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

3rd Year Annual Scientific/Technical report for the HTR-M1 Project

by

D.E. Buckthorpe

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Executive Summary

The HTR-M1 project was launched within the 5th Framework Programme to consolidate and advance HTR materials technology in Europe. Work was underway within the HTR-M project to highlight the requirements and establish a materials platform for the HTR technological developments focusing on the reactor pressure vessel, high temperature resistant alloys for the internal structures and turbine, and graphite for the reactor core. The HTR-M1 programme extends the materials platform further with a special emphasis on the effects of creep and irradiation damage focusing specifically on intermediate duration creep testing of the high temperature materials for the turbine and irradiation testing of manufacturer recommended graphites for the selection of graphite for the core. The combined results up to the third year of the HTR-M1 project are summarised in this report.

Creep tests on turbine materials

Turbine materials operate at temperatures of 850-900°C (HTR) and up to 1000°C (VHTR). High strength alloys are being considered for the blades that are capable of withstanding such temperatures and conditions in the direct cycle environment for operating periods approaching 60,000h. Creep strength and the corrosive effects of helium impurities on the properties and strength of the materials are important issues that must be evaluated. Manufacturing considerations are especially important and candidate alloys must have good properties and proven thermal stability. For the blades, cast material is not sufficient, and so directionally solidified alloys have been considered. The HTR-M investigations have highlighted the need for cooling and possibly coatings for both disc and blade alloys. Impurities in the helium at temperature can result in carburisation and/or de-carburisation of the super-alloys and lead to a lessening of material strength. From HTR-M both an Aluminium former and Chromium former were selected as potential options for the 850°C gas temperature case.

The turbine test programme within HTR-M1 covers medium term creep tests under simulated HTR environments. The impact of corrosion is an important selection criterion since the coolant gas used in an HTR contains small levels of impurities (H_2, H_2O, CO, CO_2) at low partial pressures, and these can interact with the core graphite and metallic components and cause degradation of their properties. The blade materials chosen were CM 247 LC DS and IN 792 DS with tests conducted using four simulated environments:

- as received with ageing heat treatment – I;
- decarburised – II;
- carburised – III;
- after the carburisation or decarburisation heat treatment – I(mod).

Within HTR-M1 The blade tests are performed for periods of up to 10,000h and at temperatures up to 850°C. HTR-M results in air show material strength data to be consistent with available published results and the CM 247 LC DS to have the better high temperature properties. Tests on treated materials remain to be completed to confirm this. All treatments and mechanical testing will be completed within the extension period of the HTR-M1 project (see below).

Tests on graphite

Graphite acts as a moderator and structural component and is important to safety because of the structural and property changes that occur when it is irradiated. The graphite core

also has to resist corrosion (oxidation) particularly under severe air ingress accidents. Material property data under HTR relevant conditions are required for candidate materials in order to select the best available graphite for the design of the reactor core. Given that almost all the graphites previously irradiated are no longer manufactured and that of the irradiated test results do not yet exist for current grades this graphite work is recognised as an important first step in establishing suitable materials for the future modular HTR and VHTR. A review of past information and the basis for the development of a database was established within HTR-M. It is considered that for the new HTRs and VHTRs a new graphite is required that will need significant development and confirmatory irradiation tests and because of the extent of the R&D needed, global sharing of information through international cooperation would seem to be a necessity.

Tests have been carried out within HTR-M1 to investigate the effects of oxidation under severe air ingress accidents for the candidate graphites chosen for the irradiation tests. The tests consider the graphite behaviour in air and oxygen at temperatures of 650 and 700^{o}C. Eight graphites were investigated and all but one (NBG 20) was shown to be acceptable. A number of possible causes were considered but to date no obvious reasons for the differences have been found. It is planned to continue the explorations of this graphite within the 6th Framework as part of the graphite investigations within the VHTR-IP.

In addition work has been performed to review the progress made on SiC coatings in Germany. This programme successfully achieved a very good corrosion resistant quality coating on IG 110 and V438 graphites with no damage or weight loss observed after being irradiated and corroded and dropped from a height of 50 cm. Although the original objective, to develop a coating for A3-3 graphite, was not achieved, the results obtained are a very positive indicator of its potential

For the graphite irradiation programme five graphites were selected (supplied by three manufacturers), two iso-molded and two extruded graphites, manufactured from pitch coke and petroleum coke plus IG100 (HTTR graphite), iso-moulded made using a petroleum coke, to provide a reference. A range of grain sizes (1mm down to 10 µm) was also selected. Additional 'piggy-back' samples were included in the irradiation capsule to extend the knowledge of other grades. The irradiation test provides a number of data points within a single test run by using the flux (buckling) distribution available in the HFR capsule. It is not possible within the current project timescales to provide data for candidate graphites up the peak end of life fluence. The current available locations suggest an irradiation for 1-1.5 year to EDN fluence ~6-7 1025 m-2 (approx. 8 dpa)). Although this may not yield properties to turn round behaviour it will provide information on whether the graphite is useful or not and provide an opportunity of replacing those that are unsuitable. This irradiation considers physical property changes at one temperature (750^{o}C) and requires continuation in the next framework programme not only to complete the information up to 'turn-round' but also to look at different temperatures. The irradiation in HFR started in February 2004 and is expected to be completed (including PIE) for the end of 2005.

Overall programme

This report presents the current status of the work approximately three quarters the way through the HTR-M1 programme. An extension of the HTR-M1 Programme has been requested to enable the work being done on graphite irradiation to be fully completed and reported before the end of the project. All treatments and testing of the turbine materials and irradiated graphite samples will be completed within the extension period of the HTR-M1 project.

1 Scientific and Technical Objectives

The need for reliable material property data is a key issue in the development of the HTR and VHTR technology. The development of advanced HTR concepts requires an understanding of material behaviour plus data information under representative reactor operating conditions for the main HTR components important to safety and feasibility. The HTR-M1 project deals with the selection and development of materials for high temperature components of the turbine (direct cycle) and the graphite core with specific experiments on creep and irradiation. The main objectives are:

- to add to the HTR materials platform on which to develop a future HTR technological basis within Europe;
- to identify and test materials of interest and establish the information on an HTR database of information.

The scientific and technical objectives for the two main areas of work are summarised as follows.

High Temperature Materials – (turbine disc & blade)

- Perform intermediate creep tests on HTR-M selected turbine materials (high alloy steels) at temperature in air, and under treated (corrosive) conditions.

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.

Graphite core

- Identify which of the currently manufactured graphites would be suitable for an HTR.
- Perform irradiation testing plus P.I.E. of a few selected graphites recommended by manufacturers.
- Graphite oxidation and the investigation of protective coatings.

The graphite programme is an important first step in establishing suitable materials for future HTRs 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 $<550^{o}C$ and so are not representative of HTR temperatures

2 Scientific and Technical Progress

The project considers two main areas of investigation as described above. The following sections summarise the scientific and technical progress made up to the end of the 3rd Year of the HTR-M1 Project.

2.1 Work package 1 of HTR-M1 – Intermediate creep tests on turbine materials

This task covers intermediate creep testing of turbine blade materials under simulated reactor conditions. A description of the test programme and planning for the tests is given in HTR-M 02/9-D-1-1-7 (see Table2).

The HTR-M1 creep tests are carried out on two turbine blade alloys (CM 247 LC DS and IN 792 DS) for durations of up to 10,000h at 850°C. Note that tensile and short-term creep tests are available from the HTR-M project on those alloys at temperatures up to 850°C and for creep periods of up to 300h to provide a basis for the estimated stresses and effect of environment (Figs. 1 to 3).

The Blade materials were supplied by Doncasters and the specimens machined and heat treated (16 mm dia.X 150 mm length, FPI, X-rayed – heat treated in vacuum (1220°C/2h + 1240°C/2h + 1246°C/2h/80°C/min to RT+ 1080°C/4h/FGC + 900°C/8h/FGC) and subjected to dye penetrant and X-ray examination to ensure that no cracks or open porosities were present.

