites, test conditions and programme are discussed in more detail in the next section.

For nuclear applications, the graphite has to be as free as possible from impurities. The impurities present will become activated during the operating life of the reactor. This 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.) The difficulty is that the use of fluorides is prohibited, and traces of the chlorine remain in the graphite. The latter becomes chlorine-36 which gives rise to active waste disposal problems.

## 4. Testing under irradiated and non-irradiated conditions

The technological survey and database work identified significant shortfalls in information for the proposed steels and graphites being considered for future HTR's. A test programme was therefore established to fill some of the most important gaps.

***Vessel:***

For the vessel, the test programme concentrates on qualification of Mod 9Cr 1 Mo, which has a much wider potential operating temperature range. The test programme involves irradiation tests in the High Flux Reactor (HFR) at Petten in the Netherlands and focuses on qualification through a series of mechanical tests to look at the integrity of the welded joints. The un-irradiated tests provide the reference condition from which the effects of irradiation will be assessed. The HFR LYRA irradiation facility, which allows accurate control of the specimen temperatures during irradiation and use of a helium atmosphere, will be used for the irradiation (Figure 2). Tungsten plates shield the specimen holder to avoid high gamma heating and electrical heaters are used to maintain the nominal temperature levels. The temperature levels are measured using 24 K type thermocouples within the irradiation rig.

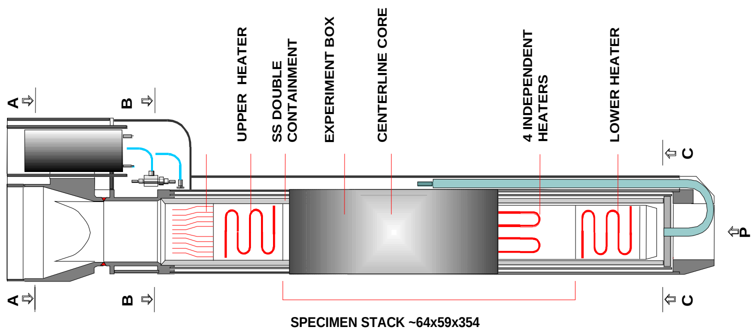
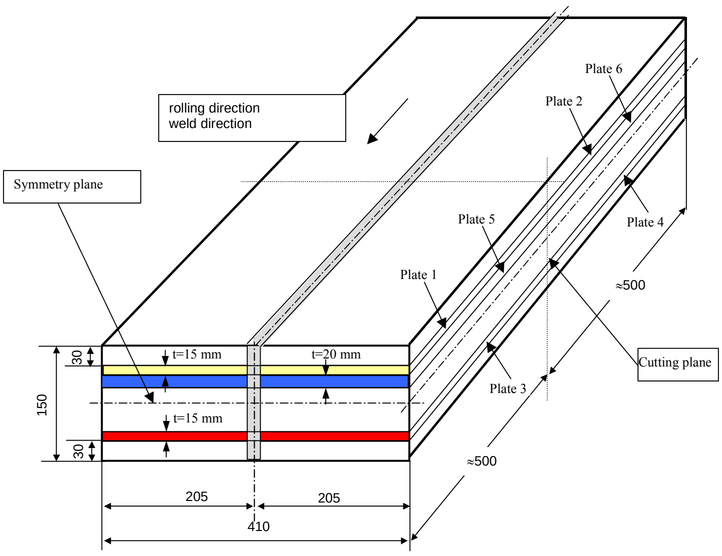


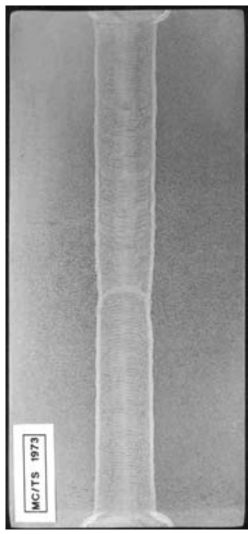

**Figure 2 Lyra test rig used for Irradiation of vessel material within the HFR**

Specimens for mechanical testing are selected from welded joints manufactured using a narrow gap Tungsten Inert Gas (TIG) process. The plate is 150mm thick with a weldment length of approximately 1 m. A schematic drawing of the specimens plus macrographic cross section are shown in Figure 3. The specimens are to be tested in the main stress directions with sections taken from cutting planes of approximately 15mm thickness and parallel to the weld direction. The tests are for both the reference condition and End of Life (EOL) condition. The root, bottom and top will be avoided in order to produce well-specified and homogeneous material. The mechanical tests include the following:

