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SP4 Summary Report on Materials Development

Author(s): **D . Buckthorpe (NNC)**

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RAPHAEL *(ReActor for Process heat, Hydrogen And ELectricity generation)*

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SP4 Summary Report on Materials Development

Abstract

The development of the Very High Temperature Reactor (VHTR) requires an understanding of the material behaviour under reactor operating conditions. Specific work on the development of materials has been made within SP4 of RAPHAEL. These developments address new materials and materials information for the Reactor Pressure Vessel, the Reactor Core and for the high temperature components including those within the heat transport circuit. The materials investigated are ferritic and nickel based alloys, superalloys, graphite and carbon composites. The non-metallic work on graphite and composites directly involve manufacturers and include an extensive irradiation program at high temperatures on new graphites, examining their potential for block type and pebble bed type designs. It is concluded that significant advances have been made in all areas with the vessel and graphite developments providing the current state of the art and leading other comparable activities within GIF.

The results of this work are to be provided as Euratom contributions to the Generation 4 International Forum (GIF) in the area of materials development.

Revisions

Rev.	Date	Short description	Author	WP Leader	SP Leader	Coordinator
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01	14/04/2010	Final issue	Derek. Buckthorpe NNC	Derek. Buckthorpe NNC	Derek Buckthorpe NNC	

Contents

Abstract

Within RAPHAEL-IP, the materials issues are addressed within Sub Project 4 (SP4) and they focus on the outstanding issues for the reactor core, the vessel and the heat carrying/ generating components. The materials investigated are medium and high temperature metals and alloys, carbon composite materials and graphite. The graphite and composite work which is for the core area and has been carried out in conjunction with materials suppliers (who are partners within the project).

Materials for the Vessel

Work on the advancement of the Reactor Pressure Vessel (RPV) material has focused on the development and use of a more temperature resistant material (Mod 9Cr 1Mo steel) compared with the established vessel steel used for the LWR. The application of Mod 9Cr 1Mo steel simplifies the design and avoids the need for an additional cooling circuit and protection from the inlet gas during operation. Application of the LWR steel (16MND5) for the HTR vessel also relies heavily on LWR past experience and developments and requires temperatures to be held at LWR conditions throughout operation.

The materials work within RAPHAEL and former projects currently provide the state of the Art on this Mod 9 Cr 1Mo steel vessel development. The investigations established the first fabrications, investigated the effects of irradiation and material data (fracture toughness, tensile and creep) at vessel temperatures and the effects of ageing and reduced scale component tests have also been performed. The main safety and structural integrity concerns of the RPV 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 aging and corrosion) the main environmental considerations. Key issues for the RPV are manufacture and welds and resistance to irradiation.

The effects of irradiation and environment on representative RPV Mod 9Cr 1Mo steel was investigated in the Fifth Framework Programme HTR-M1 for a thick section weld (150mm) fabricated using a narrow gap TIG process with a double V weld preparation and post weld heat treatment. At first micro-cracks were observed in the weld metal, seen when performing a micrographic examination. These heat cracks were later eliminated through adjustments to the welding procedure. This fabrication work provided a significant step forward in using this material for the HTR vessel and showed the importance of optimizing the filler material composition and welding procedures to avoid cracking and to maximize strength. Specimens were cut from the weld and tested in the main stress directions with tests carried out for the reference condition and End of Life (EOL) conditions. The mechanical tests included impact, tensile, creep and facture toughness (CT) tests. Four conditions were selected: plate material in the parallel and perpendicular directions (with respect to rolling), the heat affected zone (HAZ), and the weld metal (WM). The specimens were loaded into the LYRA test facility in the High Flux Reactor (HFR) at Petten and the irradiation, which was started at the beginning

of 2004 and took place in the poolside of HFR and was carried out at a single irradiation temperature of 375oC and an average fluence of 9.44·1022 n/m2, E>0.1 MeV (~5.8 mdpa). Comparisons were made with other available non-irradiated data and also for creep with non-factored values from the RCC-MR Code. The results showed the weld metal to have the highest ductility and the cross weld specimens the lowest. The only systematic influence of the irradiation was seen in the cross-weld tests where testing times were longer and minimum strain rates lower. The work confirmed the effects of irradiation to be small for the range of HAZ, WM and cross weld tests covered and also that the RCC-MR weld factor Jr = 0.9 for Mod 9Cr 1 Mo steel weldments was applicable to these tests. The impact tests on irradiated and non-irradiated sub-sized specimens also showed that the lowest ductile brittle transition temperatures (DBTT) occurred in the HAZ specimens and in the weld material and the weld metal showed the highest upper-shelf energy (USE). Creep tests were carried out on irradiated and non-irradiated specimens at 550°C for periods of up to 10000h and changes due to the irradiation were within the batch to batch variations of the material.

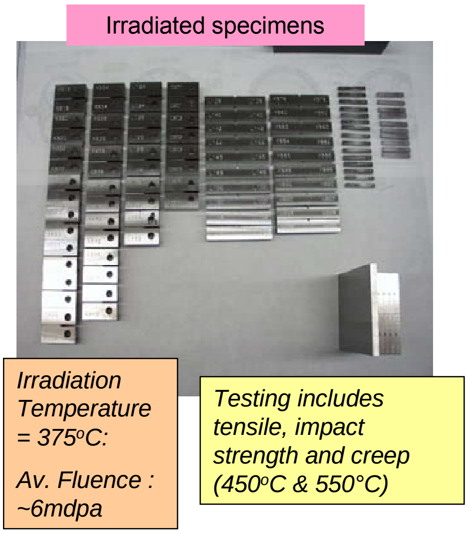


Figure 1:Specimens irradiated in HFR

Within RAPHAEL work continued on 16 pre-irradiated creep specimens that remained from the HTR-M1 irradiation tests (see figure 1). It was decided to focus on temperatures much nearer the negligible creep limit of the material. Again tests were performed on non-irradiated material to provide a comparison of the effects of the irradiation. The irradiated specimens (see Figure 1) were tests at 450°C for creep periods up to around 10,000hrs. The results showed that the changes due to the irradiation were again within the batch to batch variations of the material. The results from the base metals tests and the weld tests also showed the base-metal results to be above the RCC-MR minimum curve with a different slope for both reference and irradiated material. It was also evident that the weld metal specimens and the cross weld specimens were below the RCC-MR minimum base material curve.

Further investigations were also carried out on samples taken from a 200mm thick welded plate to investigate the effects of ageing. Samples taken from a Submerged Arc Welded plate with a chamfered narrow gap used to produce the weld. NDE checks for defects and post weld heat treatment were also carried out. Tests were performed after ageing at 450oC for periods up to 5000h. The mechanical testing included tensile and Charpy tests in the as-received and aged conditions. The results show good behaviour for the welded joint and there was no significant difference between the PWHT microstructure and the microstructure after ageing. A micro-hardness profile was taken and this showed a sharp variation which was correlated with the micro-structural variation across the welded joint and for the creep rupture, this always occurs in the base metal: the properties of the weld metal for this joint being higher than those of the base metal. Overall the results showed that ageing at 450°C does not have a significant impact on the Charpy and tensile properties and the energy values for the impact test do not change. It was considered that in order to confirm that there are no detrimental effects on material properties during reactor operation at 450^{o}C, it will be necessary to perform further tests but with much longer ageing treatment times(>10000 h).

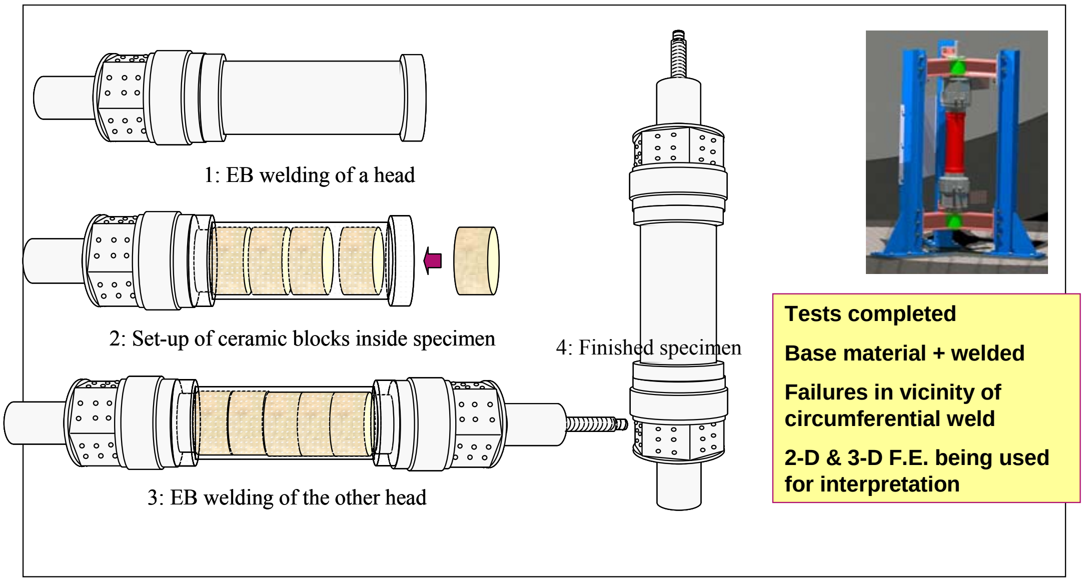
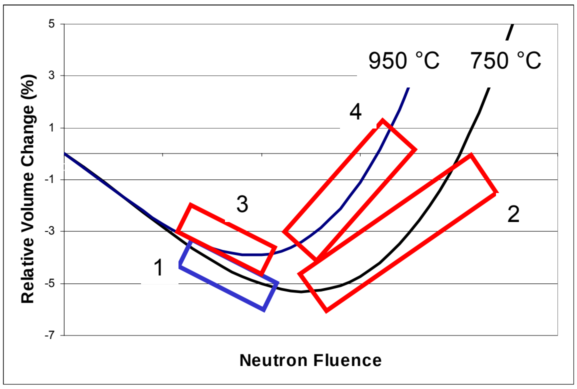


Figure 2: Reduced Scale Feature Tests

Reduced Scale Feature Tests have also been performed to investigate the behaviour of a circumferential weldment (weld metal plus base metal) under high temperature and internal pressure load. The tests were performed on a pressure vessel scaled cylinder, with a representative weld loaded under internal gas pressure (70 bar) and at elevated temperature (625^{o}C) to investigate the weldment deformation behaviour under creep conditions. The work builds on the previous small specimen tests and consolidates the fabrication advances made. Two base metal (a thermal mock-up and a reference mock-up) and two circumferentially welded samples have been tested. The main results are that all except the thermal mock-up failed following leak detection showing longitudinal cracks located in the welded zone for samples bearing a circumferential joint. The temperature gradients were also found to be more significant than expected. The results have been assessed against small specimen data and using 2-D and 3-D Finite Element modelling to understand the behaviour. It was concluded that the variation in temperature (around the circumference) has a more significant effect than the weld, suggesting that non-symmetric temperature effects could be important under accident senarios. This is a first step in developing an understanding of component behaviour.

Materials for the Core

Graphite materials are used in the HTR Core and part of the RAPHAEL materials programme includes an evaluation of the potential and properties of these materials with special emphasis on their behaviour under irradiation and oxidation. A modelling capability is also being developed to provide the basis for selection and future optimization. The properties of graphite change significantly under fast neutron irradiation and experiments are required to provide data up to the peak fluence expected over the appropriate temperature range. The target peak fluence appropriate to a pebble bed core design is around 25 displacements per atom (dpa)_graphite. The corresponding value for the replacable components of a prismatic core design should not exceed 1/3 of this value. For a HTR and (V)HTR the temperature range of interest is considered to be 600°C to 1100°C. Two irradiation experiments have been carried out within RAPHAEL in the HFR at Petten aimed at providing nominal results for different graphites at 750°C and 950°C. A schematic of the tests is shown in Figure 3.



1. INNOGRAPH-1A: 750°C, low/medium dose (FP5)
2. INNOGRAPH-1B: 750°C, high dose
3. INNOGRAPH-2A: 950°C, low/medium dose
4. INNOGRAPH-2B: 950°C, high dose

Figure 3: Graphite Irradiation tests

The first stage of the 750°C irradiation programme (INNOGRAPH 1A) was carried out within the 5th Framework Programme (FP5, HTR-M1). The INNOGRAPH 1A experiment was irradiated for 14 HFR cycles from February 2004 to April 2005. Approximately 200 small samples covering eight graphites were inserted into HFR for testing. The samples were first pre-characterised, before being irradiated to a maximum of around 1/3 peak fluence, (the peak fluence was set at ~25 displacements per atom (dpa), or ~200 x 10^{20} n.cm^2 Equivalent DIDO Nickel Dose (EDND) at a temperature of 750°C). The INNOGRAPH 1A test rig comprised a series of capsules with provision for four columns of samples in each, and each drum was then inserted in the test rig before being finally seal welded.

Within the INNOGRAPH 1A it was decide to run the upper capsules at a lower temperature (given the fluence is lower in this region). This would provide for results at 650°C in the 750°C experiment and 850°C in the 950°C experiment. For the INNOGRAPH 1A test, five graphites were selected for the test as major grades. Two graphites were produced by SGL Carbon (NBG-10 & NBG-25), two by Graftech Ltd. (PCEA & PCIB-SFG) and one by Toyo Tanso (IG-110). In addition there were three minor grades tested, one from each manufacturer (NBG-20 (SGL Carbon), PPEA (Graftech) and IG-430 (Toyo Tanso). The IG110 grade is used in both the Japanese HTTR and the Chinese HTR-10 reactors and was included as a reference material since a significant amount of high temperature irradiation data already exists. Samples were taken at the edge as well as the centre of the supplied graphite blocks and tests performed on specimens containing with-grain (WG) and against-grain (AG) orientations. Screening results from this experiment were obtained at the end of the HTR-M1 programme with full PIE for the INNOGRAPH1A experiment to be carried out in RAPHAEL (see below).

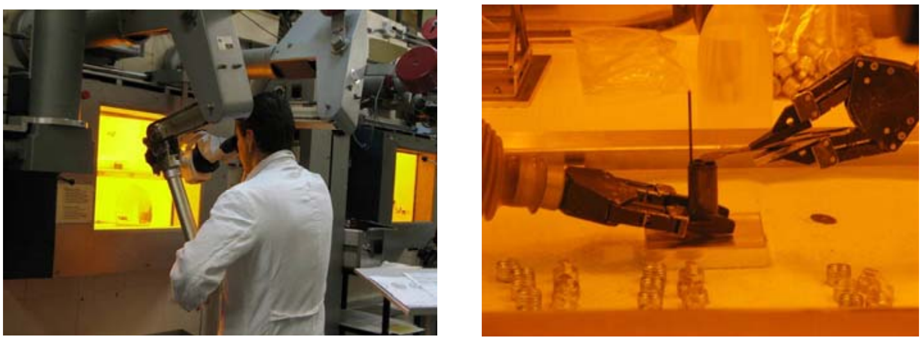


Figure4: Assembling Experiments in Hot Lab

For the INNOGRAPH 1B experiment both fresh and pre-irradiated samples were inserted which required the loading of the capsule to be carried out in shielded facilities using manipulators (see Figure 4).. The test rig for this experiment had three columns of graphite samples within the capsules in order to accommodate the expected re-expansion of the irradiated samples. Final assembly of the experiment was done outside the hot cell, with the extension to the upper section of the experiment welded on to form the top lid of the containment. Once the assembly was complete, tests were performed with respect to overall length, gas tightness and to check the resistance of the thermocouples before inserting into HFR. The INNOGRAPH 1B test was loaded into HFR on 23rd August 2007 and spent the remainder of its time (up to February 2010) in the reactor in order to receive as high a dose as possible. Screening PIE of the specimens is planned before the end of this year with full PIE to take place in a follow-on project.

Results for the full PIE assessment of the INNOGRAPH 1A experiment covering the complete first phase experiment between 5.5 and 9.9 dpa_g have been obtained for dimensional change, dynamic Young's modulus, coefficient of thermal expansion coefficient and coefficient of thermal diffusivity and conductivity. For the volumetric and dimensional changes it was confirmed that all the samples from all the grades were in the shrinkage phase. The high dose samples approach turn-round but did not pass this point. Evaluation of the INNOGRAPH 1B phase 2 results will confirm whether the graphites are suitable for a higher dose application.

For the 950°C irradiation, again two irradiation experiments were used to obtain the full design curve, the first INNOGRAPH 2A targeted at a third of the expected maximum dose (for this temperature) and the second INNOGRAPH 2B to cover two thirds of the maximum dose and to contain fresh graphite samples plus irradiated graphites from the first experiment. The target maximum dose for the 950°C condition was estimated as ~16 dpa_g. Both of the INNOGRAPH2A and INNOGRAPH2B experiments use the three column design because of the higher temperature.

The major graphite grades in the INNOGRAPH2A and 2B experiments were from SGL (NBG-10 & NBG-18) and Graftech (PCEA & PPEA). The minor grades include NBG-17 and PCIB as well as the two Toyo Tansa graphites (IG 110 & IG 430). For this experiment the drum material changed from Molybdenum to Tungsten based alloy, to generate more γ-heating to reach 950°C, and the lessons learnt from INNOGRAPH-2A and 1B (mainly issues for in-cell assembling and dismantling) were included in the experimental procedures.

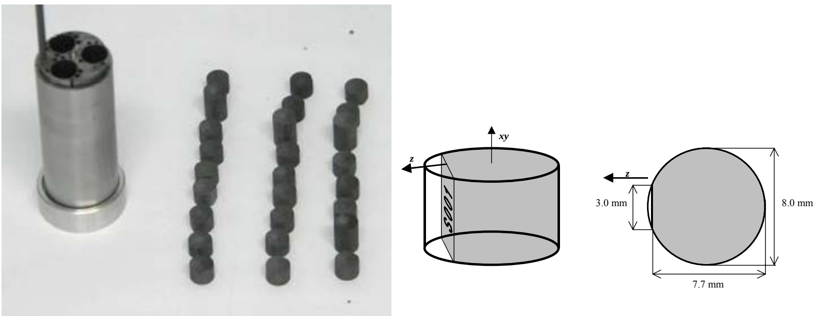


Figure5: Three column canister design

Before loading the chosen graphite samples into the INNOGRAPH2A capsules, all the specimens were again fully characterized. The INNOGRAPH2A test was loaded into HFR on July 2006 and ran for 9 cycles finishing in April 2007. After a period of cooling a preliminary screening of graphite behaviour was carried plus full PIE of 80 specimens (to be reloaded in INNOGRAPH-2B). Some of the specimens were found to have unexpected high activity randomly distributed over the grades and the rig, (caused mainly by Mn-54 which has a half-life of around ~ 313 days). However there was considered to be enough low activity specimens available for reloading into INNOGRAPH2B without a problem. The preliminary screening results for the INNOGRAPH 2A experiment suggest that turn around may have already occurred after this 4-7dpa-g level of exposure.

The INNOGRAPH2B test was loaded into HFR in July 2008 and was run as long as practicable. The experiment was finally removed in February 2010 and actions put in place to complete a screening PIE of the results. The results confirmed that turnround had occurred and that some samples had moved into the expansion phase indicating that could be unsuitable for pebble bed application. A full PIE of the INNOGRAPH1B and 2B samples is planned for a follow-on Framework project.

The overall conclusions so far from the irradiation experiment is therefore that some of the major graphite samples appear to behaved as expected and should be acceptable for the compact design at the 750°C and 950°C temperature although full PIE of the experiments is needed before this can be confirmed. With regard to application for a pebble bed reactor which exposes the graphite to higher levels of irradiation this requires further evaluation and the identification of the most suitable graphites can only be assessed after a full PIE check of the beyond turnround behaviour. From the results of the two experiments irradiation experiments a series of design curves are to be established for the different graphite samples investigated. These curves are constructed using the full PIE results which are currently only available from the first phase experiments. These design curves will form part of the basis for final graphite selection along with the results from oxidation tests and micro-structural modelling.

The test programme on graphite oxidation (due to air ingress) started in the 5th Framework HTR-M project involving tests in oxygen, and air at temperatures up to 950^{o}C. This was extended in RAPHAEL to cover effects in steam at 900^{o}C. Oxidation of the graphite also takes place during normal operation due to impurities in the cooling gases. In this case however the oxidation rate of the graphite is extremely low. The rapid oxidation data derived showed a good comparable reactivity for most of the selected graphites although for some grades there was also a major rate difference observed between the inner and outer parts of a block which can be explained by density and impurity differences between centre and edge specimens in the block.

Since investigations of the effects of neutron irradiation currently rely heavily on testing (such as that described above) and given that suitable facilities for such testing are both scarce and expensive to use and that manufacturers are continually developing and offering new graphites for such applications, in the longer term it is important to develop an alternative modelling approach (in parallel with the tests) that will both facilitate the longer-term development of graphites and minimise and reduce the extent and requirement for future irradiation testing. The technique adopted in RAPHAEL uses X –ray tomography in conjunction with model simulations to predict behaviour and has been applied to both non irradiated and irradiated samples from the experiments. The available methods have been reviewed for some of the graphites and the results indicate that irradiation-induced micro-structural (pore) changes can be detected in the tomography data which gives some confidence that the computed X-ray tomography technique can be used to gain further insight into the micro-structural changes within the graphite and also be used in the development of predictive models.

Materials for Control Rod

The potential for using carbon composite materials as an alternative to Alloy 800H for the control rod has also been investigated. Work has focussed on architecture design, modelling and some preliminary irradiation testing of possible materials (in EXTREMAT). A specification was prepared that covers a description of the application, environmental conditions, material composition, electrical and mechanical properties, stability and product characteristics. The work progressed to an improved understanding of materials and property requirements for this type of component. Carbon composites have to be tailored for each application to meet design requirements with the manufacturing parameters adjusted to fulfil the technical requirements and to match the component geometry. Optimization of suitable materials requires adjustment of the fibre architecture and matrix material and therefore requires the co-operation and involvement of the manufacturer. Given the potential cost and that a substantial development would be needed for this type of material as well as new codes and standards, its likely that this material would be used only for very high temperature operation. Nevertheless the work performed represented a good initial study of the possibilities and it is expected that following investigation of the irradiation tests within EXTREMAT irradiation, that further evaluations and recommendations for composite material applications will be made in a future Framework.