Initially, it was proposed to carry out mechanical tests (creep tests on notched sample) plus micro-structural investigations (crack lengths, orientations, location...) to study damage evolution in the blade alloys and identify parameters required for damage models. However, following discussions (held principally during a specialist meeting prior to the 3rd HTR-M1 & M1 progress meeting (Refs. 1 & 2)) it was agreed to abandon these since with this programme only two tests per environment for each grade (air and HTR helium containing impurities) would be possible. At this meeting it was also agreed, since the helium atmosphere in the reactor is largely unknown and the results may be sensitive to atmosphere changes, to abandon the "representative helium" condition and instead to test the specimens in four states as indicated below.

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

These would apply to both the HTR-M and HTR-M1 tests and would be carried out on both the turbine disc and blade materials.

Originally it was proposed to carry out the tests for conditions I and I(mod) at CEA in air, and conditions II and III at JRC in argon. After some discussion it was decided that all tests are to be carried out in air. Ageing would be carried out in an argon atmosphere, using oversized blanks.

The test matrix and proposals for the test conditions were as far as possible planned so that they bound the temperature and transient cases considered for the first row of the turbine but using a maximum temperature of 850°C.

The proposed test matrix for the HTR-M1 turbine material tests is given in Table 3. Damage analysis will be confined to the following activities:

- observation of fracture surface by SEM;
- identification of damage type (intergranular, transgranular) on longitudinal cuts;
- identification of damage parameters that would be suitable to fit the creep data using the usual creep damage models for super-alloys (i.e. MacLean and Dyson models).

The start of the medium term creep tests on the treated specimens was planned from early 2004 following the completion of the short-term HTR-M tests. However problems during the construction and commissioning of the corrosion rig at JRC (Fig. 4) resulted in significant

delays to the treating of the specimens for conditions (I) and (II) and only the tests in air at CEA could be started to programme. The testing of specimens in the de-carburised (II), carburised (III) conditions had to be put back until the corrosion rig was fully operational.

For the corrosion rig the first dummy tests on IN 617 have been performed and the carburisation analysed by FZJ. A carburising depth of 0.9 mm was given after 1000h at 950°C. Start of tests on the turbine specimens is from October 2004 and will continue to early 2005 with the HTR-M1 tests performed in parallel.

The conclusion from the short term HTR-M results in air (Ref. 3) was that CM 247 LC DS has the better high temperature properties (than IN 792 DS) and therefore may be the best candidate. This remains to be confirmed however by the follow-on tests on the treated specimens and the HTR-M1 results.

It was discovered in September 2003 that the IN 792 material was not properly heat treated by the manufacturer (a second solution step (1080°C/4h) and the final ageing (850°C/ 24h) was missing. At the time some IN 792 bars had already been machined at CEA and some others had started a 10000h thermal ageing at 850°C within the HTR-M1 Programme. It was therefore decided to restart the whole heat treatment on the aged bars and to simply add the missing heat treatment steps to the other bars.

A HTR-M1 Programme extension has been requested in order to fully report the results from the graphite irradiation tests (see section 2.2) and this also provided an opportunity to complete the creep tests on blade alloys started within HTR-M (long term tests + condition denoted as I_{mod} in the test matrix) in addition to the planned HTR-M1 tests (creep and tensile properties after long term ageing for IN 792). A revised schedule for this is given in Appendix A. The revised dates for the remaining deliverables from this work package are shown in Table 1. All treatments and mechanical testing will be completed within the extension period of the HTR-M1 project.

2.2 Work package 2 of HTR-M1 – Graphite Irradiation Tests

The specific tasks involved in this work package are as follows.

- Task 1 Graphite Irradiation tests
- Task 2 Graphite with SiC coatings
- Task 3 Graphite oxidation

(a) Graphite Irradiation tests

A key feature of existing and future high temperature reactors is the use of industrial graphite for the neutron reflector. It was recognised that graphite grades used in nuclear plants in earlier decades are no longer available and new grades and associated data plus a qualification programme has to be established for new nuclear graphites envisaged for future Very High Temperature Reactors (VHTRs). A major part of the HTR-M1 programme is this re-establishment of experimental facilities within Europe plus the completion of a first phase of irradiation tests (with PIE) on graphites recommended by manufacturers for the VHTR. This work is being done by NRG with the use of the High Flux Reactor at Petten. Several grades of graphite are being irradiated at a temperature of 750°C up to fluence equivalent to around 8dpa (11 to 15 HFR cycles) with the second phase of the work planned to continue in 2006 as part of the VHTR-IP up to the completion of full fluence level of around 25 dpa.

For the HTR-M1 programme three main graphite manufacturers have been approached to identify which of the available graphites can be offered for the next generations of reactors. Five graphites were selected and identified for the programme, two iso-moulded and two extruded graphites, manufactured from pitch coke and petroleum coke. A range of grain sizes (1mm down to 10 µm) has also been selected. IG100 (HTTR graphite) has 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. It is expected that the available data should provide a useful comparison with the data obtained from the new graphites.

The irradiation experiment uses a test rig (INNOGRAPH1) 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 and therefore adopted for the HTR-M1 tests. The work includes development of the loading rig (INNOGRAPH1), pre-characterisation of the test pieces, installation and removal of the loaded rig from HFR plus post irradiation examination (PIE) in shielded facilities. The rig design requires the stacking of around 150 to 200 samples, 8mm diameter (either 6 or 12mm in height), covering two main directions (15 samples per grade per direction).

Characterisation of the graphites has been performed at facilities at NRG covering dimensional change, CTE (coefficient of thermal expansion), dynamic Young's modulus and thermal diffusivity. For CTE a commercial dilatometer is to be used.

In addition to the five (major) grades of graphite 'piggy-back' samples have also been included in the irradiation capsule in order to obtain an early indication of the irradiation behaviour of some of the other proposed graphites. These (minor) graphites selected are UCAR grade PPEA, SGL grade NBG-20 and Toyo Tanso grade IG-430 (Table 4). A report on the specimen procurement and fabrication is given in Ref 4.

The irradiation test provides a number of data points within a single test run by using the flux (buckling) distribution available in the HFR capsule. A third order polynomial describes the behaviour and a sufficient number of points should be obtained from the two series of test measurements to describe the distribution.

Ideally the test conditions for the experiment are 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 is 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 framework therefore provides a first sorting and as the experiment progresses there is the possibility of 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.

The five main plus piggy-back graphites were supplied to NRG who performed the necessary pre-characterisation tests. X-ray tomography has also been performed at Manchester University. Figures 6 to 9 show details of the graphite irradiation specimens and the rig assembly.

The irradiation rig and specimens were inserted into the core of the HFR early in 2004 and the irradiation test started at the HFR reload in February 2004. PIE of the specimens is

currently planned for Autumn 2005 with final reporting for the end of December 2005. However soon after loading it was found that as result of small gaps and eccentricities in the assembled geometries there were variations in the temperatures of the specimens (see Fig 10). Since each of the specimen temperatures was individually monitored and maintained and in order not lose any tests, it was decided to adopt the resulting axial variation temperatures for the experiment. This meant that 75% of specimens (upper part) are being maintained at 750^{o}C and 25% of specimens (lower part) at 670^{o}C. It is considered that there are sufficient specimens at each temperature not to impair the statistical balance of the test results. As of November 2004 the experiment has reached its ninth cycle.

An extension of the HTR-M1 programme to the end of December 2005 was requested in order to report these results. The work could not be done in the original time frame because during the programme it was decided to include 200 instead of 150 specimens in the irradiation experiment. In addition the design of the irradiation experiment took more time because of the need to take account of the re-irradiation of the specimens to a higher dose in the 6th framework programme. Keeping the specimens as clean as possible and making the rig loading as easy as possible were aspects that needed extra attention.

The revised schedule for completion of the graphite irradiation work is shown in figure 11. It is planned to move the dates for the deliverables from the graphite irradiation work (D9 to D12) to the end of December 2005. The revised dates for the remaining deliverables from this work package are shown in Table 2.