- Impact tests (sub-size and full sized Charpy) – KLST type specimens
- Tensile tests – 20mm gauge length
- Creep tests to rupture – same as tensile
- Fracture toughness tests – CT specimens




Typical cutting plane of weld joint




Typical macrographic cut of welded joint

**Figure 3 Vessel welded Joint**

Four conditions are selected for the mechanical testing: plate material in the parallel and perpendicular directions (with respect to the rolling direction), the HAZ, and the weld metal. The specimen geometry for the reference testing and post irradiation examination (PIE) testing are the same for all specimen types. The irradiation will take place in the pool side facility of HFR at a single irradiation temperature of 375°C. The estimated EOL fluence is $8 \cdot 10^{22}$ n/m$^2$, E>0.1 MeV (~10 mdpa, extendable up to 20mdpa), which can be achieved in two or more cycles. After irradiation, the flux buckling and self shielding will be determined using neutron monitoring sets. The specimen fluence gradients will be kept as low as possible.

The detailed design of the irradiation rig and the loading scheme are complete. Manufacturing of rig components are underway. The start of irradiation is planned for September 2003. A series of round robin tests between NRG and JRC have also started and a comparison will be made before the irradiation tests begin. NRG will perform the reference (non-irradiated testing) and NRG the PIE.

***Turbine (disc & blade):***

Experiments on these materials will involve short and intermediate term tests. High temperature short-term mechanical/ creep tests are proposed for the turbine disc and blade materials in four simulated environments:

- As received with ageing heat treatment – I
- Decarburised – II
- Carburised – III
- After the carburisation or decarburisation heat treatment – I(mod)

All tests will be carried out in air. Ageing will be carried out in an argon atmosphere, using oversize blanks. The test matrix and proposals for the test conditions will as far as possible bound the temperature and transient cases expected to be experienced by the turbine.

For the blade material two grades are being examined (IN 792 DS & CM 247 LC DS), and for the disc one grade (Udimet 720). Udimet 720 is considered to have potential for operation up to 700°C and to be the best available using established manufacturing techniques for this application. 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 to test two grades of directionally solidified materials (Cr- and Al-oxide formers) under tensile and creep conditions at temperatures up to 850°C. Procurement of suitable quantities of the materials is complete, and characterization tests on the disc material have started. The duration of the intermediate creep tests is expected to be up to 3000h. Tests will include the disc and blade turbine material grades and aged samples. The setting up of the test rig for the treating the materials is in progress and conditioned specimens are expected to be available for testing by the end of 2003.

***Graphite core:***

The graphite irradiation experiment (called INNOGRAPH) will use a test rig that allows distinct temperature levels to be obtained. The temperature range of interest is 550°C to 950°C). A temperature of 750°C was selected for these first tests as it is in the middle of the range, and could correspond closely to the temperature at the peak flux position. The rig design requires the stacking of up to 150 samples, 8mm diameter (either 6 or 12mm in height), covering two main directions i.e. with grain and against grain. For five selected graphites this gives15 samples per grade per direction. Pre and post irradiation measurements include specimen dimensions, Young's modulus, coefficient of thermal expansion (CTE) and thermal conductivity.

The programme uses currently available graphites (including some recently developed). The development of a new material would take 3-5 years. The graphites selected for testing are given below in Table 1. Large graphite blocks have been delivered to NRG for sectioning and machining. The specimen shape is cylindrical with a flattened plane along its length. This plane is used to retain the z-direction of the original graphite blocks. Although five (major) grades of graphite have been selected for full testing in the experiment (shown in Bold Italics in Table 1), it was decided that it would be useful to have an early indication of the irradiation behaviour of some of the other candidate graphites. These will be included as 'piggy-back' samples in the irradiation capsule. The (minor) graphites selected are shown in Table 1 in brackets.