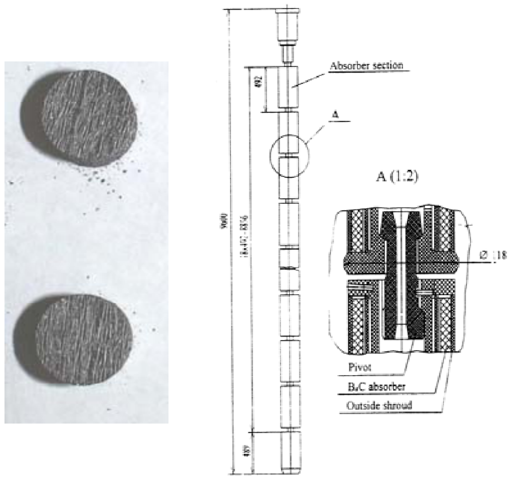


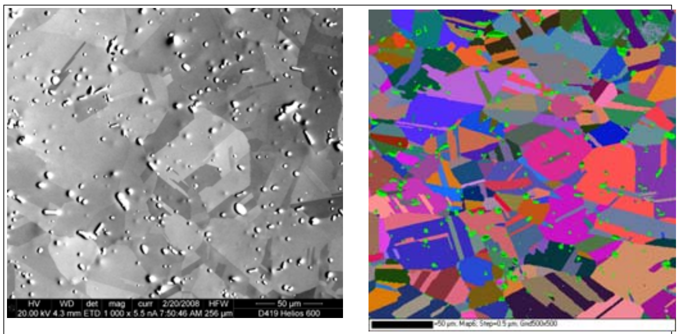
Figure 6: Carbon composite investigations

Materials for the heat carrying/ generating components

A review of available materials for the key high temperature components (internals, hot gas duct, heat exchangers) plus a short list of most promising materials for evaluation was performed. The review considered requirements, composition, physical and thermo-mechanical properties of candidate materials. No industrially developed material is currently available for operation at Very High temperatures. ODS steels provide a possible option however these have not been developed sufficiently with respect to fabrication requirements for heat exchanger application.

Currently the two main candidates for the IHX are considered to be IN 617 and Haynes 230 which are considered good candidates for long operating periods (<850^{o}C) and potential transients. Alloy 617 already has a very large database with numerous mechanical properties and creep data measured in air and a helium atmosphere. A list of information requirements was established and an outline of a test programme covering creep behaviour and effects of ageing on materials strength was established with Haynes 230 alloy selected for the tests (at 850 and 950^{o}C). The investigations include a study of the effect of cold work (induced by manufacturing) and some thermal ageing tests, looking at microstructure observations and comparing the mechanical properties with and without the ageing effects.

The cold work was introduced using tensile tests at room temperature (strain rate of 4.10-4s-1), 20mm gauge length and 4mm diameter, interrupting the tests at selected total elongation levels. Creep tests were then performed at 850 and 950^{o}C. At 850^{o}C the addition of cold work is to significantly decrease the steady state creep rate and to extend the time to rupture. The % elongation is also markedly reduced and the time to reach a given elongation increased. The effect of 10% cold work on creep properties appears beneficial however an increased scatter is observed with a 17% test. At 850°C cold work produces a strong hardening reducing the transient creep stage and strongly reducing the steady state creep rate. The same trend has been observed on Inconel 617. At the higher temperature of 950°C, only the 10% cold work level; seems to have a beneficial effect on steady state creep rate and life time and at this temperature the 17% level of cold work on Haynes 230 should be avoided. The ageing tests involve the determination of mechanical properties with and without ageing (durations 1000h and 5000h) in the temperature range of 850°C-1000°C. Mechanical tests were performed (tensile tests, impact toughness tests), as well as micro-structural examinations (SEM + TEM). Haynes 230 material was again used and, tensile tests were performed at room temperature and in the temperature range of 850°C and 1000°C to cover IHX operating conditions and accident conditions. The results show the tensile properties (yield strength and tensile strength) to decrease more than 50% at 850°C compared with values at room temperature. On the other hand the elongation at rupture increases significantly. These results show an increase in ductility for material at high temperature.



Grain size ; 30 µm to 50 µm

Many carbides M6C enriched in W

Figure 7: Metallurgical Characterisation of Haynes 230

The effect of ageing for 1000h suggests that there is no strong effect on strength but there is a strong effect on ductility. Charpy tests were also performed at room temperature on two specimens for each condition (as received and aged). The results show a large decrease in impact toughness after ageing of 1000h. Repeating the tests but with a 5000h aged specimen showed a similar result to the 1000h case. The reduction in the mechanical properties and impact strength for all aged states appears to be linked to a growth in rapid intergranular carbides (type $Cr_{23}C_6$) and it is likely that properties such as impact values may not be stabilized after 5000h of ageing and have to be investigated further for longer exposure times. Overall the results show that the effect of cold work is important to the selection of the materials also the effect of ageing. Further testing is needed on a larger number of samples in order to provide conclusive evidence on behaviour.

Conclusions and Recommendations

Significant advances have been made in the development of a more temperature resistant material (Mod 9Cr 1Mo steel) for the vessel with important breakthroughs in fabrication, understanding of thick weld behaviour, sensitivity to thermal ageing and irradiation. The application of such a steel simplifies the design and avoids the need for an additional cooling circuit and protection from the inlet gas. The graphite core is a key component that affects safety and operability of the reactor.

The graphite components provide channels for fuel cooling and control rods and in-reactor irradiation induced changes to graphite properties and dimensions affect graphite component integrity which is important for design. The graphite materials work. The RAPHAEL programme comprises irradiation tests at 750^{o}C and 950^{o}C with irradiation levels up to 23dpa. These tests provide the basis for selection for both the compact and pebble bed designs. PIE of the second phase experiments remains a priority for a next proposal. Within GIF these tests represent the state of the Art. Additional tests are planned particularly with irradiation creep but these will not come to a conclusion until a few years time. The EU irradiation programme is therefore likely to provide the basis for graphite selection for the next deployment of the HTR.

For the heat transport components nickel based alloys are proposed since these materials offer the best potential for resistance to high temperatures. The primary issues affecting heat exchanger material choice are creep strength and corrosion resistance. The current range of potential materials include IN 617, Haynes 230, which are currently the primary choices for HTR temperatures. No industrially developed material is currently available for VHTR operation. Tests have been performed on Haynes 230 covering effects of creep, ageing and cold work which have contributed to the current state of the Art for this material. Within GIF IN 617 remains a prime choice since this has the most extensive database, however work continues on all other options.

1 Introduction

This report summarises the work performed within SP4 ML on the development of HTR materials. The areas of work covered by the materials work within RAPHAEL IP are as follows:

- Reactor Pressure Vessel
  - Tests on Mod 9Cr 1Mo steel welded joints under irradiated and non-irradiated conditions to determine suitability for vessel application.

Fabrication and the effects of irradiation/environment on the HTR vessel weld behaviour are crucial issues in terms of structural viability. These have been addressed in the programme and provide key results for material selection development and qualification.

- High temperature materials
  - Review and database actions for the heat exchangers
  - Tests on selected materials (carbon fibre reinforced carbon (C/C) composites, high alloy steels) at temperature and under short and intermediate times in air, including the effects of ageing

Temperatures and environmental degradation are the main concerns for the direct and indirect cycle options. Fabrication, creep and the effect of helium on properties and strength are required for the materials investigated. Fabrication and effects of irradiation/environment are crucial issues for C-based materials seen as future candidates for control rods. Recommendations are made for future investigations and tests and for other component applications.

- Graphite core
  - Oxidation tests on graphites and C composites
  - Selection of most promising currently available graphites and irradiation testing at 750ºC and 950ºC
  - Heat-up experiment
  - Micro-structural modelling & guidelines

These graphite actions are seen as important first steps in qualifying new graphites for future HTR and VHTR application, given that almost all the graphites previously irradiated are no longer manufactured. Recommendations are made with respect to a graphite selection strategy and the work on international collaboration.

In addition management, communication and education actions have been performed as well as exchanges with other Projects These include the organisation and progressing of the work through: joint technical meetings and participation at Executive Board and Steering Committee meetings; the presentation of the work at European and International Conferences (10 papers published) and the GIF forum; and the participation in educational Eurocourses for young engineers including the provision of a text book These activities are described further in section 7 of this report.

This report has been prepared as a technical contribution to the Final Report of the RAPHAEL Project.

2 Summary of SP4 Work package 1 on Vessel Materials

This section summarises the work performed within Work package 1 of SP4 ML in support of the development of Mod 9Cr 1Mo steel as a reactor pressure vessel material.

Based on results from the earlier EU projects HTR-M and HTR-M1, the objective was to test and evaluate the feasibility of Modified 9Cr 1 Mo steel as a candidate material for the non-cooled pressure vessel of the VHTR. The tasks performed include post irradiation examination (PIE) of pre-irradiated base material and weld specimens from the HTR-M Fifth Framework programme, component feature tests on reduced scale cylinders with and without a circumferential weld and ageing tests.

The results from these investigations and tests are summarised and discussed below in the light of component qualification requirements.

2.1 Vessel materials

The design pressures and internal diameters of HTR and VHTR pressure vessels fall within a relatively narrow range due to constraints imposed by requirements for passive heat removal under loss of circulation or loss of coolant scenarios and the requirement to meet the allowable stress levels in various design codes. For the HTR and VHTR the material used for the RPV is required to operate at temperatures higher than 450oC (if not directly cooled) and within an operating environment that includes exposure to neutron-irradiation and helium gas. Different materials are used for RPV’s, depending on whether the temperature of the pressure boundary under normal operation or design conditions exceeds the limit for LWR pressure vessel operation. For the investigations within RAPHAEL Mod 9Cr 1Mo steel has been selected which has an important advantage in being more temperature (and transient) tolerant than LWR steel and a higher strength at elevated temperatures.

The main concerns for the VHTR RPV are in the vicinity of the welds. The welds carry an additional requirement to satisfy the safety analysis methodology and safety studies (defect location and defect size) and have to be assessed from the point of view of non-destructive examination and potential for failure or leakage.

The main damage mechanisms to be addressed are fracture, fatigue and creep -fatigue. These have to be evaluated from an initiation aspect and for crack growth of defects under fluctuating thermal and mechanical loads. The potential effects of environment (temperature, irradiation, ageing) all have to be taken into account. Sensitive zones are areas that feature thicker sections (potential for reduced properties and strength), hot spots and regions important from a functionality point of view. This includes the flange area and belt line, which have to satisfy primary and secondary stress limit requirements and progressive deformation mechanisms. The important issues to address are toughness and creep behaviour under as-fabricated and simulated end-of-life conditions. This is to provide an understanding of the welding processes, weld metals and post-weld heat treatment required for thick welds and weld factors to be applied on the base material properties.

2.2 Tests on Pre-Irradiated specimens

Under operating conditions the reactor pressure vessel will be irradiated with neutrons, which cause damage within the material and will result in a change in mechanical behaviour of the material and in the welds. 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. 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 Mod 9Cr1Mo steel irradiated under conditions expected for the HTR and VHTR no data are available. A review showed that information has 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 Mod. 9Cr 1Mo steel welds. In view of this irradiation experiments were performed within the HTR-M project to determine the effect of neutron-irradiation on properties of the RPV thick section welds made from Mod 9Cr 1 Mo steel. The tests and subsequent post irradiation examinations are briefly described below and reported in [Refs 2-1, 2-2].

The HTR-M Irradiation tests & results of PIE

For the HTR-M irradiation tests, thick section weld test sections were manufactured (by AREVA) using a narrow gap TIG process with a double V weld preparation and post weld heat treatment. The first pilot test welds (150 mm thick) showed a significant number of micro-cracks in the weld metal. These cracks (< 0.3 mm) were not detected initially but seen when performing a micrographic examination. This was unexpected as the selected filler had the lowest tendency to hot cracking among the products tested. These heat cracks in parts of the weld were later eliminated through adjustments to the welding procedure. A specialised inspection/ cutting procedure has been developed to ensure there were no cracks present in the tested specimens. This fabrication work represented the first in a series of trials aimed at confirming the fabrication parameters for the HTR RPV application. They showed the importance of optimising the filler material composition and welding procedures to avoid cracking and optimise strength. Subsequent development work continued within AREVA to explain the reasons for the phenomenon and to help with the selection of a future filler material that would avoid this problem.

The specimens were tested in the main stress directions with sections taken from cutting planes of approximately 15mm thickness and parallel to the weld direction. The tests were carried out both for the reference condition and End of Life (EOL) condition. The root, bottom, and top were 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

Four conditions were selected for the mechanical testing: plate material in the parallel and perpendicular directions (with respect to the rolling direction), the heat affected zone (HAZ), and the weld metal (WM). The specimen geometry for the reference testing and post irradiation examination (PIE) testing were the same for all specimen types. A series of round robin tests between the two facilities undertaking the tests (one in shielded facilities) was carried out to check consistency before the irradiation tests began. The irradiation which was started at the beginning of 2004 and took place in the poolside facility of the High Flux Reactor (HFR) at Petten was carried out at a single irradiation temperature of 375°C and an average fluence of $9.44 \cdot 10^{22}$ n/m^2, E>0.1 MeV (~5.8 mdpa), achieved in two HFR loading cycles was selected as representative HFR vessel conditions. The specimens were loaded into the Lyra test facility and during the test the specimen fluence gradients were kept as low as possible. The flux buckling and self-shielding were determined using neutron monitoring sets and taken into account in the evaluation of the materials behaviour.

Post Irradiation Examination (PIE) was carried out to determine tensile, creep, impact and fracture properties in the shielded facilities within NRG. The tensile tests were performed at different temperatures, the creep tests at 550°C and the fracture toughness tests at room temperature (RT) and 375°C with the results used to construct J-Δa curves. Equivalent non-irradiated tests were also carried out on material subjected to an equivalent thermal ageing cycle. As with the PIE tests, tensile and creep data were obtained on base material specimens (longitudinal and transverse (L and T) directions), all weld metal specimens (WM) and cross weld (CW) specimens. Comparisons were made with other available non-irradiated data and also for creep with non-factored values from the RCC-MR Code. Impact values were determined using sub-size and full size specimens, the latter to study size effects. The test-matrix specification of Vessel materials PIE test programme for the RAPHAEL tests are given in [Ref.2-2]

The results of the HTR-M test results are given in [Ref. 2-3]. For all the material conditions investigated, the irradiation effect was small compared with batch-to-batch variations. A slight increase in room temperature yield strength was observed as a result of the irradiation but there was no increase in the 550°C yield strength. For the welded specimens the weld metal had the highest ductility, the cross weld specimens the lowest, and as temperatures increase the yield to ultimate strength ratio increased (especially between 450 and 550°C). From the creep tests there was no significant effect of irradiation on the rupture strengths of either the base material or weld metal. The only systematic influence of the irradiation was seen in the cross-weld tests where testing times were longer and minimum strain rates lower. The impact tests on non-irradiated sub-sized specimens showed that the lowest ductile brittle transition temperatures (DBTT) occurred in the HAZ specimens and in the weld material. Both plate directions showed similar DBTT results. The weld metal also showed the highest upper-shelf energy (USE) confirming the high upper shelf fracture toughness of this material. The irradiated sub-sized impact specimens also showed the HAZ of the weld to give the lowest DBTT as with the non-irradiated tests. The assessment confirmed that the RCC-MR weld factor Jr = 0.9 for Mod 9Cr 1 Mo steel weldments was applicable to these RPV weld tests.

Results from the RAPHAEL PIE investigations

Not all the irradiated HTR-M specimens were tested and 16 pre-irradiated specimens were held over for the RAPHAEL-IP programme in order to widen the results for the database. The aim was to expand the available database to cover creep at lower temperatures (e.g. 450^{o}C) representative of conditions closer to normal operating or design conditions. This would also provide an important confirmation that the effects of irradiation on creep strength are still negligible at this condition.

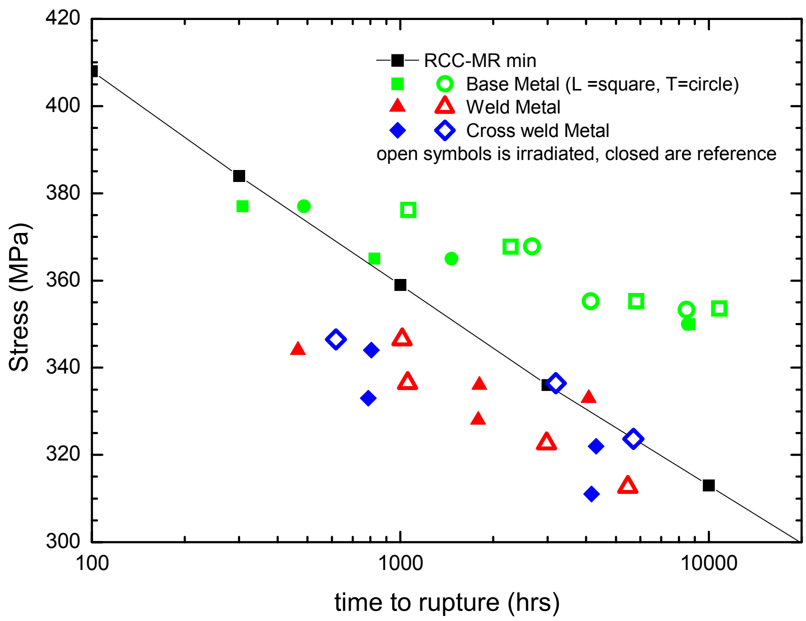


Figure 2-1 Stress-rupture plot of the base material results in transverse and longitudinal direction, the cross weld and weld metal results

The results from the RAPHAEL pre-irradiated specimens and the reference tests are reported in [Ref. 2-4]. The overall effects of the irradiation was found to be small. This was the same conclusion drawn from the HTR-M irradiations where it was judged that the changes due to the irradiation were within the batch to batch variations of the material (see [Ref. 2-5]). The results from the base metals tests and the weld tests show the base-metal results to be above the RCC-MR minimum curve, also there appeared to be different slope for both reference and irradiated material compared to the RCC-MR minimum curves for 9Cr1Mo steel (see Figure 2-

1). This change in slope may result from the post weld heat treatment that was given to the plate after welding. However despite the typical large scatter in creep-rupture, a clear difference was observed between the weld and cross weld specimens. The differences between the cross weld and weld specimens was considered to result from the differences in microstructure caused by the welding. The weld metal specimens as well as the cross weld specimens are below the RCC-MR minimum curve (see Figure 2-1). An overall comparison of the creep results at 450°C in comparison with those obtained at 550°C is shown in Figure 2-2.

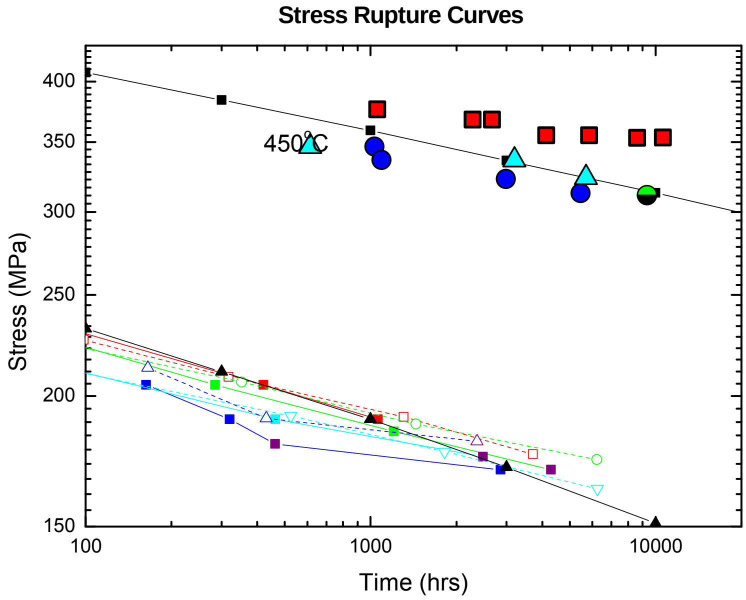


Figure 2-2 Stress-rupture plot showing the 450°C base material and weld results in comparison with those obtained at 550°C in HTR-M.

2.3 Reduced Scale Component Testing

Reduced Scale Feature Tests have been performed within RAPHAEL to investigate the behaviour of a circumferential weldment (weld metal plus base metal) under high temperature and internal pressure load. The tests were performed on a pressure vessel scaled cylinder, with a representative weld loaded under internal gas pressure (70 bar) and at elevated temperature (625°C) to investigate the weldment deformation behaviour under creep conditions. The main purpose of the tests was to provide a more accurate interpretation of the weldment creep factor, which is dependent on loading and geometry. The work builds on the small specimen tests of HTR-M & M1 and consolidates the fabrication advances made in these projects. The basis is that the representative structure will be more creep resistant compared with laboratory specimens and this can be taken into account in the modelling.

The cylindrical test pieces are machined from a 70 cm length of pipe (extracted from an initially procured piece (dia: 310 mm; thickness: 50 mm; length: 5.4 m) and comprise the sample plus ceramic blocks (to reduce the gas volume inside) and two headers welded to each end. The assembly is sited in a facility designed for burst prevention and uses thermocouples located inside and outside the mock-up. Radial displacement transducers were used on the periphery and different insulation options investigated to help stabilize and maximise the extent of the temperature profile.

Two base metal (a thermal mock-up and a reference mock-up- see Figure 2-3) and two circumferentially welded samples were tested [Ref. 2-10]. Base metal characterization tests were performed in the as delivered and heat treated conditions to confirm the properties. The main results show that all except the thermal mock-up failed following leak detection due to the formation of longitudinal cracks, which were located in the welded zone for the samples bearing a circumferential joint (see Figure 2-4). The temperature gradients were also found to be more significant than expected. The results have been assessed against small specimen data and using 2-D and 3-D Finite Element models to understand the behaviour. A large number of parametric calculations have also been performed [Ref. 2-11] to investigate the influence of temperature variations with time, temperature gradients, presence of the weld, old verses new base metal coefficients, etc.. This is a first step in developing an understanding of component behaviour.

Figure 2-3 View of instrumented test piece

The Finite Element calculations show that it is possible to evaluate the creep life of the base metal and welded tubes using creep strain and damage constitutive equations from the Ecole des Mines of Paris. The method requires a detailed mesh of all the influencing parts, i.e. the weld metal, the Heat Affected Zone (HAZ), and the base metal. It has also been found that the presence of the weld and the HAZ are not as significant on creep life as the thickness of the tube and variation in temperature. A variation in temperature (around the circumference) of 5°C had a larger effect than the weld, for the specimens. Also the 3D calculations have shown that a circumferential temperature gradient of 5°C leads to a relatively good representation of the radial displacements measured around the tubes.