(b) Graphite with SiC coating

Work carried out in Germany to develop SiC coatings for graphite materials for protecting the graphite against oxidation from air or steam ingress under severe accidents has been reported (Ref 5). Investigations were done using the matrix German graphite A3-3 to measure corrosion rate for temperatures up to 1200^{o}C, and on coated pebbles using different graphites. Most of the coating experiments used the Japanese graphite IG 110 adopted for the structures and fuel blocks of the HTTR. Corrosion tests, irradiation tests plus PIE were carried out to assess coating integrity and different coating methods (Chemical vapour deposition (CVD), Paste Siliconisation (PS)). A very good corrosion resistant quality was eventually achieved with IG 110 and V438 using CVD. No damage or weight loss was observed after being irradiated and corroded and dropped from a height of 50 cm. Although the original objective, to develop a coating for A3-3 graphite, had not yet been achieved, the results obtained so far are a very positive indicator and have attracted considerable interest Internationally (from USNRC).

(c) Graphite Oxidation

In addition to the oxidation tests performed in the HTR-M Programme, FZJ carried out further investigations on oxidation characteristics of the new graphites under investigation in the HTR-M1 graphite irradiation tests. This work was not originally scheduled and has been provided by FZJ within their original resource allocation.

The investigations cover oxidation measurements (oxygen/air) of eight chosen structural graphites at temperatures of 650 and 700^{o}C performed in the THERA facility. Measurements were carried out up to a final weight loss of 70-100%. Most of the materials were shown to have a comparable reactivity. Most of the new graphites were similar or better than V4837, an established nuclear graphite from a former HTR programme. The exception to this was NBG 20 (see Fig. 5). Note that because of the apparent difference in rates (at least a factor

of 3 higher) a number of samples of NBG 20 were tested but without any obvious differences in behaviour (Ref. 6).

The graphite NBG 20 was considered unsuitable for application to the HTR. A number of possible causes were considered (graphitisation, purification, ash content) but to date no obvious reasons for the differences have been found. It is planned to continue the explorations of NBG 20 within the 6th Framework as part of the graphite investigations within the VHTR-IP.

3 Discussion

3.1 Overall Progress

The project is progressing reasonably well however an extension request has been necessary to enable the work on graphite irradiation to be fully completed and reported within the HTR-M1 project timescale.

Turbine

There have been some delays in the testing of the corrosion treated turbine materials within HTR-M which has delayed the start of the HTR-M1 intermediate creep tests. Tests in air are underway or concluded. The carburisation and decarburisation treatments are now underway and the corresponding HTR-M1 creep experiments will commence as soon as practical. The intermediate creep tests are expected to continue to the end of the extended HTR-M1 programme.

Graphite

The graphite selection and characterisation has concluded as planned and specimens introduced into INNOGRAPH1 and loaded into the core of the HFR. The completion of the graphite irradiation is expected to be in April 2004 and following a period of cooling the PIE will start with reporting to be completed by the end of the extended HTR-M1 programme.

3.2 Overall Scientic value

The technical tasks addressed by the project are on key components and deal with issues that are at the forefront of the technology with respect to materials behaviour and HTR application. It is expected that in these two areas, the findings from this work will have an important impact on the final materials selection and development for future HTR and VHTR modular designs.

Turbine

The turbine review and test work of HTR-M on the two chosen Turbine blade materials confirmed the consistency of the materials with available published information in air and identified CM 247 LC DS has having the better higher temperature properties. Impurities in the helium at temperature can result in carburisation and/or de-carburisation of the super-alloys and lead to a worstening of material strength. Two blade materials (Al_20_3 former and Cr0 former) were chosen for the tests as the precise atmosphere was not known. The short and medium term test work following exposure to treated atmospheres should provide an overall indication of the relative bounds on strength such carburising and decarburising effects can have. If shown to be significant there may be important implications with respect to environmental control and acceptable levels of impurity levels in the reactor helium gas.

Graphite

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. 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. The result is that for the new HTRs and VHTRs a new graphite is required that will need significant development (irradiation tests) and require global sharing of information through international cooperation.

The HTR-M1 test programme has been initiated on candidate graphites in the High Flux Reactor at Petten. The materials tested have been chosen in full co-operation with graphite manufacturers and are therefore considered the best potential materials available. This irradiation looks at physical property changes at one temperature (750^{o}C) and requires continuation in the next framework programme to complete the information up to 'turn-round'. More information is needed (at higher temperature) also model development initiatives towards developing a predictive methodology. The first stage PIE towards the end of 2005 will enable some initial sorting of a possible candidate to be done, however the major impact will come at the end of the 6th framework when the full curve behaviour can be described.

4 Conclusions

The main emphasis for the third year of the HTR-M1 project has been to progress the experimental work on the turbine materials and graphite irradiation tests. A key objective of the HTR-M1 project is to improve knowledge of materials for future HTRs and VHTRs. The HTR-M1 project deals with the selection and development of materials for high temperature components of the turbine (direct cycle) and the graphite core with specific experiments on creep and irradiation. The Scientific and technical progress for the HTR-M1 project has been described and issues that are likely to influence the technical decisions on the next stage of the programme reviewed. Testing is in progress with the main test on the treated turbine materials and graphite irradiations set to continue to the end of the extended HTR-M1 programme.

For the turbine (blades) following discussions with Turbine materials manufacturers, candidate alloys have been selected for the test work. Material has been procured and machined and testing completed in air plus a number of specimens are being treated to carburising and decarburising atmospheres to assess the impact of the environment. This pre-treatment of the specimens is underway and medium term tests in air are completed or set to continue in 2005.

Three principle graphite manufacturers and suppliers have collaborated closely with the HTR-M1 project and supplied new graphites for irradiation testing in HFR. Following selection, procurement, machining and charcterisation the samples have been inserted in the test rig (INNOGRAPH1) and placed in the core of HFR. The test started in February 2004 and is not expected to conclude until April 2005 after which a period of cooling and P.I.E will be required. Oxidation tests on the HTR-M1 candidate graphites have been carried out by FZJ. One of the minor graphites was shown to be unsuitable for HTR application. Work is continuing to understand this more fully. The work on review of coatings for graphite

materials for protection against oxidation has shown SiC to give good results for Chemical vapour deposition (CVD) with IG 110 and V438.

An extension of the HTR-M1 Programme has been requested to enable the work being done on graphite irradiation to be fully completed and reported before the end of the project. The work could not be done in the original time frame because of a decision to include more specimens in the experiment, and because of the extra care needed in preparation/pre-characterisation to make sure the rig and specimens were capable of being reloaded for the higher dose in the 6th framework programme.

The irradiated samples are due to be removed from the core of HFR in April 2005 and following a period of cooling will undergo post irradiation examination (PIE). The results of that examination will be reported in December 2005. This is the first phase of the 750^{o}C graphite irradiation work (up to 8dpa), which is planned to continue in 2006 within the VHTR-IP up to full fluence levels of around 25 dpa. Note that a further irradiation will also take place within the VHTR-IP at a much higher temperature (~950^{o}C) and this will be the focus of the early part of the VHTR-IP work. The late finish of the current irradiation has already been taken into account in the planning of the VHTR-IP programme.

5 References

Ref	Title
1	D. E Buckthorpe Minutes of the HTR-M / M1 Progress Meeting on 15-17 April 2002, Julich, Germany Report No. HTR-M1-03/12-M-0-0-20
2	H. Rantata Notes of a HTR-M & M1 Specialist Meeting on Corrosion & Carburisation Report No. HTR-M 03/07 M 2 0 72
3	R. Couturier, J. Calapez, N Scheer, H. Rantala Materials for Turbine: Final Report on results from HTR-M Turbine material investigations Report No. HTR-M 04/10 D 2 3 103
4	Vreeling HTR-M1 Report on specimen fabrication and procurement for the graphite irradiation tests Report No. HTR-M1 03/04 D 2 2 13
5	B. Schröder Graphite with Si C coatings Report No. HTR-M1 03/09 D 2 5 14
6	K. Kuhn Comparison of the oxidation behaviour of 8 HTR-Graphites Report No. HTR-M1 04/12 D 2 0 27

Table 1. Revised HTR-M1 Deliverables list (WP1)

Deliverable No	Deliverable title	Delivery date	Current planned	Date Achieved	Main Organisation
D1	Report on selection of materials & testing requirements	12	01/10/2002	11	CEA
D2	Report on specimen fabrication and procurement & testing requirements	18	01/04/2003	17	CEA
D3	Intermediate report on material testing	47	31/07/2005	-	CEA
D4	Final report on blade alloy properties	51	31/12/2005		CEA
D5	Review of results and updating of data base	51	31/012/2005	Suppressed not needed	CEA
D6	Final Report summary	51	31/12/2005	-	FRA