The irradiation test will provide a number of data points by using the flux (buckling) distribution available in the HFR capsule. A sufficient number of points should be obtained from the test for each grade to describe its dimensional change behaviour, and the variation of the important properties, over the full fluence range.

Ideally the results from this experiment, and hopefully others in the future, will give all the necessary data for candidate graphites up to the peak EOL fluence, and at incremental temperatures covering the full range. 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 years to an EDN fluence of $\sim 6\text{-}7\ 10^{25}\ m^{-2}$ (approx. 8 dpa). This is not expected to yield properties up to turn round behaviour but will provide information on whether the graphite is useable or not. The current work 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. It is hoped that such a position will become available in the near future.

**Table 1 Graphites proposed for irradiation tests**

| **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*** | (NGB-20) |
| Iso-molded | | ***NGB-25*** |
| **Grades suggested by Toyo Tanso** | | |
| | **PITCH COKE** | **PET COKE** |
| Extruded | | |
| Iso-molded | (IG-430) | ***IG-110*** |

The Irradiation will start in the Autumn of 2003 with the first PIE in the spring of 2005 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 HTR's for normal operation:

- improvement of the experimental data base for advanced graphite oxidation models
- experimental investigation of innovative C-based materials with respect to their application to HTR's.

This experimental work [3] uses the thermo-gravimetric facility THERA and the induction furnace facility INDEX available at FZJ. 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 mechanisms. The data requirements for new advanced oxidation models are burn off dependent chemical (regime I) reactivities and in-pore diffusion coefficients. THERA was used for the regime I (chemical reactivities); diffusivity data are from DIVA and from the literature. In addition, for model validations, INDEX experiments were performed for regime II. 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 the kinetics. Long-term (regime I) experiments at low temperature in air used an annealing furnace.

The second series of experiments looked at the performance of CFC materials under typical accident temperatures (about 1000 $^oC$) in steam and in air. Oxidation effects were measured by weight loss of the sample. Experiments were also performed to determine the properties of CFC materials irradiated and non-irradiated (up to 1dpa). These included measurements of strength, dimensional change, thermal diffusivity and heat capacity. The thermal diffusivity of NB31 and NS31 decreased under irradiation (1 dpa, 200 °C) by factors of 3 (800 °C) to 10 (100 °C).

**Table 2 Graphite Corrosion Tests in steam – Investigated materials**

| **Sample** | **Description** | **Density [kg/m$^3$]** | **Rate [%/s] for 1173 K** | **at Burn off [%]*** |
|---|---|---|---|---|
| **A3-3** | HTR fuel element matrix graphite consists to 90 % of a filler graphite and 10 % of a coked resin binder | 1735 | 1,10E-03 | 3,8 |
| **NB31** | 3D-CFC | 1920 | 7,64E-04 | 25,2 |
| **NS31** | 3D-CFC infiltrated with 8 – 10 % liquid silicon | 2120 | 5,50E-04 | 5,0 |
| **AO5** | 2D-CFC | 1870 | 5,67E-04 | 32 |
| **V483T5** | Fine grain nuclear graphite | 1810 | 6,33E-04 | 50 |

* (rate maximum)

[3] Behaviour of Carbon based materials in contact with oxidising gases, paper 3031, ICAPP 03 Conference, Cordoba, Spain, 4-7May 2003.

## Summary and conclusions

This paper reviews the research activities on the HTR-Materials projects in support of the modular HTR technology development within Europe. The review and data base investigations on the vessel steels, turbine alloys and C-based materials and graphites are maturing and planning and preparations for the experiments are well advanced. The graphite oxidation experiments have concluding and results are available [3]. The irradiation-testing phase of the work on vessel steel and graphites will start shortly. Post irradiation examination of specimens is planned to begin in 2004 for the steel and Spring of 2005 for the graphite. The high temperature short and medium testing phase will occupy much of 2004 with reporting planned for the end of next year.

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