Figure 2-4: View of longitudinal crack across the weld from one HAZ to the other

Overall, with respect to the modelling, the finite element parametric calculations show that the thickness of the tube and the width of the HAZ are the only parameters driving the size effect on the creep life of welded component at 625°C. Thus, an alternative rule to the classical joint creep factors is possible giving the life reduction in terms of time or in terms of stress. However, in view of the small number of results, this alternative rule should not be used for another material, or geometry or for a temperature below 600°C. Further investigations are required to extend the validity of these results to different configurations.

2.4 Material Ageing and Embrittlement

Within SP4 ML Work Package 1 some investigations were performed on the effects of ageing on material properties. Ageing describes a continuous time or operational degradation of the material due to operating conditions, that can result in a significant reduction in strength over the operating life.

The severity of the ageing effect depends on a number of factors including:

- The prior austenite grain size of the material.
- The concentration and distribution of the trace elements (P, Sn, As, Sb).
- The cooling cycle after tempering.
- Time at temperature(s) within the critical range.
- The property considered.

The base materials and weld metals for both cold (LWR) and warm HTR (Mod 9Cr 1Mo steel) vessel normally have very fine grain sizes. The most likely embrittlement location is therefore the coarse grained heat affected zone. Because the LWR (cooled) vessels operate at temperatures below the material embrittlement range, it usually can only occur for this material during transients involving exposure to higher temperatures for tens or hundreds of hours. The (non-cooled) Mod 9Cr 1Mo steel vessel can operate at higher temperature and in the ageing temperature range throughout their lifetime and so for the Mod. 9Cr 1Mo steel vessel ageing can become more important for integrity. The approximate duration after which ageing effects can be signifiacnt can be the order of 20-30 years and its predictability depends upon technical understanding of the conditions and the quality of the operational experience data. Its predictability is also dependent on the ability to monitor the condition of the component and to model it.

Details of the RAPHAEL ageing experimental programme

No ageing tests were performed in the HTR-M 5th Framework Programme but such tests were recommended for RAPHAEL. The RAPHAEL tests were carried out on parent plate and weld specimens taken from a 200mm thick welded plate (Figure 2-5) fabricated by AREVA, [Ref. 2-6]. Specimens were cut from the weld metal and heat affected zone regions (HAZ positioned in the centre of the test piece), see Figure 2-6 and tests performed on as-received and 5000h aged (450^{o}C) material with impact and tensile tests carried out at ambient and the temperature of the heat treatment to assess the effects of the ageing on material strength [Ref. 2-7]. Additionally micro-structural examinations in the form of optical and SEM examinations (TEM examinations if necessary for behaviour understanding) were carried out. For the construction of the Charpy test curves (longitudinal and transverse directions) 16-20 specimens were used for each curve .

Figure 2-5 Welded plate used for Ageing tests

The results show good behaviour for the welded joint with no significant difference between the post weld heat treatment (PWHT) microstructure and the microstructure after ageing for 5000 h at 450°C.

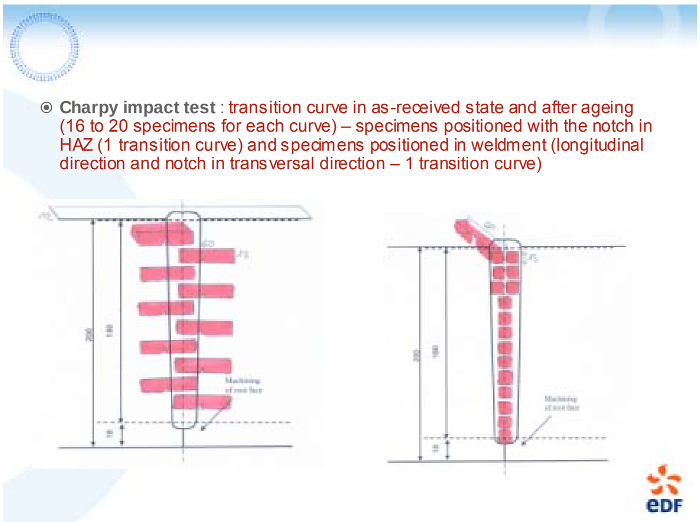


Figure 2-6 Specimen layout for Charpy aging tests

For the mechanical behaviour, the micro-hardness profile shows a sharp variation which has been correlated to the micro-structural variation across the welded joint (figure 2-7). Tensile properties of the base material are not affected by the cutting direction and for the samples cut across the welded joint, creep rupture always occurs in the base metal: the properties of the weld metal for this joint being higher than those of the base metal. Charpy transition curves for the different parts of the joint show the ductile-brittle transition temperature (DBTT) is always lower than the corresponding value at room temperature. Lower results were obtained in the HAZ with higher values in the weld metal which also has the lower level of ductility. Overall the results show that ageing at 450°C does not significantly affect the Charpy materials properties (Figure 2-8) and the tensile properties or the energy values for impact test do not change after ageing for 5000 h at 450°C. To confirm there are no detrimental effects on material properties at longer times, it is recommended that further tests are performed with ageing treatment times >10000 h.

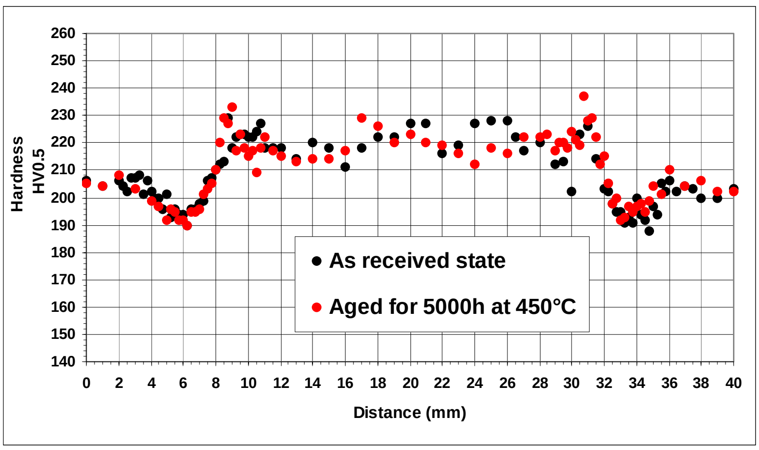


Figure 2-7 – Cross weld hardness (HV0.5) profile after PWHT and ageing for 5000 h 450°C

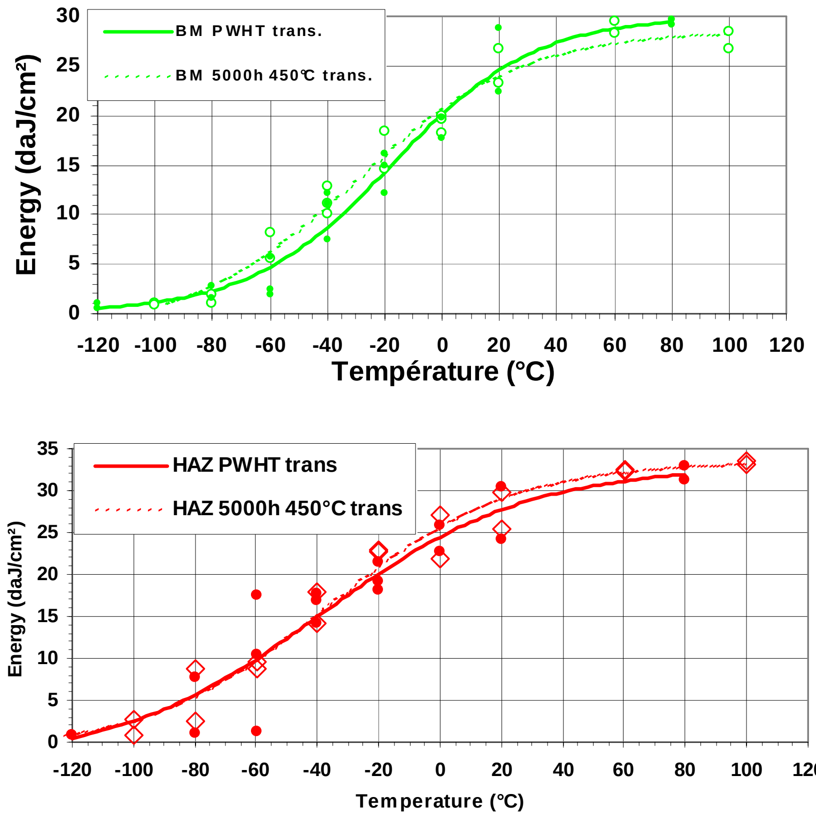


Figure 2-8 Charpy transition curves: comparison of PWHT and behaviour after aging for 5000 h at 450°C

2.5 Overall Conclusions and Recommendations on the Vessel Material Investigations

Effects of irradiation at 450°C

The creep tests at 450°C suggest that:

- The effects of the pre-irradiation on material behaviour was small and considered to be within the batch to batch variation of the material.
- The Mod 9Cr 1Mo steel base material behaves similar in both the Longitudinal and transverse directions
- The weld metal and the cross weld specimens were shown to be weaker than the base material
- The slope of the stress versus rupture time curves appears slightly different to the RCC-MR minimum curve
- Similar difference between weld metal and base metal were found as in the earlier HTR-M tests at 550°C

It is recommended that further evaluation of the negligible creep limit and effects of creep/fatigue interaction is performed at representative operating conditions for the vessel.

Reduced scale component tests

The reduced scale component tests at 625°C and 150 bar suggest that:

- The thickness of the tube and the width of the HAZ are key parameters driving the size fect on the creep life of the welded component
- A temperture variation of 5°C around the circumference was more significant on behaviour at these temperatures than the presence of the weld.
- An alternative rule to the classical joint creep factors is possible in terms of time or stress. However, because of the small number of results, the developed rules should not be used for another material, or geometry or for a temperature below 600°C,

Further tests are recommended to extend the validity of these results to different welding configurations (i.e. include longitudinal welds).

Material Aging tests

The results suggest that:

- No significant effects of ageing were observed at 450°C and 5000h aging time,
- At this temperature and duration there was no significant impact on the effect of materials properties and metallurgical variations.

Further tests will be necessary using much longer ageing treatment times(>10000 h) to confirm that there will be no detrimental effects on material properties at 450°C during reactor operation,

Contribution to the qualification of Mod 9Cr 1Mo as a RPV VHTR steel

Significant progress has been made on understanding the behaviour of the weld and parent plate material under irradiation and at temperatures nearer and above the normal operational temperature of the vessel.

Further tests are recommended in future framework programmes using longer dwell times and different weld parameters and geometries.

Creep tests at 450 and 550°C up to 10000 h show that the rupture stress is a little weaker for welded joints compared with base metal (about 10% lower). The next target would be to confirm that the long term strength of the joint in the same temperature range is consistent with this, and that the design weld factor is applicable in both normal operation and accidental conditions.

Future effort should concentrate on very representative samples, with the base material ideally from prototype forged parts and the welded joints representative of HTR/VHTR vessel fabrication route with regard to weld metal properties and welding procedures including simulated post weld heat treatments and covering the fabrication hazards in a pessimistic way.

Summary reports on the effects of environment and recommendations for future vessel material development have been produced [Ref. 2-12, 2-13], also a summary report on the WP1 findings [Ref. 2-14]. To date little effort has been channelled towards inspection issues and in particular inspection of welds and In Service Inspection (ISI) requirements. Such methods for thick section Mod 9Cr Cr1Mo steel plate and forgings will need to be developed, as currently most applications for this material are in thin sections such as for heat exchangers. Such NDE method development is considered a high priority by Regulators and will be particularly important to demonstrate for a first of a kind vessel.

2.6 Vessel Material References

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2-2 J.B.J. Hegeman, *Loading report LYRA-08 (304-08), irradiation in the frame of HTR-M*, NRG Report 20585/03.56740/P, December 2003

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2-4 J.B.J. Hegeman, PIE & 450^{o}C Creep testing of pre-irradiated weld specimens, RAPHAEL 0904 D-ML1-4

2-5 D. Buckthorpe, HTR-M Final Report

2-6 D Pierron, Modified 9Cr-1Mo steel Vessel – Procurement and Manufacture of the welded joints, RAPHAEL 0710 D ML1.3

2-7 M. Blat-Yrieix, et al. Results from Ageing tests on thick section Mod 9cR 1Mo steel welds for VHTR Reactor Pressure Vessel application, RAPHAEL 0904 S ML1.3

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2-13 O Gelineau, D Buckthorpe, R. García, Recommendations for the VHTR , RAPHAEL 0904-D-ML1.8.

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3 Summary of SP4 Work package 2 on High Temperature Metal Materials

Investigations on high temperature metallic materials within RAPHAEL-IP is addressed within WP2 and covers material selection for the high temperature components such as the heat exchanger. The activities covered include a review of possible materials and their limits, the selection and testing of an IHX alloy (Haynes 230) under cold work and an investigation on the effects of ageing. Also studied are certain higher strength metallic materials such as oxide dispersion strength (ODS) steels and TiAl alloys. This section summarises the work performed together with the support activities given by SP ML to SP CT on metallic components and the status of the Metals Working Group of GIF.

3.1 Investigations of the limits of High Temperature Materials

A review of available materials for the key high temperature components (internals, hot gas duct, heat exchangers) of the VHTR plus a short list of most promising materials for evaluation has been made in [Ref. 3-1]. This was preliminary investigation from which a more detailed study was initiated as part of a joint exchange with SP CT on requirements for the heat exchanger. From this work candidate materials were selected for testing and further investigation. A recommendation with regard to a generic test programme (short term plus ageing tests) has also been made. The review considered requirements, composition, physical and thermo-mechanical properties of candidate materials. Nickel -Chrome solid solution alloys such as IN 617, Haynes 230 , Hastelloy X, Alloy 800H are the most promising candidate materials for the VHTR Internals, Hot Gas Duct and Intermediate Heat exchanger. Other alloys such as ODS alloys will be considered due to their high thermal stability and creep strength.

Currently the two main candidate materials for the IHX are considered to be IN 617 and Haynes 230. Alloy 617 already has a very large database with numerous mechanical properties and creep data measured in air and a helium atmosphere. A draft code case for this material was developed in the early 1980's but this work was stopped as the need for the code case disappeared. Within RAPHAEL a list of information requirements was made and an outline of a test programme covering creep behaviour and effects of ageing on materials strength was developed

The available materials and their limits for the key high temperature components (internals, hot gas duct, heat exchangers) of the VHTR have been reviewed (see Table 3-1) in a second report [Ref. 3-2]. The report provides a review of materials and their limits for the key high temperature components (internals, hot gas duct, heat exchangers) together with a short list of materials for evaluation. From this review the most promising available candidate materials have been selected and recommendations made for future development of some new alloys more adaptable to the VHTR upper temperature limit requirements.

Material	Temp Limit °C	Limiting Factor	Further Development	Issues for VHTR/ RAPHAEL
Mod 9Cr 1Mo steel	**Operating: 550°C**	Long term Creep strength	ODS version capable of 100°C Higher temperatures - Fusion development – to be tested in future framework.	Established material for sodium and Gas cooled systems—moderate temperatures
	Max: 650°C			
Austenitic Stainless Steel	**Operating: 550°C**	Long term Creep strength		Established material for sodium and Gas cooled systems—moderate temperatures
	Max: 750°C			
Alloy 800 & Alloy 800H	**Operating: 650-750°C**	Long term Creep strength		Used in gas cooled and Sodium cooled systems.
	Max: 920-980°C	Corrosion		
Alloy 617	**Operating: 750-850°C**	Long term Creep strength <10Mpa @ 100,000h	ODS version possible for Higher temperatures Effect of cold work (forming) on creep strength - limit of 5%	Established in German HTR Programme - KTA Rules developed For ODS version Micro-structural development required as a first step -
	Max: 920-980°C	Corrosion; Cr_2O_3 evaporation		
Alloy 230	**Operating: 750-850°C**	Long term Creep strength <10Mpa @ 100,000h	ODS version possible for Higher temperatures Effect of cold work (forming) on creep strength absent from National Programmes	Selected as industrially developed alternative to IN 617 – for corrosion resistance For ODS version Micro-structural development required – Investigation of cold work on creep strength & ageing Developments to be included in next stage of RAPHAEL programme
	Max: 920-980°C	Corrosion; Cr_2O_3 evaporation		
Alloy XR	**Operating: 750-850°C**	Long term Creep strength <10Mpa @ 100,000h	Effect of cold work (manufacturing) on creep strength - limit of up to 10% Tests @800-1000°C	Established for HTTR Operation at 750°C
	Max: 920-980°C	Corrosion; Cr_2O_3 evaporation		
ODS - Ferritic	**Operating: 550°C**		Manufacturing development required Investigated in EXTREMAT	Not developed for heat exchanger application – significant programme required on joining/forming involving manufacturer
	Max: 650°C			
ODS - Austenitic	**Operating: >550°C**		Manufacturing development required Investigated in EXTREMAT	Not developed for heat exchanger application – significant programme required on joining/forming involving manufacturer
	Max: >650°C			

Table 3-1 Heat Exchanger materials and their limits

A test program was proposed to improve the knowledge of high temperature mechanical properties and microstructure. The tests proposed included creep, thermal ageing, and effects cold work on mechanical properties (which is important for manufacture) in the temperature range [700°C-1000°C].

An interim assessment of ODS and TiAl materials was also made covering effects of thermal and irradiation creep. The alloys were assessed at temperatures exceeding 950°C using 200µm thick specimens. The results have been compared with information on Ni based alloys and found to give increased creep strength compared with Alloy 617. The assessment showed the TiAl material to be a factor of 2 stronger in terms of creep rupture stress. Although this is a promising conclusion a number of issues remain to be addressed including micro-structural stability, longer term data and data scatter, possible production routes, environment, low temperature brittleness and potential for bonding. For ODS results at 850°C and 1100°C for the 104h creep rupture strength, showed an increased value compared with Alloy 617 at higher temperatures but at temperatures below 700°C, the creep rupture strength is lower. For this material, despite its potential for increased strength, there are many additional investigations required particularly with respect to its manufacturing capabilities

3.2 Procurement and tests on selected material Haynes 230

The RAPHAEL test program [Ref 3-3] to examine high temperature mechanical properties and microstructure was developed for the Haynes 230 alloy. The investigations include tests to study the effect of cold work (induced by manufacturing) on the creep behaviour looking at the effects of temperature and cold work levels and some thermal ageing tests, comparing the mechanical properties and looking at microstructure observations with and without the ageing effects. A second step was to improve the knowledge of high temperature mechanical properties and the microstructure of alloy under development such asODS alloy or TiAl intermetallic alloys. Details of the tests and the procurements made are summarized below:

CEA test program

The program covers the evaluation of high temperature properties covering the effects of cold work and ageing on creep. The cold work tests involve creep at 850°C and 950°C on specimens with and without pre-deformation (17% & 10%). Alloy Haynes 230 was commercially procured from Haynes International in the form of hot-rolled round bar in the as-received condition. The bars were annealed at 1230°C (held 30 minutes) and water quenched. Two hot rolled round bars were used (19,6mm and 16mm diameter rods respectively) and were obtained from two separate heats from the same manufacturer. The chemical composition is given in Table 3-2 below:

Element Content (%)	Cr	W	Mo	Co	Fe	Al	Si	C	La	B
Bar 19,05 mm	21,73	13,98	1,24	0,11	1,44	0,39	0,43	0,11	0,012	0,003
Bar 16mm	21,83	13,77	1,3	0,14	1,48	0,39	0,41	0,11	0,014	0,004

Table 3-2: Chemical composition of the two bars.(in wt.% Element Contentaries)

The variation in chemistry between the two bars was minor, the differences being 0.2% in tungsten and 0.1% in chromium. The most notable difference was in hardness value 207HB compared to 177HB. The as-received microstructure is presented in Figure 3-1, Alloy 230 is a solid-solution strengthened material with primary carbides (tungsten -rich M6Cand primarily Cr rich M23C6) providing the grain size.

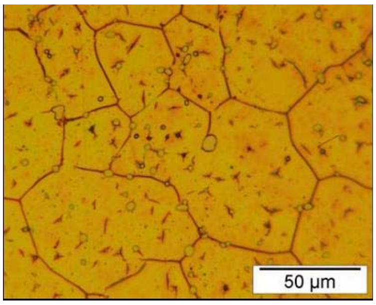


Figure 3-1: HAYNES 230® alloy bar 19mm as received microstructure.

The samples were subject to tensile tests at room temperature using a strain rate of 5. 10-4s-1 (20mm gauge length and 4mm diameter). The cold work level was obtained by interrupting the tensile test at selected total elongation levels. Tensile tests were carried out at 20°C in air at a strain rate of 4.10-4 s-1. The results of the 850°C creep tests under constant load are summarized in Table 3-3 (see also [Ref. 3-5]. The addition of cold work is to significantly decrease the steady state creep rate (i.e. 16/19 down to <1) and to extend the time to rupture. The % elongation is also markedly reduced and the time to reach a given elongation increased. The effect of 10% cold work on creep properties appears beneficial but an increased scatter is observed with the 17% test. At 850°C cold work produces a strong hardening reducing the transient creep stage and reducing strongly the steady state creep rate. The same trend has been observed on Inconel 617 [Ref. 3-4].

Cold work (%)	Specimen	Creep stress MPa	Time to rupture (h)	Elongation %	Reduction of Area %	Creep rate x10^{-5} h-1	Time (h) to reach (X %) of elongation			
							1%	2%	5%	10
0%	**sp 10**	**70**	**330" interupted**			**19**	**30**	**73**	**226**	
	sp 8	**70**	**1306**	**50**	**55**	**16**	**26**	**73**	**234**	**54**
10%	sp4	**70**	**2810**	**6,7**	**9,5**	**0,7**	**700**	**1762**	**2780**	
	sp7	**70**	**2835**	**6,2**	**7,8**	**1**	**225**	**984**	**2420**	
17%	**sp1**	**70**	**interrupte850**			**0,9**	**512**			
	sp11	**70**	**1800h on going**			**0,6**	**1400**			
	sp5	**70**	**916**	**4,2**	**11,3**	**0,6**	**778**	**899**		

Table 3-3: Creep test results at 850°C

The results at 950°C are given in table 3-4. At the higher temperature of 950°C, only the 10% cold work level; seems to have a beneficial effect on steady state creep rate and life time. The 17% cold work tests shows a dramatic drop in the creep resistance and the time to rupture (see Figure 3-2). As a result of these tests it is considered that at 950°C the level of 17% cold work on Haynes 230 should be avoided.