Table 1. (cont) Revised HTR-M1 Deliverables list (WP2)

Deliverable No	Deliverable title	Delivery date	Current planned	Date Achieved	Main Organisation
D7	Report on selection of graphites and testing requirements	12	01/10/2002	14	NNC
D8	Report on specimen fabrication and procurement	18	01/04/2003	18	NRG
D9	Report on irradiation and post irradiation tests	51	31/12/2005	-	NRG
D10	Review of results and updating of data base	51	31/12/2005	-	NRG
D11	Assessment of results and suitability	51	31/12/2005	-	FZJ
D12	Final Report Summary	51	31/12/2005	-	NNC

Table 2			HTR-M1 Project document list	
HTR No.	**Status**	**Final issue date**	**Title**	**Author / Org**
HTR-M1 02/1 M 2.0.1	Final	April 2002	Minutes of 1st Technical meeting on graphite held on 13th Nov. 2001 at NRG Petten, Netherlands	J. van der Laan/ NRG Petten
HTR-M1 02/1 M.0.0.2	Final	April 2002	Minutes of the HTR-M1 Kick-off plus 2nd HTR-M Progress Meeting on 5-7 December 2001, Grenoble	D.E. Buckthorpe/ NNC Ltd.
HTR-M1 02/5 M 2.0.3	Final	July 2002	Minutes of meeting with graphite manufacturer UCAR on 26th. March 2002, Notre Dame, France	M. W. Davies/ NNC Ltd
HTR-M1 02/5 M 2.0.4	Final	July 2002	Minutes of meeting with graphite manufacturer SGL on 27th March 2002, Chedde, France	M. W. Davies/ NNC Ltd
HTR-M1 02/5 M 0.0.5	Final	Nov 2002	Minutes of the HTR-M / M1 Progress Meeting on 15-17 April 2002, Julich, Germany	D.E. Buckthorpe/ NNC Ltd.
HTR-M1 02/5 P 0.0.6	Final	July 2002	18month HTR-M plus 6 month HTR-M1 joint management report	D. E. Buckthorpe NNC Ltd.
HTR-M1 02/9 D 1 1 7	Draft		HTR-M1: Report on selection of materials & testing requirements for the Turbine	R. Couturier / CEA
HTR-M1 02/11 P 0 0 8	Final		Mid-term report for the HTR-M & M1 Projects	D. E. Buckthorpe NNC Ltd
HTR-M1 02/11 D 2 1 9	Draft		Report on selection of graphites and testing requirements	M. W. Davies/ NNC Ltd
HTR-M1 02/12 M 0 0 10	Final		Minutes of the HTR-M plus M1 Mid Term Meeting on 28-29 November; 2002, Framatome, Paris	D. E. Buckthorpe NNC Ltd
HTR-M1 02/12 P 0 0 11	Final		24 Month HTR-M plus 12 month HTR-M1 Management Report	D. E. Buckthorpe NNC Ltd
HTR-M1 03/03 D 1 0 12	Draft		Report on specimen fabrication, procurement & testing requirements for the turbine blade material	R. Couturier / CEA
HTR-M1 03/04 D 2 2 13	Draft		HTR-M1 Report on specimen fabrication and procurement for the graphite irradiation tests	A. Vreeling / NRG
HTR-M1 03/09 D 2 5 14	Draft		Graphite with Si C coatings	B. Schröder/ FZJ
HTR-M1 03/06 P 0 0 15	Final	July 2003	30 Month HTR-M plus 18 month HTR-M1 Management Report	D.E.Buckthorpe/NNC
HTR-M1 03/07 M 0 0 16	Draft		Minutes of 2nd Technical Meeting at Petten on HTR-M & M1 Irradiation experiments	D.E.Buckthorpe/NNC
HTR-M1 03/07 M 0 0 17	Final	Nov. 2003	Minutes of the 5th HTR-M plus M1 Progress Meeting	D. Buckthorpe, R. Bestwick, A. A. Braithwaite / NNC

Table 2 (cont)			HTR-M1 Project document list	
HTR No.	**Status**	**Final issue date**	**Title**	**Author / Org**
HTR-M1 03/11 P 0 0 18	Draft		36 Month HTR-M plus 24 month HTR-M1 Management Report	D. E. Buckthorpe/ NNC
HTR-M1 03/11 P 0 0 19	Draft		Third Annual Technical Report for the HTR-M project	D.E. Buckthorpe / NNC, et al.
HTR-M1 03/12 M 0 0 20	Final	Inserted October 2004	Minutes of the 6th HTR-M plus M1 Progress Meeting at FZJ	D. E. Buckthorpe/ NNC
HTR-M1 04/05 P 0 0 21	Final	October 2004	42 month HTR-M plus 30 month HTR-M1 Management Report	D.Buckthorpe/ NNC
HTR-M1 04/05 M 2 0 22	Draft		Meeting on Nuclear Graphite Selection – 16th February 2004	A Vreeling, J van der Laan/ NRG
HTR-M1 04/07 M 0 0 23	Final	Nov. 2004	Minutes of the 7th HTR-M plus M1 Progress Meeting at EA, Madrid	D. Buckthorpe/ NNC
HTR-M1 04/11 M 0 0 24	Draft		Minutes of the 8th HTR-M plus M1 Progress Meeting at Framatome, Lyon	D.Buckthorpe/ NNC
HTR-M1 04/10 P 0 0 25	Draft		48 Month HTR-M plus 36 month HTR-M1 Management Report	D. Buckthorpe/ NNC
HTR-M1 04/10 P 0 0 26	Draft		3rd Scientific Report on HTR-M1 Project	D. Buckthorpe/ NNC
HTR-M1 04/12 D 2 0 27	Draft		Comparison of the oxidation behaviour of 8 HTR-Graphites	K Kuhn/ FZJ
HTR-M1 05/04 M 2 0 28	Draft		Notes of a technical meeting on nuclear graphite selection held at NRG Petten	A. Vreeling & J. van der Laan/ NRG

Table 3 HTR-M1 Turbine Material Test Matrix

CM 247 at 850°C.

Test / n° of specimens	T (°C)	I	I_{mod}	II	III
Tensile	20 750 800 850	1✓ 1✓ 1✓ 1✓	1 1 1 1		
Creep x MPa (3000h) y MPa (1000h) z MPa (5-10 kHr)	850	2✓ 2✓ 2✓(running)	2* 2* 2	2 2 2	2 2 2

IN 792 DS aged 10 000 hours at 850°C.

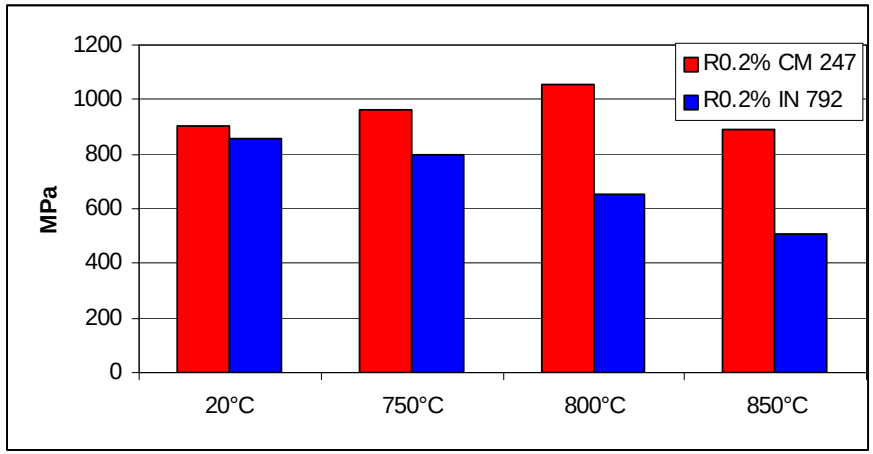
Test / n° of specimens	T (°C)	I	I_{mod}	II	III
Tensile	20 800 850	1 1 1			
Creep x MPa (3000h) y MPa (1000h)	850	3		4	4

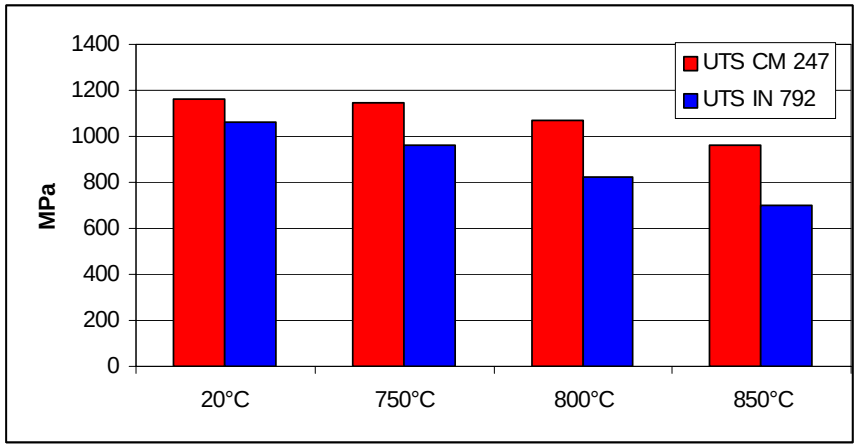
Table 4 HTR-M1 Graphite Irradiation Test Matrix

	750°C			~670°C		
	WG	AG	Total	WG	AG	Total
NBG-10	12	14	26	3	3	**6**
NBG-25	11	12	23	4	3	**7**
PCEA	11	12	23	4	3	**7**
PCIB	11	12	23	4	3	**7**
IG-110	11	12	23	4	3	**7**
NGB-20	5	5	14	2	2	**4**
PPEA	5	5	14	2	1	**4**
IG-430	6	6	18	3	3	**6**

Figure 1 Results of Tensile tests in Air on Turbine Blade Materials

Tensile tests carried out at $5.10^{-4}\ s^{-1}$ (20 to 850°C)





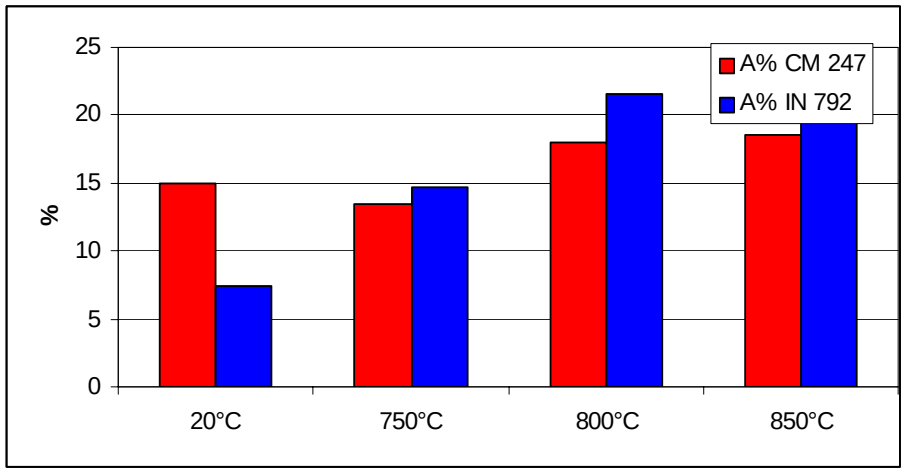
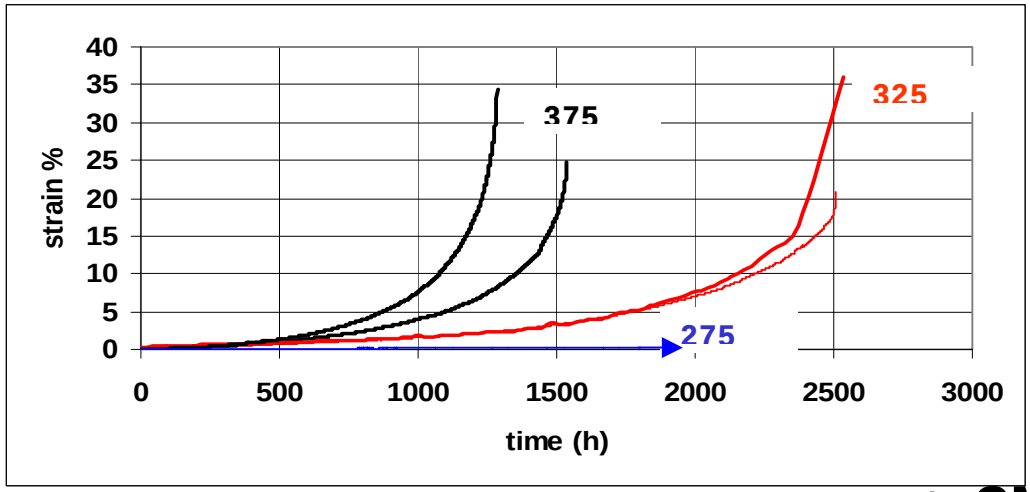


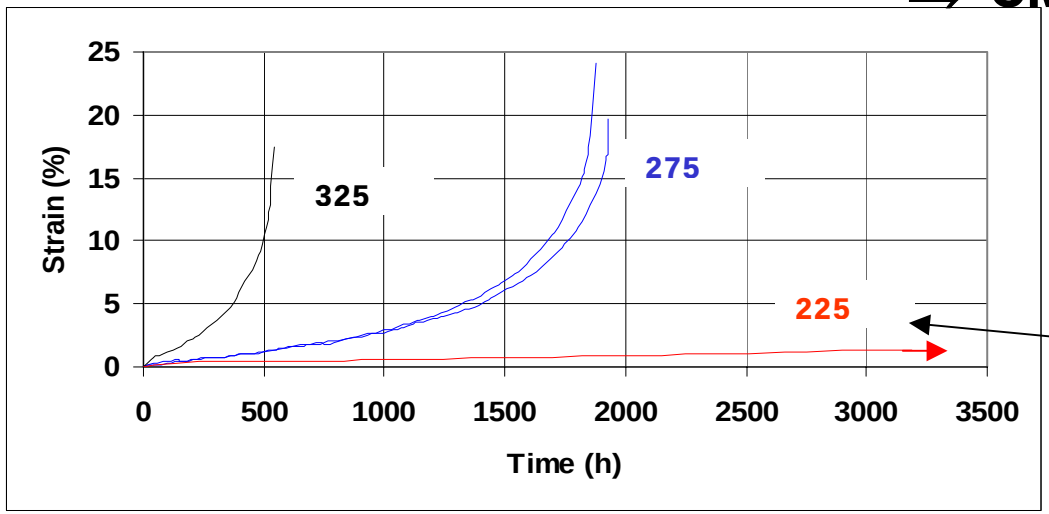
Figure 2 Results of Creep Tests in Air on Turbine Blade Materials (%Strain vs Time)