Cold work (%)	Specimen	Creep stress MPa	Time to rupture (h)	Elongation %	Reduction of Area %	Creep rate x10-5 h-1	Time (h) to reach % of elongation		
							1%	2%	5%
0	**COL20-TRA11**	**30**	**1816**	**33**	**40**	**9,5**	**104**		
19	**Flu B9**	**30**	**271,8**	**12,9**	**16**	**7**	**68**		
17	**COL20TRA4**	**30**	**124,6**	**20,9**	**27**	**15**	**52**		
17	**COL20TRA5**	**30**	**207**	**18,1**	**30,3**	**12**	**65**		
10	**COL20TRA13**	**30**	**1395**	7	**12,5**	**1,2**	**563**		

Table 3-4 : Creep test results at 950°C

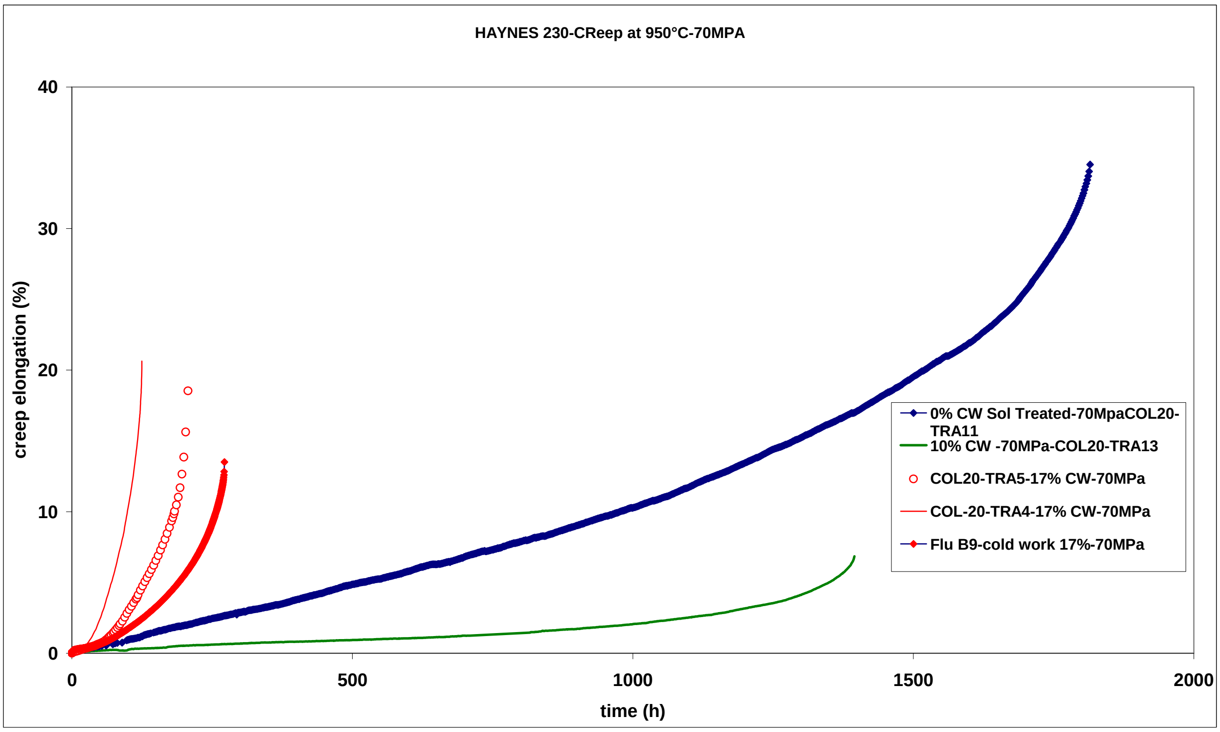


Figure 3-2: Strain-time curves for Haynes 230- creep tests 950°C-30MPa.

With regard to microstructure, the cold worked specimens show finer but longer intergranular cracks in the fracture region. Numerous smaller cracks and voids were also located on the grain boundaries (especially at the grain boundary junctions) and are considered easier to grow possibly because of the large amount of intergranular $M_{23}C_6$ precipitates observed. The precipitate size of the 17% cold worked specimen was larger and SEM observations clearly indicated that the10 % cold worked creep specimen presents the better intragranular precipitates size with the lowest density.

EdF test program

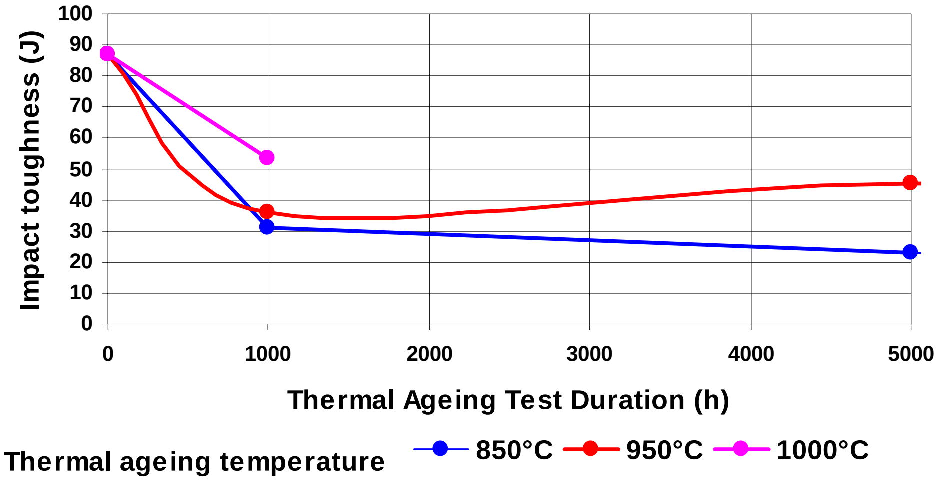
The EdF ageing tests involve the determination of mechanical properties with and without ageing (durations 1000h and 5000h) in the temperature range of 850°C to1000°C. Mechanical tests were performed (tensile tests, impact toughness tests), as well as micro-structural examinations (SEM + TEM). Alloy Haynes 230 material was also procured from Haynes International in the form of hot rolled round bar in the as-received condition. The bar was annealed at 1230°C then water quenched (its diameter being 50.8 mm). The manufacturer supplied chemical composition, which is in agreement with standard Haynes 230 specification but in comparison with CEA material, gives slightly higher contents of chromium, tungsten, molybdenum and iron. Tensile tests were performed at room temperature and in the temperature range of 850°C and 1000°C to cover IHX operating conditions and accident conditions. The results show the tensile properties (yield strength and tensile strength) to decrease more than 50% at 850°C compared with values at room temperature. On the other hand the elongation at rupture increases significantly. These results show an increase in ductility for material at high temperature.

Test temperature (°C)	As-received				Ageing for 1000h												Ageing for 5000h			
					850°C				950°C				1000°C				850°C			
	YS	UTS	A%	Z%	YS	UTS	A%	Z%	YS	UTS	A%	Z%	YS	UTS	A%	Z%	YS	UTS	A%	Z%
RT	415 402	892 892	45 47	52 52	426 391	861 851	18 17	19 19	367 369	861 851	25 26	25 26	357 358	850 845	38 38	37 37	393 405	841 886	16 21	17 18
850°C	161 169	422 447	60 97	47 76	149- 147	414 409	99 110	72 72	-	-	-	-	-	-	-	-	42 142	406 408	97 98	64 63
950°C	-	-	-	-	-	-	-	-	82 82	253 254	140 108	88 87	72 72	238 240	-	75 81	-	-	-	-

Test temperature (°C)	Ageing for 5000h			
	950°C			
	YS	UTS	A%	Z%
RT	355 356	837 839	38 39	40 39
850°C	-	-	-	-
950°C	74 76	236 238	-	75 76

Table 3-5 Effect of Ageing on Mechanical properties.

The results after ageing for 1000h and 5000h are shown in Table 3-5. The effect of ageing for 1000h suggests that there is no strong effect on strength but there is a strong effect on ductility (see also [Ref. 3-5]). The decrease can reach values of more than 50% after 1000h at 850°C but the effect is less significant for ageing at 1000°C. For longer thermal ageing periods, stabilization is observed at 850°C and an unexpected softening at 950°C in addition to the first decrease of ductility. For tensile tests performed at high temperature (850°C), after ageing, a significant increase of ductility is observed compared to as-received state. Charpy tests were also performed at room temperature on two specimens for each condition (as received and aged). The results show a large decrease in impact toughness after ageing of 1000h. Repeating the tests but with a 5000h aged specimen showed a similar result to the 1000h case.



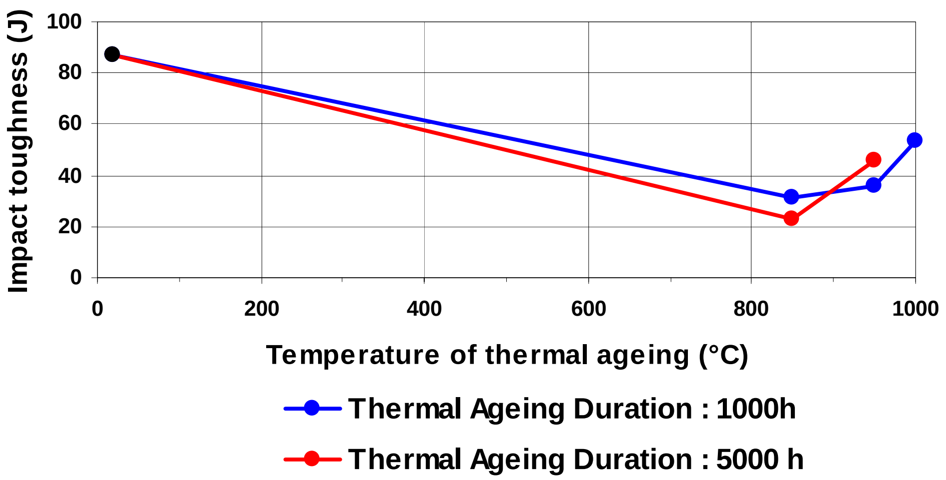


Figure 3-3 Effect of ageing on Impact Toughness.

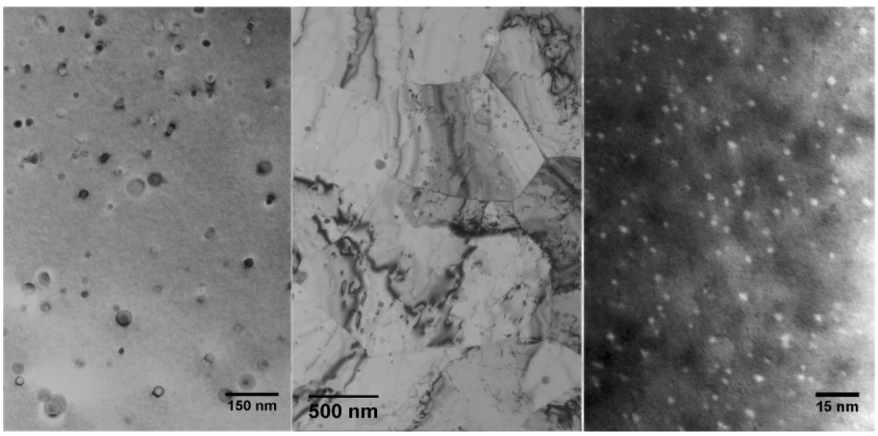
The reduction in the mechanical properties and impact strength for all aged states appears to be linked to a growth in rapid intergranular carbides (type $Cr_{23}C_6$) with a lamellar form observed up to 950°C. The reduction in hardness and the lowest loss of impact strength and lateral expansion at 1000°C in comparison with other aged states could be explained by the change in the form of the intergranular carbides which are predominantly globular (according to observations). Nevertheless, it is likely that properties such as impact values may not be stabilized after 5000h of ageing and have to be investigated further for longer exposure times.

3.3 Procurement and tests on advanced high temperature alloys

Work was performed with different types of oxide dispersion strengthened ferritic steels and a TiAl material. The commercial ODS PM2000 [Ref.3-6] in annealed condition and in severe plastically deformed (SPD) condition, as well as the Japanese development 19Cr ODS (K1) [Ref. 3-7] were investigated and the main characteristics of these three materials identified Table 3-6. Both qualities of PM2000 had comparable dispersoid sizes and distributions. In the annealed condition very large and elongated grains were present allowing the investigation of single crystal properties. The SPD material had equiaxed grains of similar size as the ODS 19Cr grains (see Figure 3-4).

Material	Dispersoid diameter [nm]	Grain size [nm]^3	Cr [wt%]	Al [wt%]	Ti [wt%]	W [wt%]	Y_2O_3 [wt%]	Fe
PM2000 annealed	28.0	10^6x10^6x(>10^7)	19.0	5.5	0.5	-	0.5	Bal.
PM2000 SPD	28.0	500^3 (average)	19.0	5.5	0.5	-	0.5	Bal.
19 Cr ODS	2.1	200x200x700	18.4	-	0.3	0.3	0.4	Bal.

Table 3-6 Details of ODS samples investigated



PM2000 annealed PM2000 SPD 19 Cr ODS

Figure 3-4: Microstructure of the investigated ODS samples

Another set of experiments were performed on the γ-TiAl based intermetallic alloy ABB-2 [Ref. 3-8]. ABB-2 is a two-phase γ-TiAl which consists mainly of grains with lamellar microstructure but also of grains with other duplex microstructures. The two phases are a γ-phase (TiAl-phase with L10 crystal structure) and α2-phase (Ti3Al-phase with D019 crystal structure). The alloy also contains β-phase (TiAl phase with B2 crystal structure) and Ti5Si3-precipitates. More detailed descriptions of the microstructure can be found in [Ref. 3-8] and [Ref. 3-9].

The chemical composition of the alloy is given in Table 3-7. The alloy was cast as a rod with a diameter of 14 mm. The creep specimens were cut along the axis of the rod by spark-erosion, and sliced into dog-bone shaped samples with thickness of 0.3 mm, length 28 mm and width 8 mm. The samples were then mechanically polished to a thickness of 0.2 mm. The final sample had a gauge volume of 10×2×0.2 mm3.

Element	Ti	Al	W	Si	Fe	O	H	N
at%	Balance	46.11	2.02	0.48	0.017	0.168	0.040	0.093
wt%	Balance	30.26	9.00	0.32	0.044	0.077	0.002	0.009

Table 3-7: Chemical composition of TiAl investigated

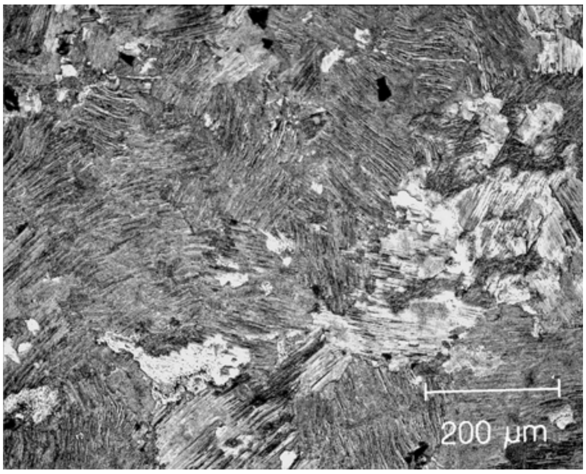


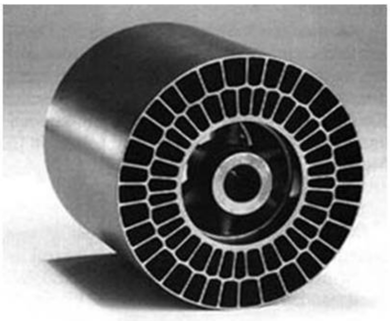
Figure 3-5: Microstructure of TiAl investigated

The choice of the TiAl was based on the good behaviour of this material at high temperatures. The reasons why these materials were not considered for (V)HTR applications might be the following:

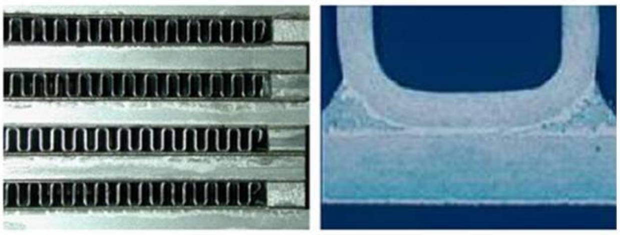
- Not included in German HTR materials database because they were not available at that time
- Usually expected to contain B, or to be on Ni-base for high temperature applications
- Usually expected to need an expensive powder process

- TiAl is usually not considered for T > 800^{o}C because of low oxidation resistance in gas turbine environments
- Not considered for fusion applications (because of the presence of the Al)

The reasons why this material was included in the present considerations was because despite its favourable high temperature properties they also offer the possibility of casting which might be an interesting option (as shown in Figure 3-6) where an original ABB (COMPREX) turbocharger rotor design (cast) is compared with elements for a plate-type heat exchanger.



COMPREX Turbo Charger (cast)



One possible realization of an IHX (AREVA) (sheet forming, fusion)

Figure 3-6: Comparison of a cast turbocharger rotor with a "built" IHX construction

Creep testing
Creep tests were performed in a vacuum and both the applied stress and the temperature were kept constant during each test. The temperatures ranged from 700˚C to 900˚C and the stresses from 100 to 300 MPa. The strain of the specimen was measured by several variable displacement transformers (LVDT) and the temperature was monitored with thermocouples. Three LVDTs were used to measure the strain, two which were fixed to the upper sample clamp and one which was fixed to the lower sample clamp. This allowed for a precise measurement of the strain. The data acquisition and the data processing were performed using a lab-view based computer program.

In situ irradiation creep under He-implantation was performed using the compact cyclotron of Forschungszentrum Juelich. Details of the experimental set up are described in [Ref. 10]. The 0.1 mm thick samples were 3D-homogeneously irradiated under constant uniaxial stress. Irradiation was performed with 24 MeV 4He++ ions passing through a magnet scanning system and a degrader wheel with 24 Al-foils of variable thicknesses. More details on the experimental conditions can be found in literature [Ref. 11]. A technical paper on creep results from miniaturized samples is also given in [Ref. 12].

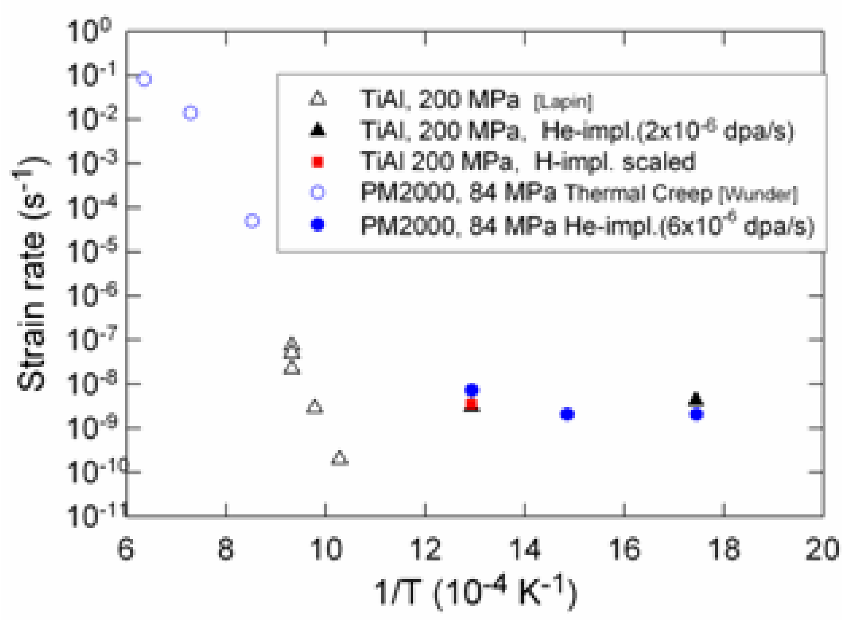


Figure 3-7: Creep strain rates as a function of temperature showing thermal and irradiation creep

Thermal and irradiation creep

Creep problems in nuclear plant are reviewed in [Ref. 13]. Materials under an irradiating environment undergo two types of creep: thermal creep and irradiation creep as can be seen from Figure 3-7. At temperatures above about 600°C the expected Norton-behaviour is observed. At temperatures below 600°C the irradiation leads to deformation which is almost independent of temperature.

Thermal creep life can be characterized with stress rupture curves and the Monkman-Grant rules (see figures 3-7 and 3-8).

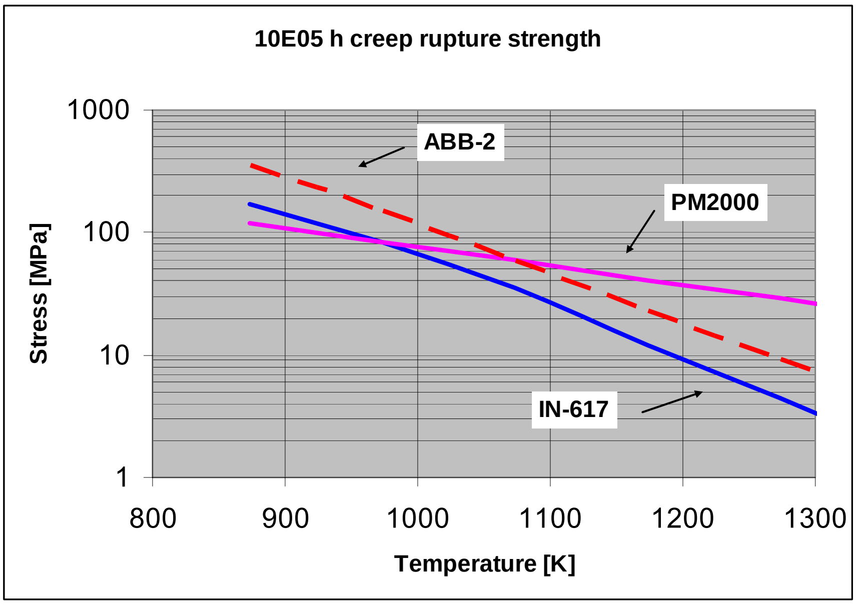


Figure 3-8: Stress rupture behaviour of PM2000 and TiAl in comparison with IN-617

For experimental studies of irradiation creep, often ion implantation of thin tensile samples in accelerators is used. Irradiation creep experiments in an accelerator were performed with two qualities of ferritic ODS steels with He-ions. One material (19Cr ODS) came from a Japanese laboratory. This material had an average dispersoid diameter of only 2.2 nanometer (see Figure 3-4) . The other material was the previously mentioned PM2000 in the annealed condition with an average dispersoid diameter of about 25 nm.