Target: 1000h; 3000h and 9000h to failure



CM 247

⇒ CM 247 > IN 792 792

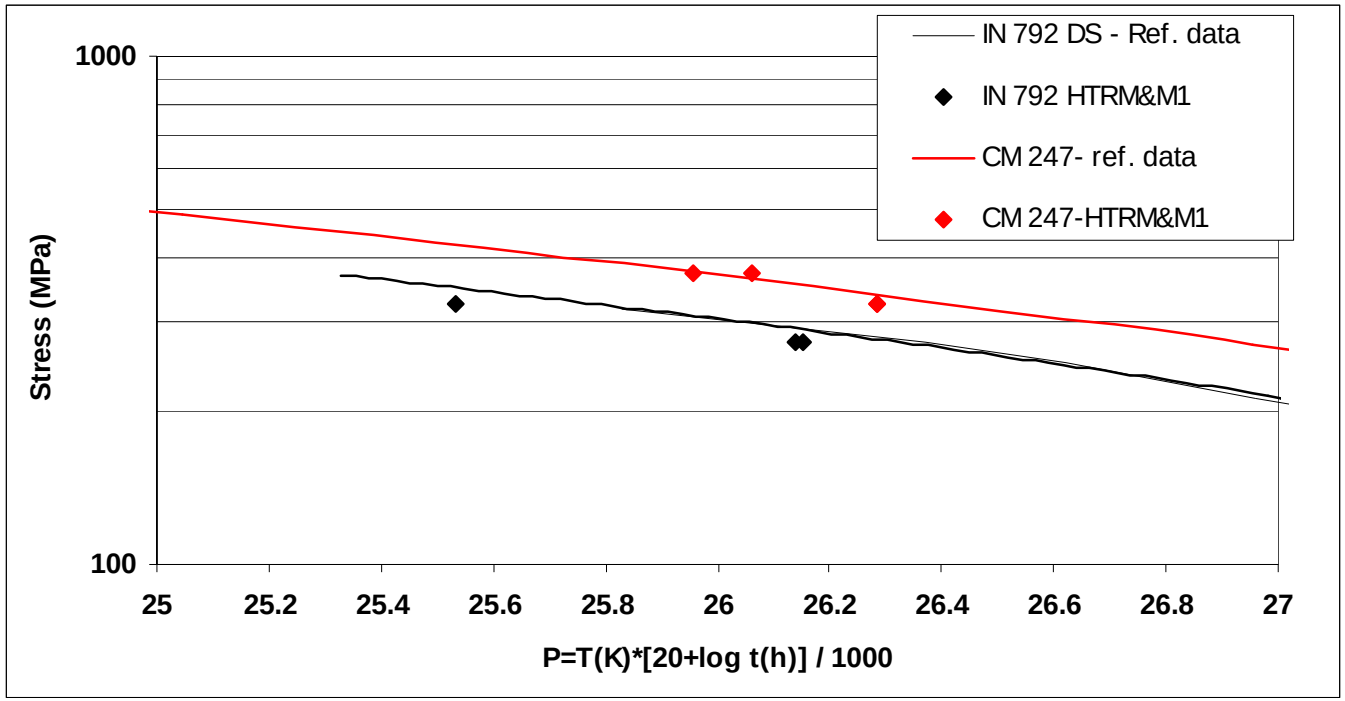


IN 792

Applied stress level (Mpa)

Figure 3 Results of Creep Tests in Air on Turbine Blade Materials (Larson-Miller Plot)

Larson Miller plot of results (running tests not included)



Results consistent with biblo-graphic Data

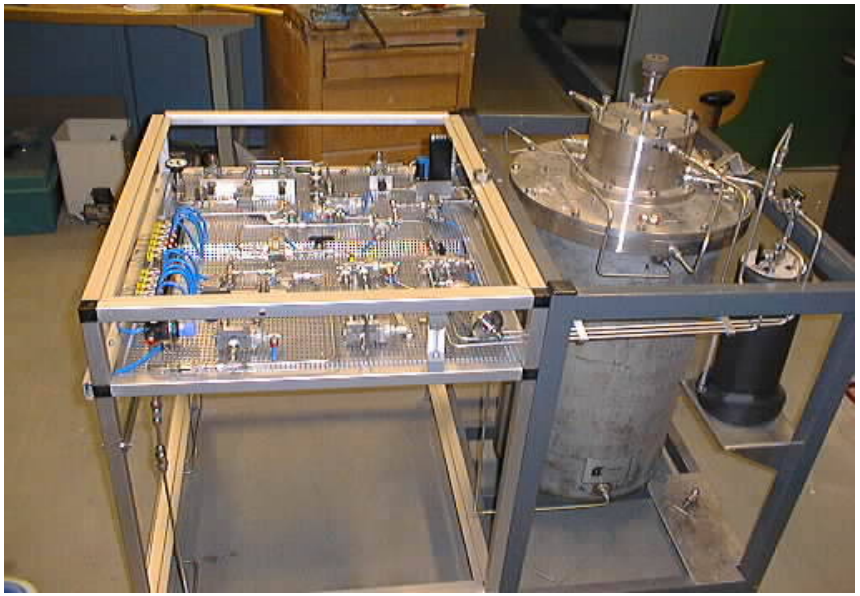
Figure 4 Details of Carburising Rig for High Temperature Alloy Tests

Turbine materials – Results

Materials Testing

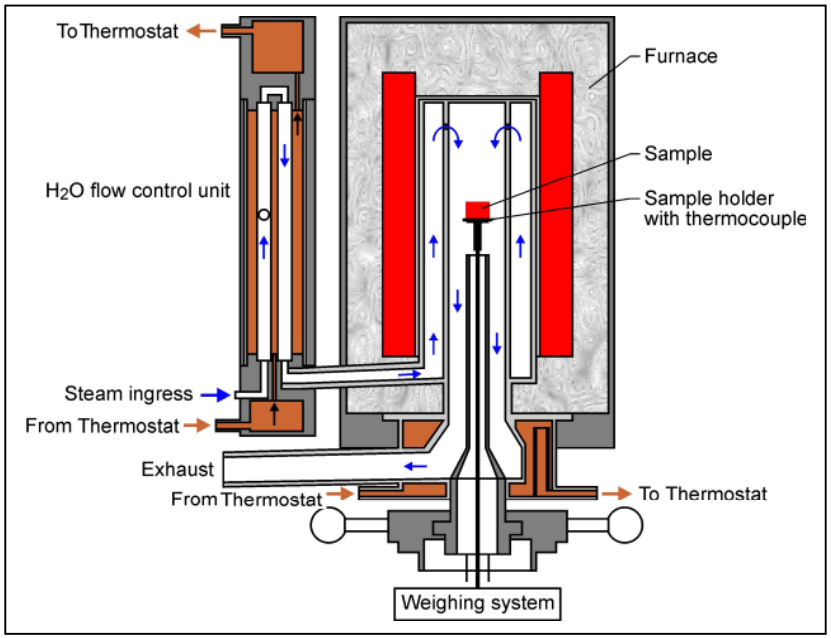
Materials treated at JRC (carburization, decarburization) to complete the experimental work.

As received (with aging HT)→ **I (CEA)**
De-carburised → **II (JRC)**
Carburised → **III (JRC)**
After Decarb or Carb HT → **I_{mod} (CEA)**



Gas system & Rig at JRC

Figure 5 Results of HTR-M1 Graphite Oxidation tests



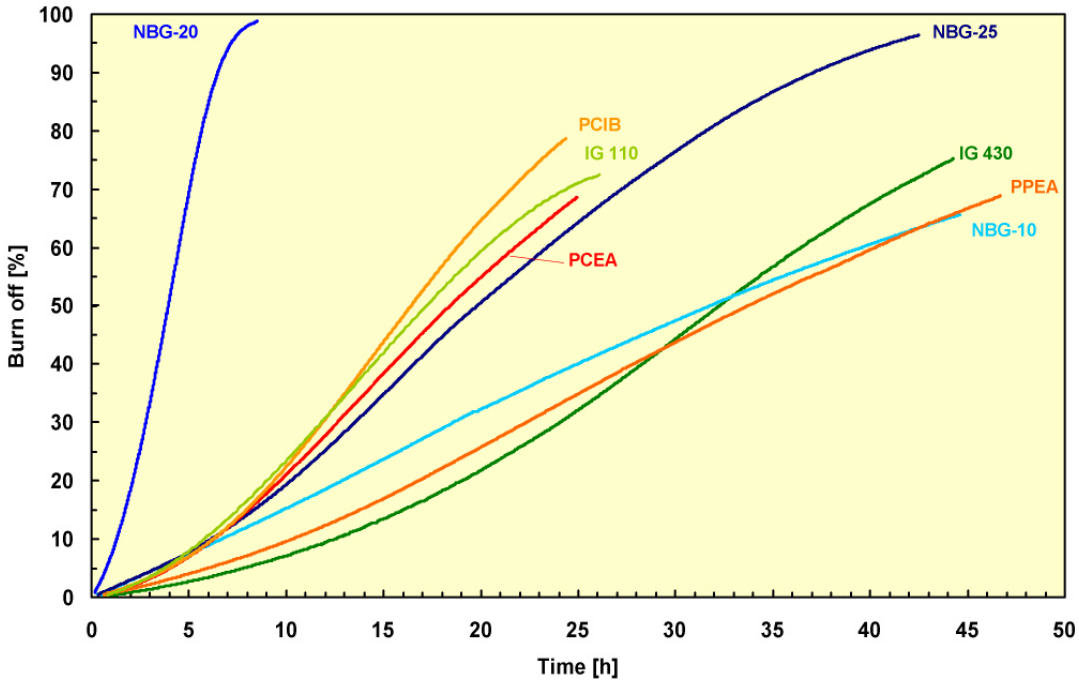
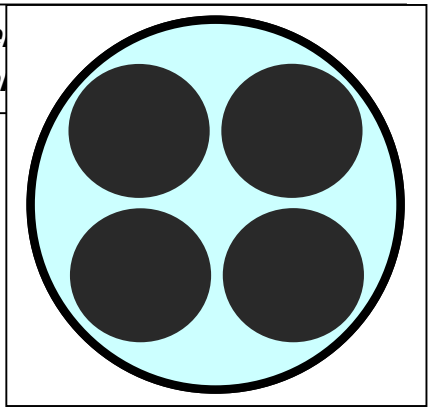
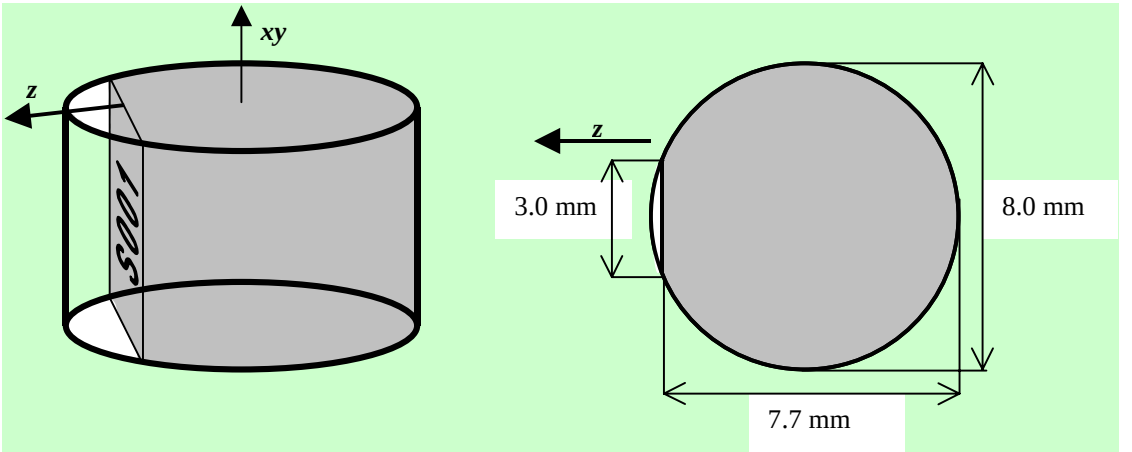


Figure 6 Details of graphite specimens

SGL	PITCH COKE	PET COKE
EXTRUDED	**NBG-10**	*NBG-20*
ISO-MOULDED		**NBG-25**
GRAFTECH	PITCH COKE	PET COKE
EXTRUDED	*PPEA*	**PCEA**
ISO-MOULDED		**PCIB-SFG**
TOYO TANSO	PITCH COKE	PET COKE
EXTRUDED		
ISO-MOULDED	*IG-430*	**IG-110**

Layout in the Irradiation Rig Drum





Specimen Design

Cylinders Dia 8 mm and H = 6 or 12 mm

Flattened side for specimen ID

Figure 7 Details of irradiation in HFR

HFR

•45 MW Pool type reactor
•Core lattice is 9 x 9 array containing 33 fuel assemblies, 6 control rods, 19 experimental positions and 23 Be reflector elements •Active height 600 mm
•More than 11 cycles (25 FPD) per year: typical total 280 Full Power Days

In-core positions C7/E7 or equivalent
–Fluence rate $Ø_{tot}$ = 6-9 E18 $m^{-2}s^{-1}$–EDN fluence ~ 6-7 E25 m-2, or ~ 8 dpa
–Required 11 to 15 cycles take 1-1.5 years
–Ratio min/max ~ 65%
Irradiation temperature 750°C
Helium environment
–6.0 quality helium, slow purge
–moisture monitoring
–CO addition possible
Rig design based on D85 series (German HTR-Modul progamme up to 1994), and actual SiCSiC irradiation (2002)

Overview HFR loading geometry

	A	B	C	D	E	F	G	H	I
1	Be	Be	Be	Be	Be	Be	Be	Be	Be
2	F	F	F	D2	F	F2	F	H2	Be
3	F	F	C3	F	E3	F	G3	F	Be
4	F	CR	F	CR	F	CR	F	H4	Be
5	F	F	C5	F	E5	F	G5	F	Be
6	F	CR	F	CR	F	CR	F	H6	Be
7	F	F	C7	F	E7	F	G7	F	Be
8	F	F	F	D8	F	F8	F	H8	Be
9	Be	Be	Be	Be	Be	Be	Be	Be	Be

PSF
WEST

F Fuel element
CR Control rod
Be Reflectorelement
E5 Experiment



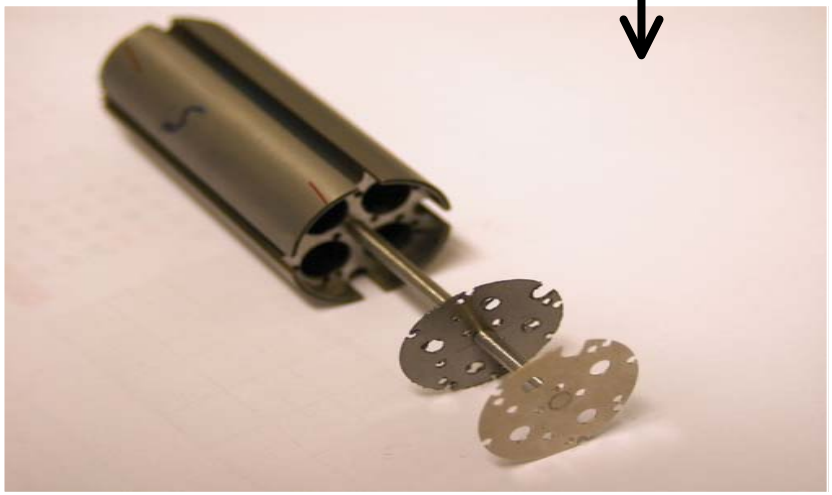
Figure 8 Details of Graphite Specimens

Graphite Irradiation in HFR

•4 stacks of 60 mm stacking height per drum

• Graphite specimens are separated from TZM-alloy by thin (80 µm) graphite foil.





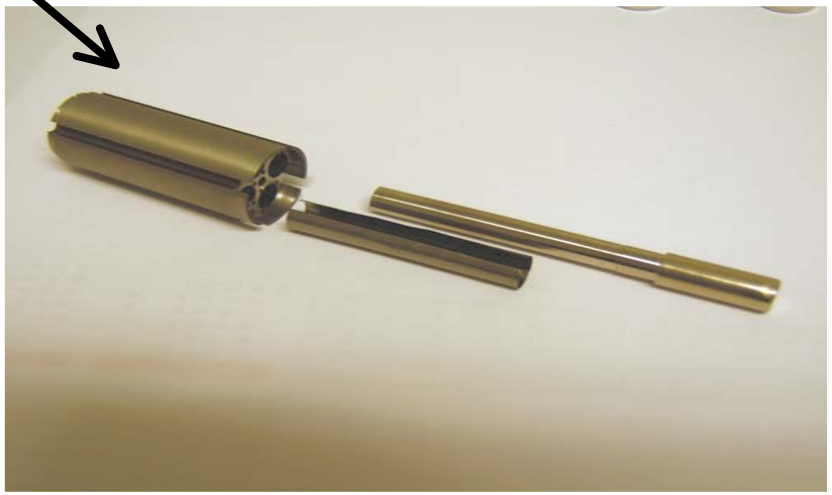
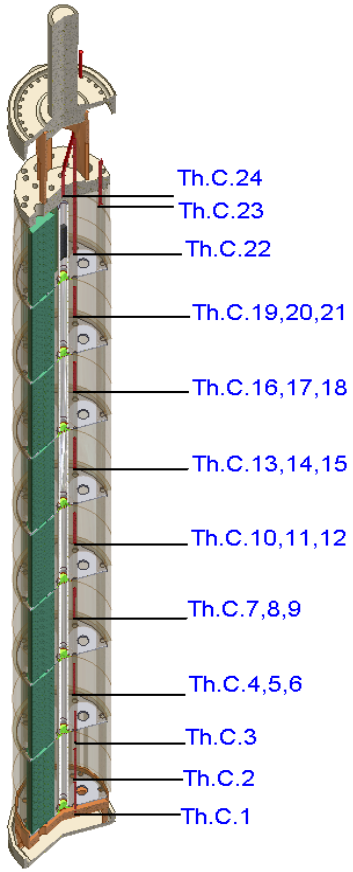