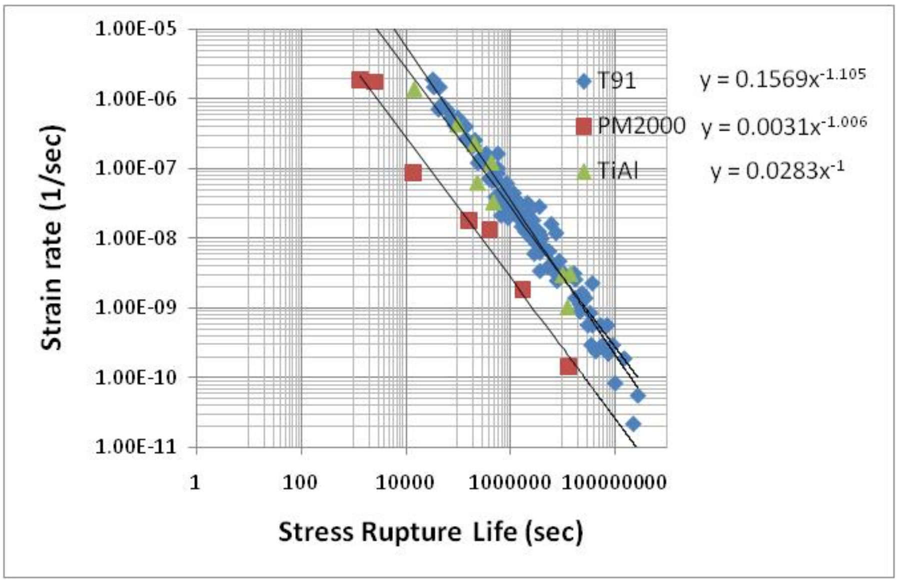


Figure 3-9: Monkman-Grant relationship for different materials

This significant difference in dispersoid size led to expectedly large differences in yield strength for the two materials and also significantly better stress rupture behavior for the ODS materials with small dispersoid diameters. Irradiation creep is frequently described in terms of the irradiation-creep compliance, C. This quantity is related to creep strain rate, ε*, stress, σ, and dose rate, dpa, by the following relationship:

$$C = \varepsilon^* / (dpa.\sigma)$$

As shown in the literature [Ref. 14] only a very small difference in the compliances for PM2000 and Cr19 ODS was found in a temperature range from 300 C to 500° C. This was surprising because it clearly states that the dispersoid size is not important for irradiation creep. It is therefore reasonable to assume that irradiation creep is a matrix property and several ferritic and ferritic-martensitic materials show a comparable irradiation creep behavior. An attempted was made to carry out a joint evaluation of the data on ODS materials with literature data from in-reactor creep tests. In these tests pressurized tubes from the ferritic steels H9, Grade 91 and 21/4 Cr 1Mo were irradiated. Hoop stress and hoop strain were determined. The results are shown in Fig. 3-10.The irradiation-creep compliance was plotted as a function of the dose (dpa). The curve shows the expected shape referring to high irradiation creep rates at the beginning reaching a saturation stage after a few dpa. Taking into consideration that different irradiation conditions, different materials, different sample geometries and different loading conditions (hoop stress vs. tensile stress) are compared, a good agreement is found. In other words, only a small influence of the dispersoids seems to exist for irradiation creep. The high number density of nano-particles may, however, improve the tolerance to neutron irradiation damage by providing efficient sinks for trapping point defects and nuclear reaction products such as helium. The irradiation creep behavior of the TiAl appears to be very close to the ferritic materials.

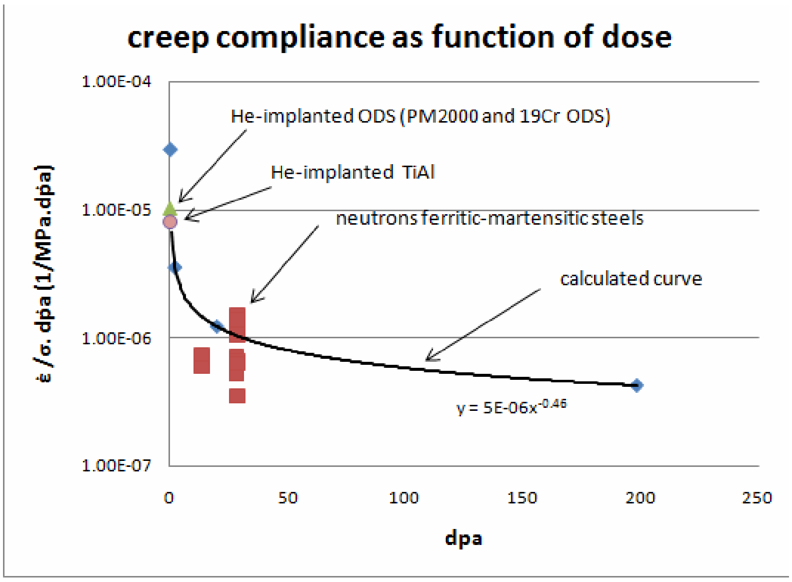


Figure 3-10: irradiation creep compliances as a function of dose

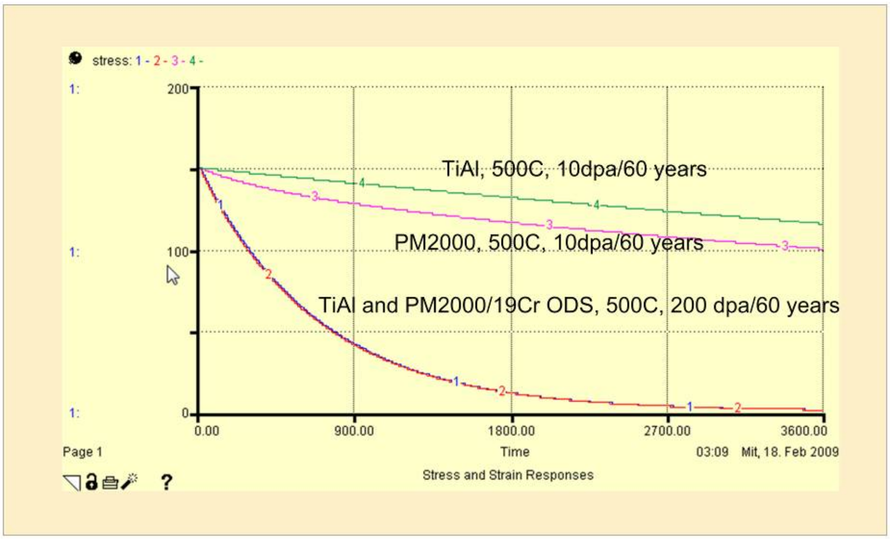


Figure 3-11: Calculated stress relaxation curves under irradiation for different materials

For an illustration of the technical relevance of irradiation creep, the expected relaxation times for the different materials were calculated u at 500°C sing stress rupture and Monkman-Grant data as well as the irradiation creep compliance (see Figure 3-11). It can be seen that assuming a total dose rate of 10 dpa /60 years the thermal creep is still dominant. At a dose rate of 200dpa/60 years stresses equally relax in all investigated materials.

3.4 Results on Creep behaviour of Carburized Udimet 720

This work was started in HTR-M & HTR-M1 5th Framework contracts and has been completed within the RAPHAEL activities at JRC [Ref. 15]. The report summarises the results of a candidate material for a turbine disc for a direct cycle HTR. The work was continued within JRC under a Safety Institutional action. Tests and metallographic investigations have been performed on very heavily carburized Udiment 720 alloy which showed a densely carburized layer of 700µm. The results show that the addition of the carburized treatment shows a marked loss in creep strength and an increase in minimum creep rate. The loss in creep strength is mainly associated with grain growth and micro-cracking and the dissolution of tertiary and coarsening of γ particles. From the tests performed the reduction in creep rupture life appears to be of the order of a factor of 2 to 3 on life compared with as-received material.

3.5 Support activities to SP CT & Feedback from SP SI

An important part of the SP4 ML metals activities was to provide materials support to the RAPHAEL components work performed in SP5 CT. This has included the provision of materials information on IN 617 to support structural integrity assessments in evaluation of the IHX development. Translation of material property values was provided from the KTA Design

Code [Ref. 16]. Other support activities on metals included provision of material information for the impellor of the Circulator and advice with respect to the welding of IN 617 steam generator tubing. SP ML partners also gave presentations at joint SP ML/ SP CT meetings on corrosion and other issues. Requests were made from the System Integration activity (SP-SI) for information to support the experiments on metals (vessel and IHX) for which feedback was given at SP SI meetings and via a number of joint SP ML / SP CT meetings.

3.6 Status of the activities of the Metals Working Group of GIF

The Metals Working Group (MWG) is a forum established within the Generation IV VHTR Materials Project management Board (PMB) to coordinate details of the national metals R&D programmes in support of VHTR. The work programme involves data acquisition and design methodology. Its coordination activities include: collaborations; sample exchanges; corrosion and environment effects, Design Codes, Micro-structural modelling, fabrication and process development, review of collaboration plans and assessment of data produced by participating nations (or their subordinate organizations) within the framework of the GIF Materials PMB. The MWG will also review the scope of national R&D programs and recommend additions to close any identified data gaps.

Metalllic Materials Task	EU involvement
TASK1.1 - Data generation- Reactor Pressure Vessel materials	Yes
TASK1.2 - Data generation- medium and high temperature alloys	No
TASK1.3 – Data generation – very high temperature alloys	Yes
TASK2 – Database generation and maintenance	Yes
TASK3 – Corrosion and environmental effects	Yes
TASK4 – Nuclear design and Codes and Standards	Yes
TASK5 – Microstructural Modelling	No
TASK6 – Fabrication and process development	No

Table 3-8 Metals Working Group Tasks

Euratom members of the GIF VHTR Materials PMB are as follows:

D. Buckthorpe (AMEC) - PMB Member
R Hurst (JRC) – MWG Member
D Buckthorpe (AMEC) – MWG Member

The tasks to be addressed by the MWG are listed in Table 3-6. The RAPHAEL materials partners have agreed that all the results from the RAPHAEL materials subproject can be used as part of the Euratom contribution to Generation IV programme, hence the results from the activities summarised in this report will be submitted as part of the Euratom contribution to the MWG. Exchange of information is to begin after all Generation IV Partners have signed a

Project Arrangement which details a five year programme of collaboration for the period 2007 – 2012. JRC are the signatories on behalf of Euratom. Details of the Euratom (labelled JRC) contributions and deliverables to be submitted to the MWG are given in [Ref. 17].

3.7 Overall Conclusion and Recommendations on High Temperature Metals

The status of the activities of the Metals Working Group of GIF has been summarised also the exchanges with SP CT and SP SI and a summary of the findings within WP2 metals reported [Ref. 2-14]. The work completed for high temperature metals within Work package 2 of RAPAHEL SP4 ML includes:

Investigations of the limits of High Temperature Materials

The available materials and their limits for the key high temperature components were reviewed;

- The most promising available candidate materials were identified as IN 716 and Haynes 230.
- Recommendations made for future development of some new alloys more adaptable to the VHTR upper temperature limit requirements.

Procurement and tests on selected material Haynes 230

A test program was performed to examine high temperature mechanical properties and micro-structural changes for the Haynes 230 alloy:

- The effects of cold work was investigated at 850°C and 950°C and for the latter the level of 17% cold work on Haynes 230 should be avoided. Further testing is needed on a larger number of samples in order to provide conclusive evidence on behaviour.
- The effect of ageing for 1000h suggests that there is no strong effect on strength but there is a strong effect on ductility.
- For longer thermal ageing periods, stabilization is observed at 850°C and an unexpected softening at 950°C
- Charpy tests at room temperature on two specimens for each condition (as received and aged) show a large decrease in impact toughness after ageing of 1000h which is not significantly changed after 5000h

Procurement and tests on advanced high temperature alloys

Tests on advanced alloys for HTR application has:

- Compared the creep behaviour of ODS material covering thermal creep and irradiation creep and shown that under certain conditions irradiation creep is important..
- The irradiation creep behaviour of TiAl has been investigated and compared with other high temperature materials
- From the corrosion tests performed on Udimat 720 the reduction in creep rupture life appears to be of the order of a factor of 2 to 3 on life compared with as-received material.

3.8 High Temperature Metal Material References

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4 Summary of SP4 Work package 2 on Composite Materials

Carbon Composites are candidate materials for some of the structural parts of the VHTR because of their attractive properties, such as high strength and Modulus, thermal shock resistance and stability at high temperatures. Some of the main structural components that could be manufactured with these materials include the control rods, the hot pipes, core straps and supports. Part of the RAPHAEL WP2 materials investigations includes an evaluation of the potential of such materials for these applications with special emphasis on the Control Rod. The work performed is summarised in the following sections.

4.1 Specifications for the control rod and for other applications

A specification was prepared based on past information defined within the HTR-M and EXTREMAT projects [Ref. 4-1, 4-2, 4-3] and additional information provided by RAPHAEL partners. For the Control Rod this covers a description of the application, environmental conditions, material composition, electrical and mechanical properties, stability and product characteristics [Ref. 4-4]. A brief summary of the findings is given below:

Control rods

Description:

The Control rods consist of several segments connected to each other, containing the absorber material. Each control rod assembly is supported by mechanisms in the top head of the reactor. The materials envisaged must support boron carbide compacts. Two systems are considered:

- Control rods located in the side reflectors at the boundary of the core. These are inserted to varying heights for control during operation and completely inserted for protection.
- Automatic control rods used only during start up/shutdown and refueling operation, located in the active core at the boundary with the inner reflectors. They are completely removed when the core is critical.

Environment

The gas environment under normal and accident conditions has been summarized for start-up/shutdown and operating control rods under normal and accident conditions [Ref. 4-3]. Under normal operating conditions the temperature range of interest is 400°C < T < 1100°C with temperatures at the lower end for the start-up/shutdown control rods and the gas impure helium. Under air ingress accident conditions the temperatures may be as high as 1500°C and the fluid medium is air. The maximum permitted fluence level (for exchangeable control rods) is 4dpa with the expected plant life duration of 30 years.

Composition

The impurity levels within the materials have to be controlled to limit the radioactivity of the material after irradiation (to minimize waste management.) and to prevent excessive oxidation.

The highest purity level will have to be chosen for HTR applications. A summary of allowable levels is given in [Ref. 4-4].

Product characteristic
Existing control rods are made up of two concentric tubes with absorber materials in between. The outer tube diameter ranges from 100/120 mm and the inner tube diameter 60/70 mm. Their thickness is between 4 and 8 mm. The length of one segment is between 400 and 800 mm. The space inside the rods is used for cooling. The total length of the rod is about 9 m.

The fabrication process used must be reproducible and provide material that does not vary significantly between batches and lots. A proposed GT-MHR arrangement is shown in Figure 4-1 below:

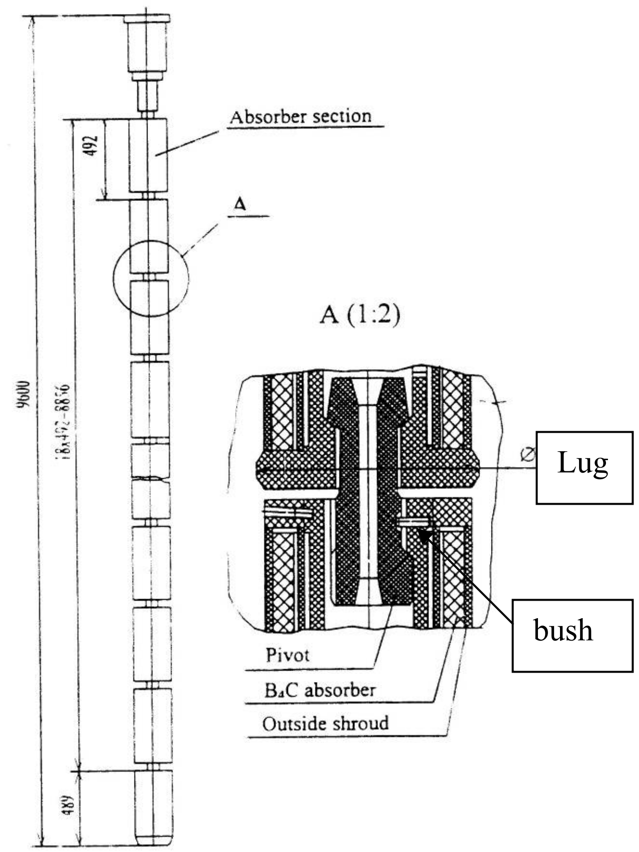

Figure 4-1 Schematic of VHTR C/C Composite Control Rod

Electrical and mechanical properties
A list of electrical and mechanical property requirements has been provided in [Ref. 4-4]. The mechanical strength required is greater than 70MPa temperature and under irradiation. Thermal conductivity values should be in excess of 20-30 $W.m^{-1}.°C^{-1}$.

Stability

The criterion for wear resistance has not yet been established. No criteria has been specified at the moment for creep resistance under impure helium (T°~1000°C, ~5MPa, 10-30 years). Dimensional changes after irradiation should be < 2% for each direction and after a dose of 4dpa. No criteria are available for air reactivity under accident conditions. The material must be compatible with Boron Carbide.

Other applications

Some other components such as straps or pipes could be made with composite materials. The requirements for pipes should take into consideration previous work undertaken in the German HTR programme [Ref. 4-5, 4-6 & 4-7]. For straps the requirements are not well developed however temperature and fluence levels will be much less than for control rods.

4.2 SGL Recommended materials

Carbon composites have to be tailored for each application to meet design requirements with the manufacturing parameters adjusted to fulfil the technical requirements and to match the component geometry. Optimization of suitable materials requires adjustment of the fibre architecture and matrix material and therefore requires the co-operation and involvement of the manufacturer.

Potential materials

2D composite materials have been proposed by SGL with suggestions for the matrix composition and fibre system provided in [Ref. 4-4]. 3-D composites are also of interest (although more expensive) since they have no weak orientations and are more isotropic in behaviour. Pitch and/or PAN based fibers are recommended for the tubes and connecting elements. The suggested matrix system is resin/pitch matrix and/or CVI matrix. It is also recommended that the finished parts are purified (less than 20ppm) and SiC coating is used for improvement of the oxidation properties. The proposed manufacturing characteristics are given in [Ref. 4-4].

Impurity levels

The impurity levels should be controlled by chemical element analysis of the material. SGL composite materials contain a very small amounts of ash.

Material properties

Available requirements are given in [Ref. 4-4]. Values are given for density and porosity. The information on strength (requirement >70Mpa) and thermal properties is to be defined later.

Feasibility and availability of supply

The manufacturing of cylindrical structures such as control rods is considered easy to perform by filament winding according to the specified geometry. No cost evaluation can be made at the present time, as this strongly depends on the fibre lay-up architecture and manufacturing method.

At the current stage (and most probably in the future) it will be extremely difficult to ensure the raw material suppliers. The supply of the material will ideally need to be guaranteed for more than 15 years (i.e. the same grade of raw materials (fibre type, pitch grade, etc.) and the same manufacturing processes will need to be used during material qualification and final components manufacturing). Currently however (and most probably in the future) it is thought that given the specific but small amounts of material grades needed it will be extremely difficult to ensure that raw material suppliers are in compliance with this.

4.3 Material test programme

Recommendations are made with respect to testing with suggestions on sample size, testing procedure, key issues and a draft test programme [Ref. 4-4]:

- Both flat and round samples are proposed with advice on size effect given
- Issues concerning Standards, reproducibility, dispersion, anisotropy are summarised
- No standards are currently available for tests on tubular structures and some advice is given
- The main issues for the R&D program on composite materials for control rods have been identified namely:
    - irradiation effects at high temperature (e.g. 4.3 1021 n/cm² (or 3.44 dpa) within 30 years)
    - oxidation resistance at high T° (under impure He or air for air ingress accident)
    - component design
- The draft test programme is to concentrate on the effects of irradiation (initially via EXTREMAT-IP- whose irradiation experiment has now been extended into 2010) and through the evaluation of corrosion properties (some investigations underway in RAPHAEL) and modelling aspects.
- A specialist meeting was held to discuss the materials and sample selection and post irradiation examination (PIE) requirements [Ref. 4-8]. The type of samples to be irradiated in HFR will be agreed by NRG and SGL (material grade, sample size, specimen number) within the EXTREMAT-IP programme.
- A report was prepared giving general guidelines on the machining of specimens for the irradiation programme. A key recommendation is to make sure that the samples taken from 1D, 2D and 3D composites are machined to take account of the orientation as described in this report. A key point is to maintain the same consistency between each of the specimens [Ref. 4-9]

Description and status of the EXTREMAT-IP irradiation test programme

A link meeting between RAPHAEL SP4 ML Work package 2 partners and EXTREMAT SP3 partners was held in 2007 to discuss practical issues for connecting the work within RAPHAEL on VHTR Composite material development with the planned irradiations within EXTREMAT-IP [Ref. 4-10]. Following this SGL (who are also involved in EXTREMAT) proposed to supply their best available material for this application in time for the irradiation experiments. The progress of the SGL actions was monitored at successive EXTREMAT and RAPHAEL progress meetings. SGL have supplied both 1D and 2D composite material for these experiments and propose to investigate the effect of irradiation on the physical properties of the materials. A brief description of the two EXTREMAT irradiation experiments to be carried out by NRG in HFR is given below.

Two irradiation capsules have been developed providing a lower dose irradiation (1.4 dpa) at 300 & 550oC and a higher dose irradiation (5 dpa) at 600 & 900^{o}C. The first irradiation capsule started in February 2008, this contains samples of dispersion strength 12Cr steels, composite materials including pitch and uni-directional samples and small grain graphites from SGL. The second experiment which was loaded mid 2008, contains ODS steels, tensile and Charpy specimens, graphite and composites to provide information on swelling, CTE and bending strength. SiC samples are also included. It is important that the fibre size and architecture of the CFC's is representative which can be a difficulty with small sized samples.

It is important to realise that these experiments are aimed at screening and not developing design data. This type of experiment is a necessary first step as part of the material optimisation process and before testing on a larger scale and performing some component testing.