Figure 9 Details of Graphite Irradiation in HFR(INNOGRAPH1)

Graphite Irradiation in HFR (INNOGRAPH)

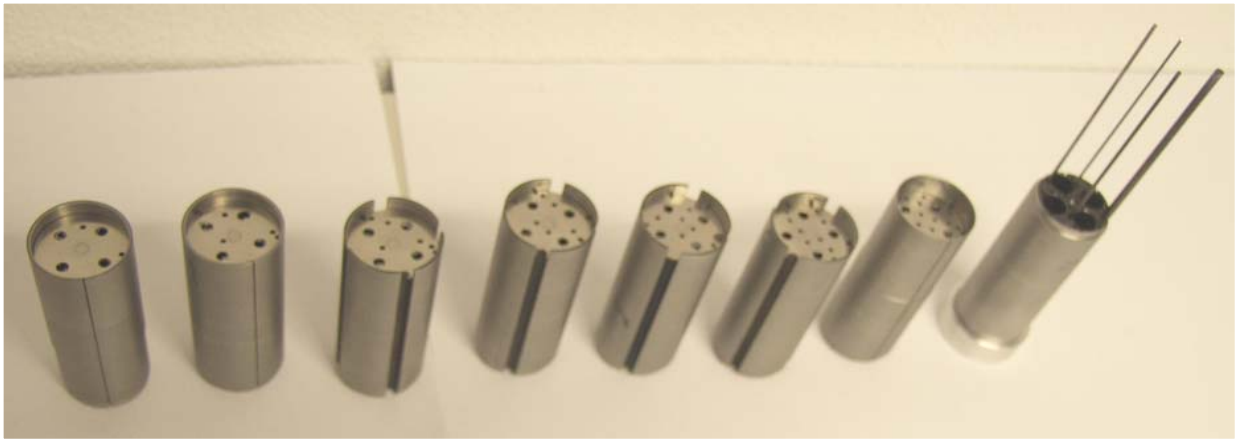


Drum-based configuration

- Heating by gamma heating
-Temperature control by He/Ne gas mixture in 2nd containment

Scientific instrumentation:

- *24 Thermocouples.*
- *9 neutron fluence detector sets*

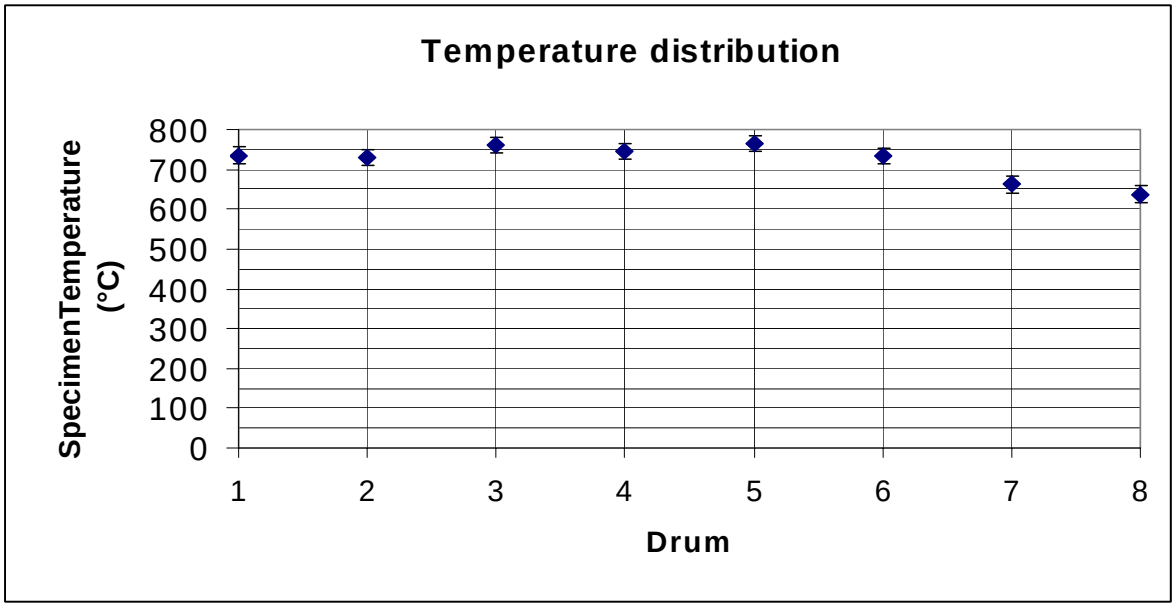


Commercial-in-Confidence

Figure 10 Progress of Graphite Irradiation in HFR

Irradiation started: 12 Feb 2004

Temperature drop



CAUSES

-Drums are slightly eccentric
- Rig is oblique (top part)
- Strips to center drum act as heat sink

CONSEQUENCES

specimen temperatures are monitored
No data will be lost: results at different temperatures will be available.

~75% irradiated at ~750°C
~25% irradiated at ~670°C

Commercial-in-Confidence

Figure 11 Description of Overall HTR-M1 Work plan

			2001				2002				2003				2004				2005			
WP 1	**Turbine materials**																					
	Task	Sub-task																				
	1	Review materials & testing needs																				
	2	Material procurement & machining																				
	3	Intermediate creep tests & testing after ageing																				
	4	Analysis & asessemnt																				
	5	Synthesis & updating of data base																				
WP 2	**Graphite**		2001				2002				2003				2004				2005			
	Task	Sub-task																				
	1	Review graphites & testing needs																				
	2	Material procurement & test piece manufacture																				
	3	Irradiation of specimens & post irradiation tests																				
	4	Assessment of results & suitability																				
	5	Irradiation with corrosion resistant coatings																				
	6	Synthesis of results & updating of data base																				

Appendix A: Updated programme for HTRM-1 extension,

Activities on turbine blade materials.

CEA work is focused on tensile and creep evaluation of two turbine blade alloys: CM 247 and IN 792. Some work was already done on these grades within HTRM project.

An extension of HTRM1 project up to the end of 2005 would allow:

- 1) to finish creep tests on blade alloys started within HTR-M (long term tests + condition denoted as I_{mod} in the test matrix);
- 2) to finish the work initially planned within HTR-M1 (creep and tensile properties after long term ageing for IN 792).

***The detailed technical work will include*:**

1) HTRM-1 test matrix on IN 792 DS aged **5000 h** at 850°C: 4 tensile tests (20-750-800-850°C) + 2 creep tests (1000h).

2) HTRM-1 test matrix on IN 792 DS aged **8000 h** at 850°C (ageing ends in 02/05): 4 tensile tests (20-750-800-850°C) + 2 creep tests (1000h).

3) From HTR-M programme: 3 long-term creep tests on IN 792 and CM 247 (9000h) are still in progress. These tests will be stopped and analysed, and the data will be entered in the database.

4) From HTR-M program for condition I_{mod} (corresponding to the initial heat treatment + the 1000h/950°C thermal cycle associated with carburisation performed at JRC):

- 4 tensile tests (20-750-800-850°C) on both CM 247 and IN 792
- 2 creep tests at 850°C on both CM 247 and IN 792 (1000-3000h)

Deliverables :

As the test will continue up to the end of 2005, we suggest modifying the CEA deliverables as follows:

- D3 : intermediate report on mechanical testing : 07/05;
- D4 : Final report on blade alloy properties (+ comments on the update of the JRC database) : 12/05;
- D5 : suppressed (the data are sent to JRC on a CD-Rom and no specific report is needed for this part of the work).

Revised Programme on Graphite Irradiation Experiments

	April	May	June	July	August	September	October	November	December
End of graphite Irradiation Experiment									
Cooling and dismantling									
Post irradiation testing (Screening): • **Mass** • **Dimensions** • **Thermal Diffusivity** • **Thermal Expansion** •									
Reporting									