Oxidation testing with RAPHAEL SP4 ML

Some initial oxidation tests have been carried out at FZJ and presented at the 6th Progress meeting of RAPHAEL SP4 ML. [Ref. 4-11]. To date, 3D-CVI CFC (SGL) material has been examined at 700^{o}C in air and compared with results for V483T graphite. The 3D-CVI Composite has so far shown a better oxidation resistance than the graphite, the burn-off rate being much lower. Further experiments are needed to evaluate behaviour and to investigate the different composite material options being considered for the VHTR Control Rod.

Its possible that further work is needed as with graphite to investigate the effects of sample size on the results and to consider the need for coatings.

Micro-structural modelling (within EXTREMAT-IP)

To support the development of composites for the Control Rod within the EXTREMAT-IP the micro-structural modelling of composites was undertaken by University of Manchester under a third party agreement with NNC.

Investigations have been carried out using a new predictive capability for advanced carbon fibre reinforced composites using an image-based route for generating finite element (FE) meshes which are used for predicting the un-irradiated behaviour of composite materials The approach can be extended to predict the behaviour of materials under irradiation if the input property data are known at the irradiation dose levels and appropriate images can be obtained. This should, for example, account for any dimensional changes under fast neutron irradiation.

The aim of this study was to illustrate a methodology for modelling composite behaviour based on the actual structure of the composite [Ref. 4-12 to 4-16]. This was achieved by combining X-ray micro-tomography, which gives a high spatial resolution image of the microstructure in 3D, with finite element packages. Here the real structure is converted directly into a 3D FE mesh. Hence the approximations associated with the assumption of an idealisation of the structure are minimised.

4.4 Overall Conclusions on Carbon Composite Materials

The status of the work carried out on Carbon Composite materials within Work package 2 of RAPHAEL SP4 ML for the VHTR Control Rod and other components has been summarised and is reported in Ref. 4-17. The work includes:

Specifications for the control rod and for other applications

- A brief specification for Control Rod application has been given. Other components such as straps or pipes could be made with composite materials

SGL Recommended materials

- SGL composite material manufacturer has made some recommendations with regard to potential materials, impurity levels, properties and feasibility of supply

Material test programme

- Recommendations are made with respect to testing with suggestions on sample size, testing procedure
- The effects of irradiation on selected samples is being investigated using some preliminary testing (in the EXTREMAT-IP). Micro-structural modelling of these materials has also been started within EXTREMAT-IP
- Corrosion tests have been performed on supplied composites and compared with graphite. The composites have shown better corrosion properties

4.5 Carbon Composite Materials References

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5 Summary of SP4 Work package 3 on Graphite Materials

Graphite materials are used for the Core of the HTR and VHTR and part of the RAPHAEL materials programme includes an evaluation of the potential and properties of these materials with special emphasis on their behaviour under irradiation and oxidation. A modelling capability is also being developed to provide the basis for selection and future optimization. The work is performed within SP4 ML WP3. A summary of the Euratom position and contributions to be made within the Generation IV Graphite Working Group (GWG) is also given.

5.1 Graphite Irradiation experiments

The properties of graphite change significantly under fast neutron irradiation and experiments are required to provide data up to the peak fluence expected for a (V)HTR over the appropriate temperature range. The peak fluence appropriate to a pebble bed core design is around 25 displacements per atom (dpa)_graphite. The corresponding value for the replacable components of a prismatic core design should not exceed 1/3 of this value. For a (V)HTR the temperature range of interest is considered to be 600°C to 1100°C. It is recommended that the data be obtained for the selected core graphite at 100°C intervals, but since the fluence at the upper and lower temperatures will be small, the data should be obtained at 100°C intervals between 650°C and 1050°C inclusive. Two experiments are being undertaken in the HFR at Petten as part of the RAPHAEL irradiation experimental investigations aimed at providing results at 750°C and 950°C. Figure 5-1 below gives a schematic of the data range to be expected from the irradiation experiments the 5th Framework Programme (FP5, HTR-M1), blue box in Figure 5-1 and the RAPHAEL-IP, red boxes 2,3 and 4 in Figure 5-1.

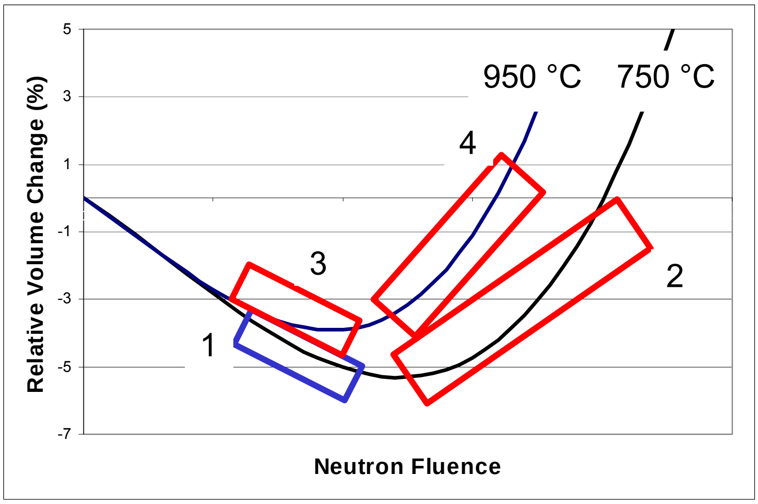


Figure 5-1 Schematic overview of the different irradiation experiments within HTR-M1 and Raphael-IP

It is also known that when irradiated graphite is thermally heated (annealed) above its irradiation temperature there is some recovery in thermal properties due to partial annealing of the irradiation damage. This recovery might be helpful to take into account when making a safety case for a reactor. A heat-up experiment has also therefore been performed to investigate this phenomena and to determine the extent in recovery in properties in samples from the 750^{o}C irradiation.

Graphite Irradiation at 750^{o}C (1 & 2)

INNOGRAPH1A Experiment- (1)

The first stage of the 750^{o}C irradiation programme (INNOGRAPH 1A) has already been carried out in HTR-M1. The INNOGRAPH 1A experiment was irradiated for 14 HFR cycles from February 2004 to April 2005. Approximately 200 small samples covering eight graphites were inserted into HFR for testing. The samples were first pre-characterised, before being irradiated to a maximum of around 1/3 peak fluence, (the peak fluence was set at ~25 displacements per atom (dpa), or ~200 x 10^{20} n.cm^2 Equivalent DIDO Nickel Dose (EDND) at a temperature of 750^{o}C).

For the INNOGRAPH 1A test, five graphites were selected to be tested as major grades. Two of the graphites are produced by SGL Carbon (NBG-10 & NBG-25), two by Graftech Ltd. (PCEA & PCIB-SFG) and one by Toyo Tanso (IG-110) (See Table 5.1). In addition there were three minor grades tested, one from each manufacturer namely (NBG-20 (SGL Carbon), PPEA (Graftech) and IG-430 (Toyo Tanso). The IG110 grade is used in both the Japanese HTTR and the Chinese HTR-10 reactors and was included as a reference material since a significant amount of high temperature irradiation data already exists. Samples were taken at the edge as well as the center of the supplied graphite blocks (to investigate through block variations) and tests performed on specimens containing with-grain and against-grain orientations.

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**

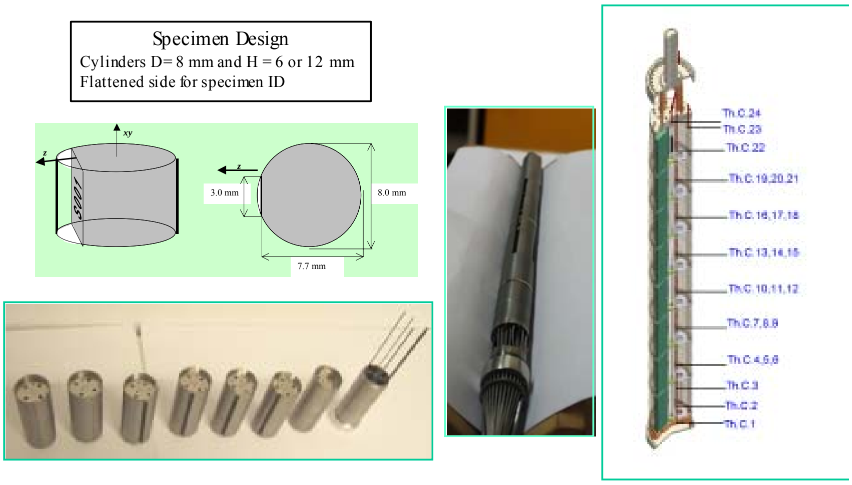
TOYO TANSO	PITCH COKE	PET COKE
EXTRUDED		
ISO-MOULDED	*IG-430*	**IG-110**

Table 5.1: Graphites selected for INNOGRAPH1A

To construct design curves at different temperatures, data points are required as a function of irradiation damage. The dose range within one irradiation experiment is ~65% to 100% of the highest dose attained due to flux differences between the centre and ends of the capsule (i.e. flux buckling). Thus an irradiation experiment up to the maximum dose of interest (~25 dpa_g) would only give information between ~16dpa_g and 25dpa_g. which is not sufficient for a design curve. It was decide therefore to have two irradiation experiments, the first one targeted at a third of the maximum dose and the second to cover two thirds of the maximum dose. This second experiment would also contain fresh graphite samples plus irradiated graphites from the first experiment. In this way data will be generated at 1/3, 2/3 and 3/3 of the end of life dose, points between these dose levels will be obtained by making use of the flux buckling.

The INNOGRAPH 1A test rig comprised a series of capsules with provision for four columns of samples in each, and each drum was then inserted in the test rig before being finally seal welded (see Figure 5-2). The INNOGRAPH1 A experiment was loaded in HFR and irradiated up to ~10 dpa, or ~70 x 10^{20} n.cm^{2} EDND which is very close to the target value. (This should be sufficient data at this temperature for a prismatic design.) Shortly after INNOGRAPH 1A started it was noticed that two of the capsules were only running at ~670^{o}C. A small number of samples have therefore been irradiated at a temperature that is close to the lower temperature value of interest (650^{o}C) and so will provide useful data. This will hopefully remove the requirement for a separate experiment at a lower temperature.

Details of INNOGRAPH1A



4 stacks of 60 mm stacking height per drum

Figure 5-2 INNOGRAPH1A Experiment

Screening results from the INNOGRAPH1A experiment are reported in [Ref. 5-1] and shown in Figure 5.3 below, and the design, loading and assembling of the experiment is summarized in [Ref.5-2]. The screening results suggested that turn-round had not yet occurred although this can only be confirmed after a full post irradiation examination (PIE) of the specimens. The peak dose from the INNOGRAPH1A experiment was 9.9dpa_g.

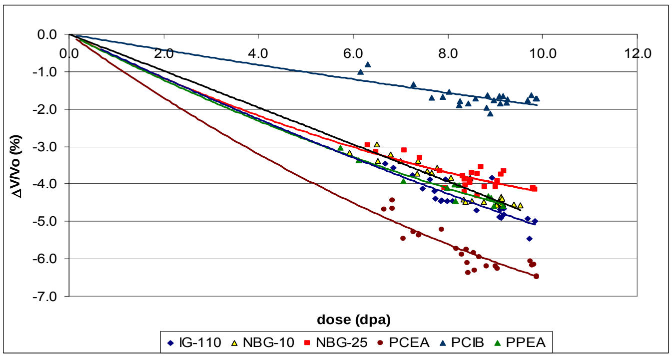


Figure 5-3 Preliminary Screening results from INNOGRAPH1A

Prior to inserting irradiated samples into the follow-on experiment (INNOGRAPH1B), 80 of the samples were fully examined and their condition recorded and used as a basis for assessing the changes introduced by the INNOGRAPH 1B experiment.

The full PIE results for the INNOGRAPH 1A experiment have also been carried out within the RAPHAEL programme and the results are reported in [Ref 5-4]. They cover the complete PIE of the 750°C first phase experiment between 5.5 and 9.9 dpa_g. Results are given for dimensional change, dynamic Young's modulus, coefficient of thermal expansion coefficient and coefficient of thermal diffusivity and conductivity. For the volumetric and dimensional changes it was confirmed that all the samples from all the grades were in the shrinkage phase. The high dose samples approaching turn-round but did not pass this point. PCEA shows the highest shrinkage rate and PCIB the lowest. NBG-20 has the highest shrinkage rate. The grain directions (WG and AG) have a significant influence on the length change with anisotropic behaviour clearly observed. The differences between the two grain orientations tends to be larger for the iso-moulded grades compared to the extruded grades. Dynamic Young's modulus initially increases upon exposure to neutron irradiation up to a plateau value before increasing again at medium dose values. The results from the experiment shows this second increase in value with the lower dose values giving a good indication of the first increase. The highest Eirr to Eo ratio was found to be 2.9 for the IG 430/AG samples. For the coefficient of expansion (CTE) the values increase slightly at low dose and then start to decrease. The results within the INNOGRAPH 1A experiment are beyond the initial increase and in the slight decrease phase. The highest CT_{Eirr} to CT_{Eo} value was 1.3 for NBG-25 (at the low dose end) and the lowest 0.5 (PPEA) at the high dose end. There are no significant differences with grain direction. For the coefficient of thermal diffusivity (and conductivity) there is a rapid decrease upon exposure to neutron irradiation before the rate of change flattens and a plateau is reached at medium to high dose. This plateau can be seen in the results of the INNOGRAPH 1A measurements. Results were obtained over the range 25 to 700°C using the laser flash method which showed the thermal diffusivity (conductivity) ratios were reduced to 0.5 and 0.2 at 700°C and 25°C respectively. For these measurements the specific heat is assumed to be the same before and after irradiation.

INNOGRAPH1B Experiment – (2)

The second stage of the 750°C irradiation programme (INNOGRAPH 1B) was carried out in the RAPHAEL and its status is reported in [Ref 5-5 to 5-7]. Details of the procurement and manufacture of the test pieces is given in [Ref. 5-3] and information on the status of the experiment at the end of the project is given in [Ref. 5-4]. The test rig for this experiment had three columns of graphite samples within the capsules rather than four in order to accommodate the expected re-expansion of the irradiated samples. For the INNOGRAPH 1B experiment both fresh and irradiated graphites were loaded into the test rig. A special transport device had to be designed and constructed to move the INNOGRAPH 1B device with irradiated samples to HFR (see Figure. 5-4). The irradiated samples were loaded into the test rig using the shielded facilities available at NRG (Figure 5-5). Again the extra drum at the top of the experiment was targeted at a lower temperature 680°C as occurred in INNOGRAPH 1A in order to obtain a comparable set of lower temperature results.

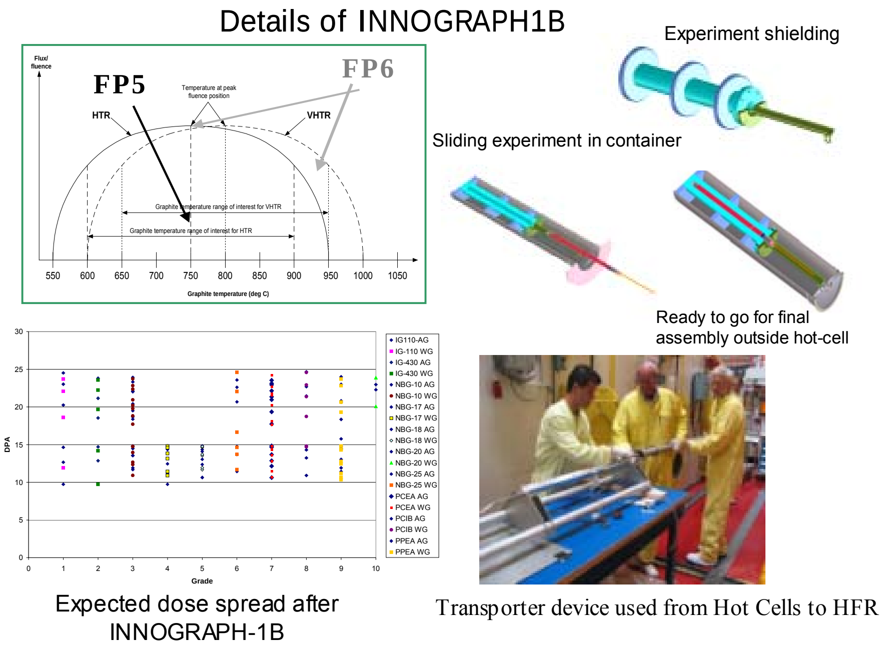


Figure 5-4 INNOGRAPH 1B Experiment

The major graphite grades for INNOGRAPH1B experiment were from SGL (NBG-10 & NBG-18) and Graftech (PCEA & PPEA). The minor grades included NBG-17 and PCIB as well as the two Toyo Tanso graphites (IG 110 & IG 430) (see Table 5-2). As with INNOGRAPH 1A, the samples were taken at the edge as well as the centre of the same supplied graphite blocks and tests performed on specimens containing with-grain (WG) and against-grain (AG) orientations.

Grade	Manufacturer	Coke	Process
PCEA	GrafTech	Petroleum	Extrusion
PPEA	GrafTech	Pitch	Extrusion
NGB-10	SGL	Pitch	Extrusion
NBG-18	SGL	Pitch	Vibro-moulding

Major Graphites

Grade	Manufacturer	Coke	Process
PCIB-SFG	GrafTech	Petroleum	Iso-moulding
BAN	GrafTech	Needle	Extrusion
NBG-25	SGL	Petroleum	Iso-moulding
NBG-17	SGL	Pitch	Vibro-moulding
IG-110	Toyo Tanso	Petroleum	Iso-moulding
IG-430	Toyo Tanso	Pitch	Iso-Moulding

Minor Graphites

Table 5.2: Graphites selected for INNOGRAPH 1A

The irradiated samples were loaded into INNOGRAPH 1B using manipulators the assembly sequence is summarized in [Ref. 5-4 & 5-5]. Final assembly of the experiment was done outside the hot cell, with the extension to the upper section of the experiment welded on to form the top lid of the containment. Once the assembly was complete, tests were performed with respect to overall length, gas tightness and to check the resistance of the thermocouples before inserting into HFR.

Details of INNOGRAPH1B- Loading of irradiated graphite plus Assembly in Hot Cells

Figure 5-5 INNOGRAPH1B Assembly in the Hot Cells

This was the first time such an experiment has been performed and its realization and successful assembly is a significant milestone and result for the RAPHAEL graphite irradiation programme. This approach of using two experiments to obtain a full design curve is a major achievement that will make it possible to produce design curves from a reduced number of samples and serve to reduce the effects of material scatter to within an individual block. The method will allow a more accurate description of block material variability to be made and a more accurate design curve to be constructed.

The INNOGRAPH 1B test was loaded into HFR on 23rd August 2007. To achieve its target dose of ~14.4 dpa around 24 cycles was needed (approximately 0.6 dpa_g per cycle). Unfortunately due to three force majure events, the first involving a fault in the HFR Hot cells whilst the INNOGRAPH 1B experiment was still inside (this lead to a delay of six months before the test rig could be removed). The second was a refit of the HFR core which meant that the number of cycles required to achieve the target dose would increase (by around 25%) and the third delay came as a result of a shut down of HFR in August 2008 (until February 2009) following the observance of an unexplained feature in the bottom of the HFR primary cooling system. An extension of the project programme (of 12 months) was approved by the RAPHAEL Steering Committee following the first and second events, to allow as large as possible a dose to be achieved and except for the final few months, the experiment spent practically the whole of the 5th year in the reactor to accumulate as high a dose as possible. However because of a further temporary shutdown of HFR for 6 months, the final dose level was slightly short of the target at around 14 dpa. The time spent in the HFR meant however that it was not possible to perform the screening PIE for this 750^{o}C phase 2 experiment within the project timescales. The screening PIE will be delayed until later in the year when NRG will be able to complete the screening results for INNOGRAPH 1B in the autumn/ winter of 2010.

Design curves at 750^{o}C

From the results of the above two experiments the curves were established for the test samples investigated in the experiments [Ref. 5-8]. These curves were constructed using the full PIE results from the first phase experiment only since the second phase screening results were not available for the end of the project.

An assessment of the results from the INNOGRAPH 1A experiment showed some similarities between the different graphites in dimensional change behaviour. The extruded graphites however shrink at a faster rate than the iso-moulded graphites in both directions. The iso-moulded graphites exhibit a higher anisotropy and also have a higher scatter in the data. Although some exhibit similarities up to the maximum fluence achieved, it is unlikely they will exhibit similar behaviour beyond turn-around. This will only be known after the INNOGRAPH 1B results have been assessed. The DYM variations are as expected and completely consistent with those for previously irradiated graphites. Comparisons in dimensional change behaviour for NBG-10 and PPEA with a previously irradiated graphite, ATR-2E, shows that both NBG-10 and PPEA have higher shrinkage rates than ATR-2E, although turnaround will occur at a higher fluence. NBG-10 and PPEA also show less anisotropy but more scatter. The DYM behaviour is very similar over the fluence range covered.

Graphite Irradiation at 950^{o}C (3 & 4)

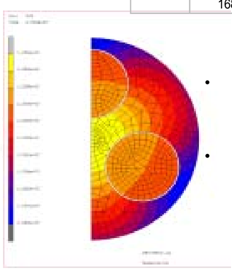
INNOGRAPH 2A Experiment- (3)

In addition to the 750^{o}C data, further data was obtained at 950^{o}C, representative of the higher temperature VHTR condition. Again two irradiation experiments were used to obtain the full design curve for this nominal temperature [Ref. 5-9 to 5-11]. The first irradiation experiment was targeted at a third of the expected maximum dose (for this temperature) and the second to cover two thirds of the maximum dose and to contain fresh graphite samples plus irradiated graphites from the first experiment. The target maximum dose was estimated as ~16 dpa_g. The INNOGRAPH 2A experiment was therefore exposed to around 6 dpa_g and the INNOGRAPH 2B experiment the remainder. Both experiments use the same design of capsule as INNOGRAPH 1B (i.e. three columns of graphite samples within the capsules) because the higher temperature would result in earlier shrinkage turnround. Also, because of the benefits of the data at the lower temperature in the INNOGRAPH 1A experiment, it was decided to run the same capsules at a temperature ~100oC lower, i.e. at ~850°C in the INNOGRAPH 2A and INNOGRAPH 2B experiments. Again, although limited, the data should provide a useful indication of the behaviour at this lower temperature.

The INNOGRAPH2A irradiation experiment was carried out (in position C3 of HFR) between July 2006 and June 2007 (cycles 2006-6 to 2007-4). The rig was loaded in a TRIO capsule which allows three rigs in one irradiation position. The major graphite grades for INNOGRAPH 2A experiment were from SGL (NBG-10 & NBG-18) and Graftech (PCEA & PPEA). The minor grades included NBG-17 and PCIB as well as the two Toyo Tanso graphites (IG 110 & IG 430) (see Figure 5-6). In comparison with the INNOGRAPH 1A experiment two new grades: NBG-18 a pre-pilot production for PBMR and NBG-17 a similar but two times smaller filler grain that would make the graphite more suitable for fine details such as prismatic blocks were included. The NBG-17 grade is being considered a suitable candidate for ANTARES. Both graphites use the vibro-moulded process. As with INNIOGRAPH 1A & 1B, samples were taken at the edge as well as the center of the same supplied graphite blocks and tests performed on specimens containing with-grain and against-grain orientations.

Details of INNOGRAPH2A

grade	6 mm	12 mm	WG	AG
NBG-10	28	4	14	14
NBG-25	12	0	6	6
NBG-18	28	4	14	14
NBG-17	12	2	7	7
PCEA	28	4	14	14
PCIB	12	4	8	8
PPEA	28	4	14	14
IG-110	7	0	3	4
IG-430	5	0	2	3
LPEB	6	0	3	3
LPIB	2	0	1	1
	168	22		



- Based on INNOGRAPH1A design
  - Three column design
  - Design temperature 950°C
  - Extra drum at top at 850°C
- Irradiation started July '06

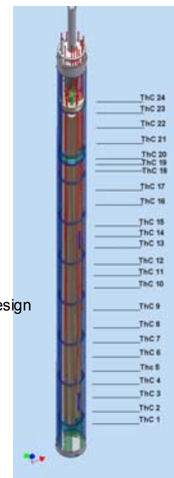


Figure 5-6 INNOGRAPH2A Experiment

Prior to loading into the INNOGRAPH2A capsules, all the specimens were fully characterized. The INNOGRAPH 2A test was loaded into HFR on July 2006 and ran for ~10 cycles finishing in April 2007. After a period of cooling a preliminary screening of graphite behaviour was carried [Ref. 5-9] plus full PIE of 80 specimens (to be reloaded in INNOGRAPH 2B). Some of the specimens were found to have unexpected high activity caused mainly by Mn-54 (which has a half-life of around ~ 313 days). The activity was randomly distributed over the grades and the rig, however there was enough low activity specimens available for reloading into INNOGRAPH 2B without a problem. Unfortunately only one activation monitor set (09) could be recovered during the encapsulation procedure of the specimen holder (the remaining eight being extremely damaged) and a combination of the measurement sets from 09 plus the monte-carlo calculations had to be used to derive the nuclear parameters inside. The measures and calculated reaction rates gave an acceptable match and the calculations were considered satisfactory. The preliminary screening results suggest that turn around may have already occurred after this 4-7dpa-g level of exposure (see Figure 5-7).

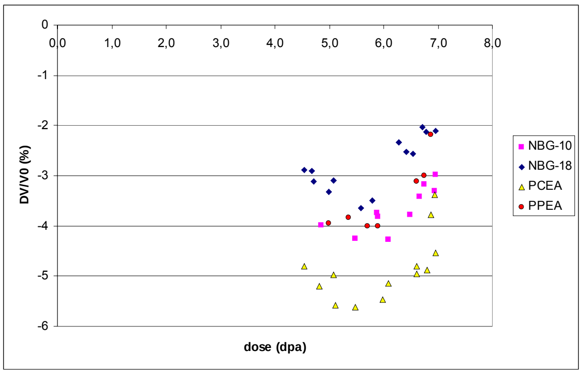


Figure 5-7 INNOGRAPH 2A Preliminary screening results

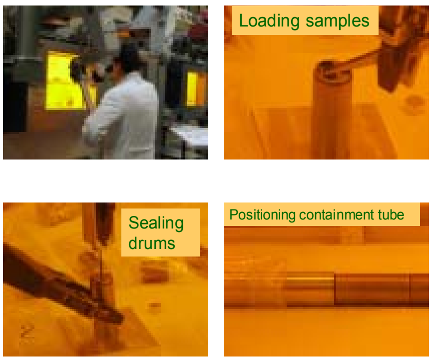
Full PIE of the specimens was carried out during the fifth (extension) year and is reported in [Ref 5-12]. The results are given for dimensional change, dynamic Young's modulus, coefficient of thermal expansion coefficient and coefficient of thermal diffusivity and conductivity. The results confirmed that all grades were beyond turn-round and were in the transition between shrinkage and swelling. The point of turnround occurs at a much lower dose compared to the results of the 750°C (INNOGRAPH 1A) experiments. PCEA shows the highest dimensional change for the Graftech grades whereas PCIB shows very little shrinkage. For the SGL grades, NBG-17 shows the smallest shrinkage values. The number of data points for the IG-110 and IG-430 is less than the other grades but again shows that they are also beyond turn-round. The relative length change of the WG samples compared with the AG samples is lower for all grades, the shrinkage for the WG direction being larger than for the AG direction. For the dynamic Young's modulus the measurements show the highest Eirr to Eo ratio to be 3.4 for PCEA/WG and the lowest to be 1.7 for NBG-10/AG. The spread in results is relatively large and the relative change in value increasing with increasing dose. For the high dose samples a second plateau has been reached. For the CTE the values decrease with increasing dpa and the highest CT_{Eirr} to CT_{Eo} value was 0.84 (for PCEA) and the lowest 0.44 (for NBG-10). There are no significant differences with grain direction. For the coefficient of thermal diffusivity (and conductivity) the diffusivity rates were about 0.6 and 0.3 at 950°C and 25°C respectively. The PCIB showed the lowest ratio at both temperatures and NBG-10 the highest (0.77). All grades show decreasing values with increasing dpa. Again the thermal conductivity was calculated using density and specific heat values with the specific heat of graphite assumed to be the same before and after irradiation.

INNOGRAPH2B Experiment - (4)

The progress of the second stage of the 950oC irradiation programme (INNOGRAPH 2B) has been reported in [Ref 5-11]. The test rig for this experiment again has three columns of graphite samples within the capsules and both fresh and irradiated graphites were loaded into the test rig. The extra drum at the top of the experiment was targeted at 850°C as with INNOGRAPH2A in order to obtain a comparable set of lower temperature results.

The major graphite grades in the INNOGRAPH 2B experiment are as for INNOGRAPH 2A, namely from SGL (NBG-10 & NBG-18) and Graftech (PCEA & PPEA). The minor grades include NBG-17 and PCIB as well as the two Toyo Tanso graphites (IG 110 & IG 430). For this experiment the drum material was changed from Molybdenum to Tungsten based alloy, to generate more γ-heating to reach 950°C, and the lessons learnt from INNOGRAPH 2A and 1B (mainly issues for in-cell assembling and dismantling) were included in the experimental procedures. Fig. 5-8 shows the different procedures in the assembly in the hot cell. The final assembly including welding is outside the hot cell.

Details of INNOGRAPH2B



	Reloaded	Virgin	Total
IG-110	2	2	4
IG-430	2	5	7
LPEB	3	6	9
LPIB	2	0	2
NBG-10	13	18	31
NBG-17	7	12	19
NBG-18	15	18	33
NBG-25	5	6	11
PCEA	16	18	34
PCIB	5	6	11
PPEA	10	20	30
	80	111	191

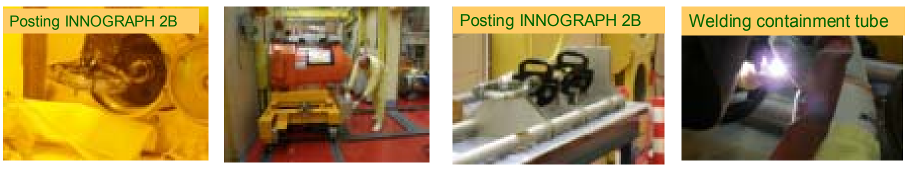


Figure 5-8 INNOGRAPH2B Assembly in the Hot Cells

The INNOGRAPH 2B test was loaded into HFR in July 2008 and was run as long as practicable until the end of the project. As discussed earlier the phase 2 graphite irradiation experiment was affected by force majure events, which meant that the number of cycles required to achieve the target dose would increase (by around 25%). The approval by the Raphael Steering Committee of an extension of the project into a 5th year meant that the final target dose could be achieved (despite a further delay as a result of a shut down of HFR in August 2008 until February 2009). The experiment was finally removed in February 2010 and actions put in place to complete a screening PIE of the results before the end of the project. The full set of PIE measurements on the samples from this experiment and INNOGRAPH 1B will not be available until >2010 since the PIE will be carried out in a future project.

Results of the screening PIE for the high dose second phase INNOGRAPH 2B experiment are reported in [Ref 5-13]. The results are given for the dimensional change, dynamic Youngs' Modulus, coefficient of thermal expansion and coefficient of thermal diffusivity and conductivity. The dimensions of 60 samples are measured. The high-dose samples show swelling with respect to the reference dimensions. Also the DYM values of 30 samples are measured for the high dose samples the DYM starts to decrease after an initial rise. For the coefficient of thermal diffusivity 15 samples are measured. The values of the high dose samples are lower than the PIE values from the first phase experiment INNOGRAPH 2A. The results from these experiments are preliminary at present however when the full PIE has been completed (in a future Framework Project) it is considered that they will provide the basis for the selection and determination of design curves for the graphites to be recommended for the HTR Core

Dimensional Change curves at 950^{o}C

From the results of the above two experiments a series of dimensional change curves were established for the major grade samples investigated in the experiments [Ref. 5-14]. These curves were constructed using the full PIE results from the first phase experiments and the second phase screening results from the second experiment. The assessment was carried out for the four major graphites used in the experiments focusing on linear dimensional changes for the WG and AG directions. Each grade showed a modest amount of shrinkage at turnaround, generally in the range 1.5 to 2.0%, but thereafter they all rapidly went into the swelling phase. After turnaround the original dimensions are exceeded in the range ~7.0 to 11.0 dpa, with those in the AG direction exceeding them before those in the WG direction. Overall it was concluded that the major graphites tested, which involve pitch and petroleum cokes, and extrusion and vibro-moulding, appear to show similarities in dimensional change behaviour at the higher temperature up to, and beyond, turnaround.

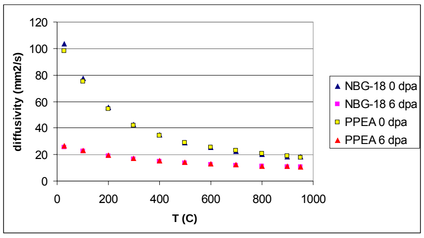
Manufacturer	Grade	Coke	Process	Classification
GrafTech	PCEA	Petroleum	Extrusion	Major
	PPEA	Pitch	Extrusion	Major
SGL	NBG-10	Pitch	Extrusion	Major
	NBG-18	Pitch	Vibro-moulding	Major

Table 5-3 Major Graphites assessed from INNOGRAPH 2A &2 B experiment

5.2 Graphite Heat-up experiment

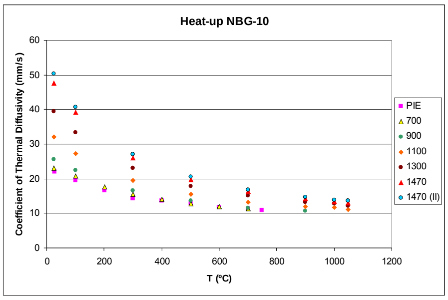
When irradiated, the thermal conductivity of graphite, falls quite dramatically and this can significantly affect the ability to conduct heat out of the core during a fault condition, and hence limit the peak fuel temperature. It is also known that the thermal conductivity of irradiated graphite partially recovers if the temperature is increased above that at which it was irradiated. It might be necessary to claim this partial recovery of the properties when making a safety case for the reactor for such a fault condition. The benefits of performing an experiment to investigated the effects of annealing irradiated graphite has been studied in [Ref.5-16] and proposes that such a heat-up experiment is carried out in Raphael to measure the extent of property recovery with temperature. The report provides an outline of the experimental set-up required and recommends samples from the major grade graphites irradiated in the INNOGRAPH 1A experiment to be used for the investigation.

Heat-up experiment



Sample	Grade	Orientation	Dose (dpa)
S040	NBG-10	AG	7,0
S141	NBG-25	WG	7,8
U008	PCEA	WG	6,3
T043	IG-110	AG	7,5
T067	IG-430	AG	7,6
U111	PCIB	AG	6,3

Recovery of thermal diffusivity:



- Recovery of Thermal Diffusivity & Dynamic Youngs' Modulus from 1100°C
- >3h heating time needed
- Coefficient of Thermal Expansion not recovering up to at least 1465°C

Figure 5-9 Heat-up experiment results

The results and further details of the Heat-up experiments performed are given in [Ref.5-16]. The effect of the partial annealing on thermal diffusivity/ conductivity and also on dynamic Young's modulus and thermal expansion coefficient was investigated. The heating takes place in a Dilatometer and uses a furnace purged with helium to prevent oxidation. The maximum temperature in the furnace is approximately 1400°C. The programme involved heating the first sample in different steps up to 700°C, 900°C, 1100°C, 1300°C and 1400°C from which the heat-up temperature and duration of heating is selected. The lower 700°C temperature is close to the irradiation temperature of 750°C. For the samples the dimensions are measured after each step to determine the effect on the property. The results show that the recovery saturates after a heating time of 3 hours, (hence often a 5 hour heating period was used to ensure saturation). All but the coefficient of thermal expansion shows a recovery of up to 80% of its initial non-irradiated value. The results also show that the higher the heating temperature, the greater the recovery in property (see Figure 5-9). For the Dynamic Young's Modulus the property recovers up to 140-160% after initially having a value of 180-220% after irradiation. The Coefficient of thermal expansion shows no recovery up to a heating temperature of 1470°C.

5.3 Graphite Oxidation experiments

Corrosion resistance at higher temperature is one of the most critical issues affecting the selection of graphite for the reactor core. The need for reliable graphite oxidation data is a crucial issue for the next generation of VHTRs. The limited oxidation resistance of porous C-based materials causes potential problems when they are in contact with oxidising atmospheres. During accident conditions this can lead to a significant localised reduction in strength at exposed surfaces. The necessary safety analyses on graphite oxidation therefore require a detailed knowledge of the oxidation kinetics. Some rapid oxidation tests have been performed on the selected graphites in the HTR-M/M1 programs [Ref 5-1, 5-17] and further investigations were proposed within Raphael [Ref. 5-18] aimed at characterising the oxidation behaviour of the graphite materials and generating kinetic data for the Boudouard reaction (C/CO_2). Such tests are necessary to determine the behaviour of the different graphites under accident conditions.

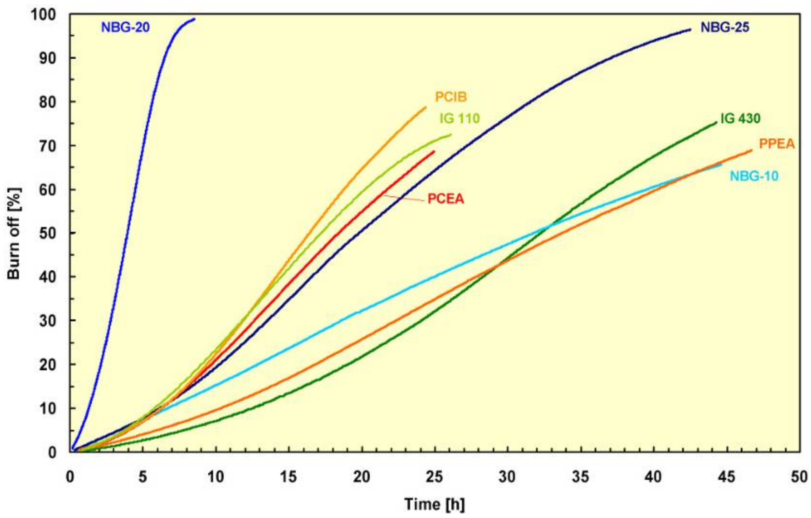
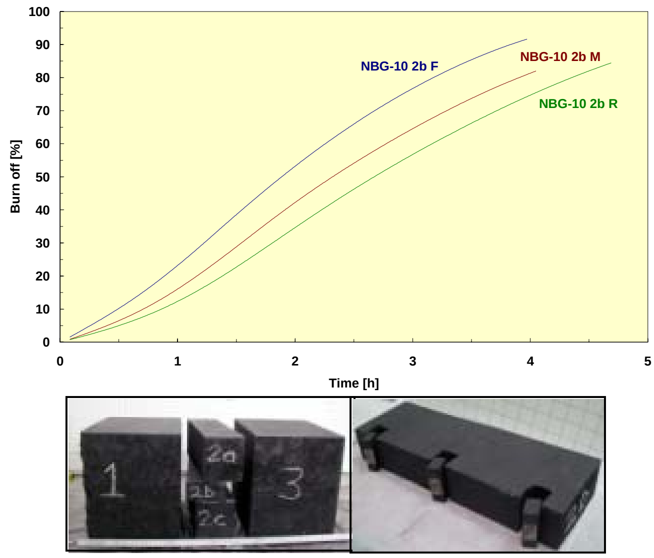


Figure 5-10 Exemplary graph from HTR-M1 for high oxidation rates of the selected graphites at 650 °C in air

The test programme of graphite characterisation started in the HTR-M projects [Ref. 5-17] involving tests in oxygen, and air at temperatures up to 950°C. This has been extended in Raphael to cover effects in steam at 900°C. Oxidation of the graphite also takes place during normal operation due to impurities in the cooling gases. In this case however the oxidation rate of the graphite is extremely low. Nevertheless it will be necessary to determine the appropriate oxidation kinetics so that through life predictions of component oxidation distributions can be determined for different levels of impurities. A proposal has been developed within RAPHAEL to study this in conjunction with a collaboration with KAERI [Ref. 5-19].

The rapid oxidation data derived from the HTR-M/M1 programme showed a good comparable reactivity for most of the selected graphites, with one clear exception (NBG-20) which had a much higher reactivity (see Figure 5-10). The reason for this "unusual" behaviour is not well understood. It was also shown that there are differences in oxidation rates depending of the location in a graphite block where the sample was extracted. Similar observations but at lower reactivities were made during new tests with NBG-10 samples extracted from different parts of a 55 kg block (Figure 5-11). Investigations of further graphites have been carried out in RAPHAEL and are reported in a series of progress reports [Ref. 5-20 to 5-22].



NBG-10 Block with parts of extracted samples (R, M, F)

R= Rear; M=Middle; F=Front

Fig. 5-11: Burn off vs. time of NBG-10 samples extracted from different locations - Oxidation in air at 750 °C.

A first progress report on the work carried out is given in [Ref 5-20]. This discusses the importance of specimen size on oxidation rates and reviews some screening experiments on the Boudouard Reaction in HTR relevant graphites. At low temperatures (regime I) the chemical reaction itself is the rate limiting step and the material is corroded homogeneously within the pore system, which leads to a severe loss of mechanical strength. At intermediate temperatures (regime II), consumption of the corroding gases within the pores becomes important and the corrosion attack becomes smaller within the depth of the material; this is because the temperature dependence of the chemical process is much larger than that of the competing in-pore gas diffusion. At high temperatures (regime III) external mass transfer to the outer surface is the rate limiting step and the reaction is restricted to the geometrical surface only; mass transfer rules are applicable for rate calculations which means that kinetics is not influenced by the properties of the solid and are not suitable for material characterization. Whilst the classical theory suggests no influence of specimen size on oxidation rate, some preliminary low temperature oxidation experiments on NBG-10 have shown a significant rate dependence on the specimen size. This was possibly due to two poorly interconnected pore systems. Additional experiments have therefore been recommended to measure the density profiles in oxidized graphites by X-ray tomography.

A second progress report [Ref. 5-21], looks at oxidation experiments in air on selected graphites including NBG-10, NBG-18, NBG-17 (SGL) and PCEA, PPEA (Graftech) plus some newly developed 3D-CFCs for use in HTR control rods. Oxidation experiments at 1500°C reveal almost the same rate for all specimen; this and the shape of the rate vs. burn-off curve reveals, that external mass transfer is dominating. Major differences are found however for lower oxidation temperatures. The rate sequence was found to be practically the same as that observed earlier in oxygen (e.g. NBG-10 is more oxidation resistant than NBG-18, PPEA oxidizes slower than PCEA). Reaction rates in 20 vol-% CO_2 at 1200°C are about the same as those in air at 700°C. A major rate difference was again observed between the inner and outer parts of NBG-17 (as discovered previously). This result can be explained by density and impurity differences between centre and edge specimens in the block (Figure 5-12).

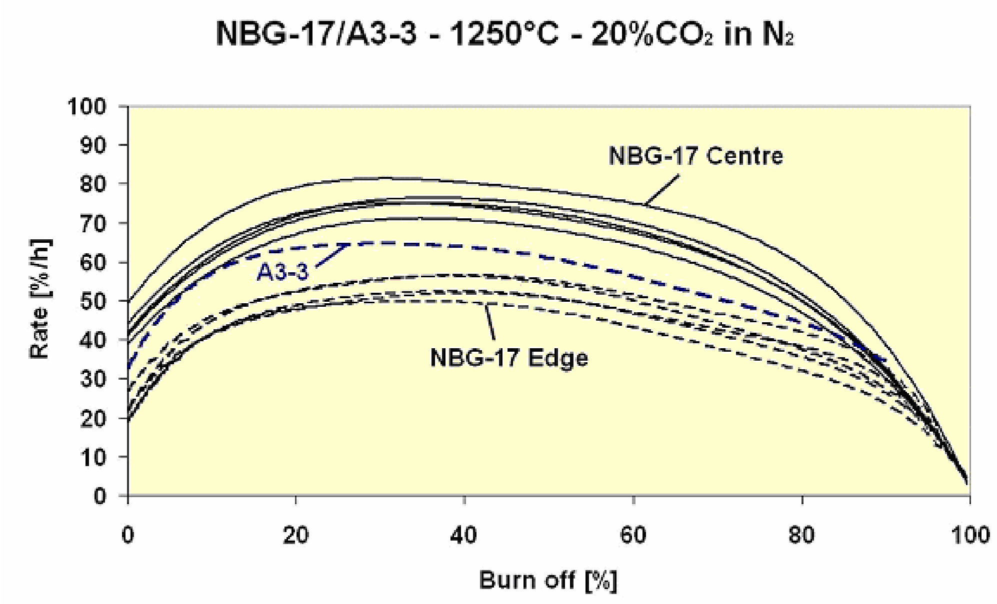


Fig. 5-12: Log (rate) vs. burn-off for transient experiments on A3-3 and on NBG-17

Six different CFC materials have been developed by SGL (in cooperation with the FP6 ExtreMat project) for use in control rods of future HTRs. Two of these materials were tested in air to assess their oxidation stability. Both materials tested were highly pure 3D-CFCs, whose temperature treatment in manufacture occurred at >2200°C. The materials were found to have significantly better oxidation stability than the reference graphite material V483T (see Figure 5-13) and compare well with other composite reference materials (such as ITER reference material NB31). The positive results from these two composite samples underlines the importance of low impurity levels and high temperature treatment as key parameters in the design of low reactive carbon materials. An overall report on the graphite & composite corrosion work at the end of the 4th year is given in [Ref. 5-22]. This has been completed with the results of PhD work on Boudouard reaction [Ref 5-23] and a final report issued on all the oxidation work [Ref 5-24].

Ref [5-24] also reports results of calculations on air ingress accidents with a graphite fire using the code REACT/THERMIX. A chimney draft representative of two leaks (one on the top and one at the bottom of the reactor vessel) was assumed. The calculations were made for the ANTARES prismatic block concept. The calculations showed the oxygen to be consumed mainly in the bottom reflector with the time available to deploy counter measures dependent on air ingress rate and burn-up. These results do not necessarily represent the worst case and further investigations are required.

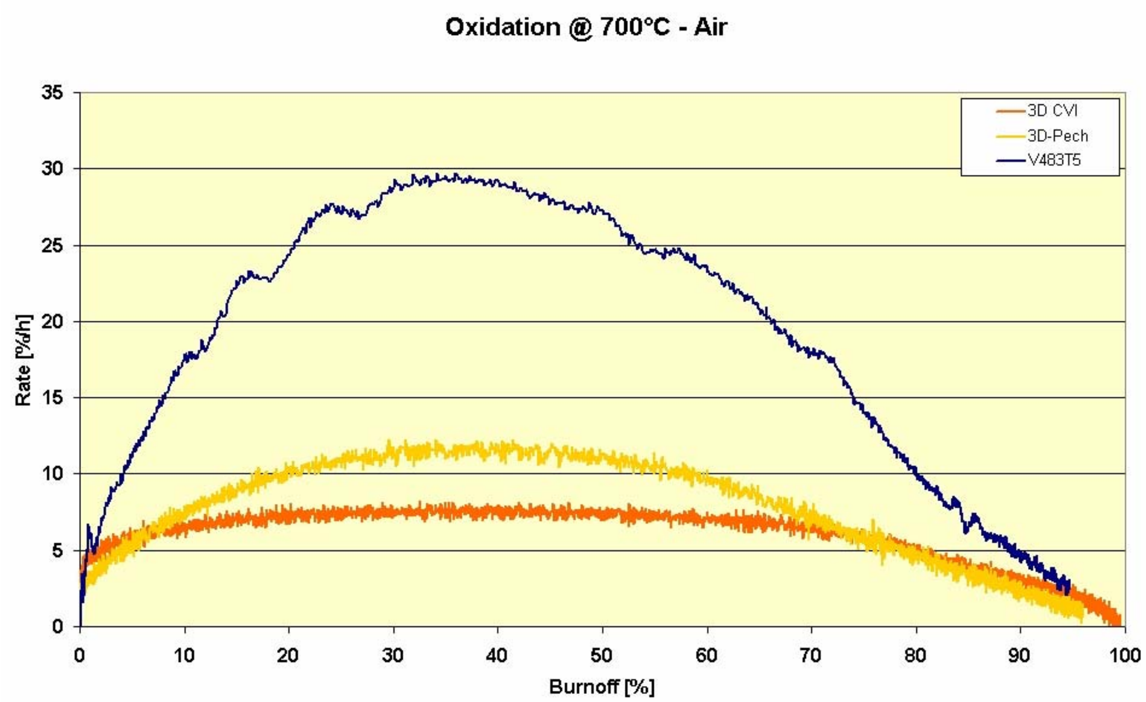


Fig. 5-13: Rate versus burn-off for 3D CFCs and V483T in air (700°C)

5.4 Graphite Modelling investigations

The RAPHAEL graphite programme includes a task on the development of micro-structural modelling. The investigation of the effects of neutron irradiation currently relies heavily on testing and given that suitable facilities for such testing are both scarce and expensive to use and that manufacturers are continually developing and offering new graphites for such applications, in the longer term it is important to develop an alternative modelling approach (in parallel with the tests) that will both facilitate the longer-term development of graphites and minimise and reduce the extent and requirement for future irradiation testing.

The work within the modelling programme aims to make use of micrographs and tomography images from virgin and irradiated graphites to understand the graphite microstructure and behaviour. The work considers different modelling methods and properties and applies these to results from two specific graphites from the irradiation programme. It is expected that this type of development will lead to a longer-term specification for manufacturers, constructors and users and give added confidence in support of licensing.

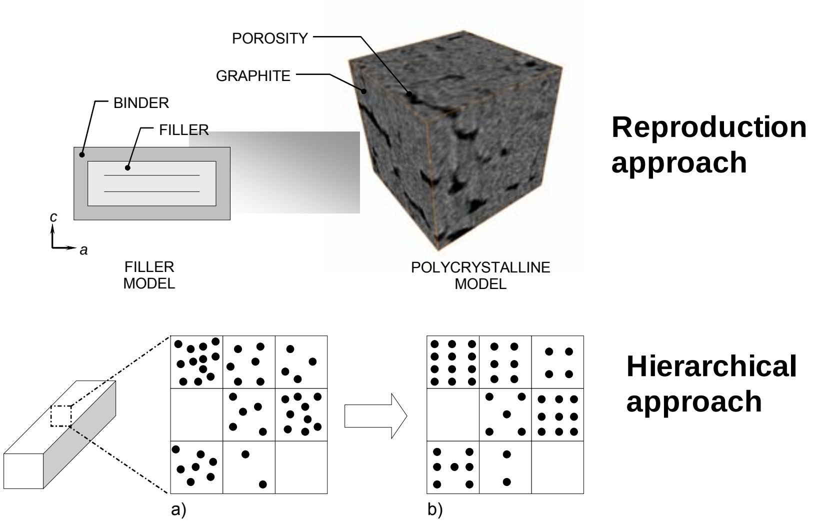


Figure 5-14 Micro-structural Modelling

A series of reports have been issued describing the progress made [Ref. 5-25 to 5-28]. The two modelling methods investigated were the reproduction approach and the hierarchical/embedded approach. These were reviewed and applied for dimensional change and Young's modulus changes to two grades of graphite PCEA and NBG-25. However initially neither approach gave a good agreement with the available experimental data. The hierarchical/embedded approach was selected as the better approach for further investigation and for the modelling of thermal conductivity in PCEA and NBG-25. Most of the approaches require constituent highly oriented pyrolytic graphite (HOPG) behaviour as an input. There are very limited data available for the required environment (dose and temperature), which could restrict the effectiveness of any models. It is recommended that such data be collected if any micro-structural models are to be developed. There are limited micro-structural studies on irradiated graphite and thus, little support for the micro-structural mechanisms behind the irradiation-induced changes in graphite. It is recommended that such investigations need to be conducted to aid the development and support of micro-structural models.

The findings from the last year's investigations are summarised in [Ref. 5-29]. This report includes the use of X-ray tomography on two irradiated lower dose samples from the experiments (NG-10 and PCEA). The results indicate that irradiation-induced microstructural (pore) changes can be detected in the tomography data which gives some confidence that the computed X-ray tomography technique can be used to gain further insight into the micro-structural changes within the graphite and also be used in the development of predictive models. In order to gain quantitative results, additional X-ray tomography scans at higher resolutions would have to be carried out.

5.5 Support to SP CT & SP SL

An important part of the SP4 ML activities is to provide materials support to the RAPHAEL components work performed in SP5 CT. This has included the provision of materials information on graphite for tribology tests within SP4 CT and for the NACOK experiment in SP SL. Recommended graphites include NBG-17 from SGL and PCEA from Graftech. For the tribology tests [Ref. 5-30], the machined surface finish should be typical of a core block (milled or turned) with all specimens measured for roughness and the faces kept dry and free of water. For the NACOK experiment [Ref. 5-31], the requirement is suitable quantities of graphite to occupy the complete height of the test rig (150x150mmx 1400mm height) and to manufacture test specimens 150 x 150mm x 400mm for including in the test area. Two differently manufactured graphites i.e. one pitch coke and one petroleum coke graphite, were recommended (one from each major supplier) as with the tribology tests and if possible one of the vibro-moulded graphites should be tested. The results of the NACOK and tribology investigations are reported in the SP CT and SP SL Sub-projects.

5.6 Generation IV VHTR Graphite Working Group (GWG) activities

The Graphite Working Group (GWG) is a forum established within the Generation IV VHTR Materials Project Management Board (PMB) to coordinate details of the national graphite R&D programmes in support of VHTR. The work programme involves data acquisition; design methodology and construction; operation and inspection; decommissioning and disposal. Its coordination activities shall include: collaborations; sample exchanges and joint experiments; review of collaboration plans: and assessment of data produced by participating nations (or their subordinate organizations) within the framework of the GIF Materials PMB. The GWG will also review the scope of national R&D programs and recommend additions to close any identified data gaps.

GWG – List of Tasks

- **Task 1 Contains – 11 Sub Tasks:**
- **Task 2 Operation & inspection (initial tasks specified)**
- **Task 3 Decommissioning & Disposal (initial tasks specified)**

Task 1 – Sub-tasks

Graphite Task	EU involvement
TASK1 - Graphite selection & acquisition strategy -	Yes
TASK2 - Graphite mechanical & physical properties (non-irradiated)	Yes
TASK3 – Graphite fracture behaviour	No
TASK4 – Graphite oxidation behaviour	Yes
TASK5 – Graphite component testing	No
TASK6 – Graphite Irradiation effects	Yes
TASK7 – Graphite Irradiation induced creep	No
TASK8 – Graphite Codes & Standards development	Yes
TASK9 – Graphite behaviour model development	Yes
TASK10 – Links with existing databases	Yes
TASK 11 – Materials Database	Yes

Table 5-4 Graphite Working Group Tasks

Euratom members of the GIF VHTR Materials PMB are as follows:

D. Buckthorpe (AMEC) - PMB Member
M Davies (AMEC) – GWG Member (currently Chair of GWG)
A Vreeling (NRG) – GWG Member

The tasks to be addressed by the GWG are listed in Table 5-4. Also indicated are the Task 1 Sub-tasks with EU involvement. The RAPHAEL materials partners have agreed that all the results from the RAPHAEL materials subproject can be used as part of the Euratom contribution to Generation IV programme, hence the results from the activities summarised in this report will be submitted as part of the Euratom contribution to the GWG. Exchange of information has begun after all Generation IV Partners signed the Project Arrangement which details a five year programme of collaboration for the period 2007 – 2012. The JRC is the signatory on behalf of Euratom. Details of the Euratom (labelled JRC) contributions and deliverables to be submitted to the GWG are given in [Ref. 5-32].

5.7 Overall Conclusions on Graphite Materials

The status of the work carried out on Graphite materials within Work package 3 of RAPHAEL SP4 ML for the VHTR has been summarised and is reported [Ref. 5-33]. The work includes:

Irradiation experiments in HFR at 750°C and 950°C and assessment of results

- Irradiation testing has been successfully performed using a two stage testing procedure which allows irradiated samples to be re-inserted back into the reactor 9along with fresh samples) to obtain results over a range of dpa levels up to high dose (25 dpa)
- The irradiation tests have been completed at two nominal temperatures (750°C and 950°C) and preliminary evaluations carried out for the high dose stage of the 950oC experiment
- As a result of force majure events occurring within the HFR, an extension of the project programme was agreed to allow the INNOGRAPH1B and INNOGRAPH2B graphite irradiation tests to be completed.
- Because of a further Force Majure event at HFR the screening of the 950°C irradiations will take place and be reported later in 2010
- Full PIE evaluation of the first stage 750°C and 950°C have been reported and the behaviour and physical properties of the different graphites compared.
- An assessment of the design curves at 750°Cand 950°C has been made and the results suggest that at the higher temperature all graphites are in the expansion phase suggesting they may not be suitable for Pebble Bed applications at this temperature. This is to be confirmed when full PIE has been carried out in a later Framework.
- Results have been obtained from a Heat-up experiment to evaluate properties of irradiated samples after a short exposure to high temperature and full recovery of the properties (except CTE) was obtained.

Corrosion testing of graphites used within the irradiation experiments and selected composites

- Corrosion tests have been performed on all the graphites used in the irradiation experiment. The results suggest the major graphites are satisfactory although some variation in properties within the block was observed for NBG 17.
- A PhD study on the Boudouard reaction of NBG-17 has been completed as part of this project work
- Results of calculations on air ingress accidents with a graphite fire using the code REACT/THERMIX have also been reported.
- Corrosion tests on some supplied SGL composites have completed and shown to provide better corrosion resistance that graphite

Graphite modelling investigations to predict the behaviour of graphite

- A series of reports have been issued describing the progress made evaluating two modelling methods: the reproduction approach and the hierarchical/embedded approach. Evaluations have so far not concluded an advantage to either approach.
- Comparisons have been made between predicted properties and measured results for two graphites PCEA and NBG-25
- X-ray tomography has been carried out on two irradiated lower dose samples from the experiments (NG-10 and PCEA). The results indicate that irradiation-induced micro-

structural (pore) changes can be detected in the tomography data which gives confidence in the use of this procedure for future model investigations.

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6 Summary of SP4 Work package 4 on Design Codes

Graphite and Carbon Composites are candidate materials for structural parts of the HTR and VHTR, graphite for the reactor core and carbon composite a candidate for the VHTR control rod. The materials investigations in RAPHAEL includes an evaluation of the potential and properties of these materials for these applications with special emphasis on their behaviour under irradiation. The metallic materials investigated within RAPHAEL SP4 ML are Mod 9Cr1Mo steel for the Reactor Pressure Vessel and high temperature alloys (Haynes 230, IN 617 and ODS materials) for the IHX. This section summarises the work performed within Work Package 4 of SP4 ML in support of Guidelines and Design Code developments for these materials. The tasks include the development of European Guidelines for composites and graphite, feedback from the ASME Graphite Codes and Standards Committee and the GIF Graphite and Composites Working Groups, summary of the work performed on metallic materials including the provision of design data and the status of the database development within ODIN at JRC which will be used to house the project materials data.

6.1 Guidelines for Graphite Materials

In order to be able to carry out the initial design of the graphite core in a (Very) High Temperature Reactor (V)HTR it is essential to have reliable graphite data and a proven design methodology. In the past, the design of graphites cores has generally been carried out by the countries in which the reactors were built (UK, France, Germany, Russia) using their own developed methodology and data on their particular graphites. However, most of the graphites that have been used in previous core designs are no longer manufactured commercially, and currently available grades are limited to a few specific sources of raw materials. There is therefore a major programme of work underway, within RAPHAEL and Generation IV (GIF), to qualify some potential graphites. In particular, long term irradiation experiments are being undertaken within RAPHAEL within the High Flux Reactor (HFR) at Petten to assess properties and behaviour and to select and develop improved graphites for the future. The methodologies developed in each country will have similarities, but there will also be differences and it is beneficial to have a unified methodology for core design within Europe and Internationally, the latter of which is being addressed through an ASME Design Codes Committee. The results from both these activities are reported in the following sections.

RAPHAEL Activities on Graphite Guidelines

The Scope and Planning for this activity is reported in Ref [6-1]. The report summarises the selected graphite grades being considered for the experiments and at specialist graphite meetings (Table 6-1). The graphite experiments within RAPHAEL are aimed at providing material properties at 750 and 950^{o}C at irradiation dose levels representative of both the prismatic and pebble bed VHTR designs. The assembled database within RAPHAEL will cover effects of irradiation and oxidation and the results are to be housed in the ODIN database Ref. [6-2].

Within Ref [6-1] the calculation requirements are summarized together with available information on existing design Codes. For the UK, a document entitled 'The Compendium of AGR Core and Sleeve Data and Methods' was developed which contains all the graphite data and methods needed to make future operational safety cases for the UK AGRs. This is a live document that is updated as required to reflect improvements in methodology and revisions to the graphite data.

Grade	Manufacturer	Coke	Process
PCEA	GrafTech	Petroleum	Extrusion
PPEA	GrafTech	Pitch	Extrusion
PCIB-SFG	GrafTech	Petroleum	Iso-moulding
BAN	GrafTech	Needle	Extrusion
NBG-10	SGL	Pitch	Extrusion
NBG-20	SGL	Petroleum	Extrusion
NBG-25	SGL	Petroleum	Iso-moulding
NBG-18	SGL	Pitch	Vibro-moulding
NBG-17	SGL	Pitch	Vibro-moulding
IG-110	Toyo Tanso	Petroleum	Iso-moulding
IG-430	Toyo Tanso	Pitch	Iso-Moulding

Table 6-1 - Selected graphite grades

The data sheets cover Young's modulus, strength, dimensional change, coefficient of thermal expansion, thermal conductivity, irradiation induced creep, specific heat and stored energy, gas transport properties (diffusivity ratio and viscous flow coefficients), virgin bulk density and open pore volume. The method sheets cover the calculation of fast neutron fluence, temperature and radiolytic oxidation distributions in components, brick geometry changes, core loads, brick internal stresses and external (load applied) stresses, brick integrity and core geometry.

Within Europe, for the VHTR, it is proposed to house the graphite data and design methodology within a similar compendium to that used for the UK and steps to achieve this are outlined with progress reported in Ref [6-3]. The main sections would be similar to the ASME code being developed, but the general structure/layout will conform to that of the UK Code. Aspects of the methodology contained within the ASME graphite code currently under development are to be referenced as required. The information contained within the Compendium would be design curves and properties developed from the RAPHAEL experiments.

Most, if not all, of the data and methodology sections will be required. All the necessary data for the grade(s) of graphite selected will be contained in one Appendix. All the methodologies required to carry out a core design will be contained in a second Appendix. The proposed structure is as follows:

Appendix A – Graphite data sheets
- (i) Stress–strain relationship (Young's modulus)
- (ii) Strength
- (iii) Dimensional change behaviour
- (iv) Coefficient of thermal expansion
- (v) Thermal conductivity
- (vi) Irradiation creep
- (vii) Oxidation behaviour (rapid + long term)
- (viii) Other properties

Appendix B – Graphite data sheets
- (i) Calculation of fast neutron fluence distributions in a component
- (ii) Calculation of temperature distributions in a component
- (iii) Calculation of oxidation distributions in a component
- (iv) Calculation of core flows and pressure drops
- (v) Calculation of component internal stresses and distortions
- (vi) Calculation of interaction with interfacing structures
- (vii) Calculation of external loads
- (viii) Calculation of applied stresses from external loads
- (iv) Calculation of core movements (channel geometry)
- (x) Calculation of component integrity

ASME Activities on Graphite Design Rules

An ASME Project Subgroup called 'Sub-group on Graphite Core Components' was initiated in 2004, and is currently developing a design code for graphite core components for HTRs. The Sub-group has members from the USA, UK, France, South Africa, South Korea and Japan. The existing draft ASME design code was used as the starting point, but because this doesn't adequately cover irradiated components, a major re-write was required. Also, the original deterministically based code will now include probabilistic methodologies. The ASME code when completed will include rules for materials, design, part machining, testing and installation, as well as quality control requirements equivalent to NCA-1 (see below).

The content of the code has to follow the basic general layout of all ASME codes although these have been modified slightly for the graphite. In addition to the main code sections, some mandatory and non-mandatory appendices are included, as well as the number of 'White Papers' which generally provide a justification for the content of the code. The currently proposed make-up of the code is described below:

Main text

Article 1000	Introduction	
Article 2000	Materials	
Article 3000	Design	
Article 4000	Machining, testing and examination	
Article 5000	Installation and examination	
Article 8000	Certificates and stamping	
Article 9000	Glossary	

Mandatory appendices

Appendix 1	Graphite material specifications
Appendix 2	Requirements for creation of a material data sheet
Appendix 3	Requirements for generation of design data for graphite grades

Non-mandatory appendices

Appendix I	Graphite as a structural material
Appendix II	Irradiation damage to graphite
Appendix III	Oxidation and its effects on graphite
Appendix IV	Recommended practice for stress analysis of an irradiated part

To speed up the ASME review process, draft sections of the code are issued for review as they are completed. Completed drafts of Articles 1000, 2000, 4000, 5000 and 9000, and incomplete drafts of Article 3000 have recently been issued for an initial review/ballot. Drafts of the mandatory and non-mandatory appendices are in preparation. A General Requirements section equivalent to NCA 1has also been produced with the assistance of the ASME Sub-group on General Requirements. This is also recently been issued for review/ballot

The complete draft of the ASME design code is not expected to be endorsed by ASME (and included in the B&PV code) until after 2010 at the earliest. Given the existing European and US irradiation programmes (within GIF), the complete data set on the irradiation behaviour of the chosen graphites will also not be available until beyond 2012. (This is mainly due to the timescale involved with the irradiation creep experiments being carried out in the US as part of its program.

Feedback from the GIF VHTR Materials Graphite Working Group

The EU has signed up to a collaborative agreement with other countries involved in the development of (V)HTR technology via the Generation IV (GIF) International Forum. In order to provide an effective collaboration between the different countries on graphite, a Graphite Working Group (GWG) was set up within the VHTR Materials Project Management Board (PMB) with membership from the EU, France, USA, South Africa, South Korea and Japan. Its charter is as follows:

'The Graphite Working Group is a forum to coordinate details of national graphite R&D programmes in support of: (V)HTR data acquisition; design methodology and construction; operation and inspection; decommissioning and disposal. Coordination activities shall include: collaborations; sample exchanges and joint experiments; review of collaboration plans: and assessment of data produced by participating nations (or their subordinate organizations) within the framework of the GIF Materials & Components Project Management Board. Moreover, the GWG shall review the scope of national R&D programs and recommend additional work scope to close any identified data gaps.'

The GWG has so far met on five occasions, and plans to meet on average about twice per year. The GWG has produced a detailed collaboration plan contained within the VHTR Materials Project Management Board (PMB) Project Arrangement (PA) [Ref. 6-4] which describes the coordinated research activities for graphite for future (V)HTRs. It covers the following areas:

1. Graphite selection and acquisition strategy
2. Graphite mechanical and physical properties (virgin material)
3. Graphite fracture behaviour
4. Graphite oxidation behaviour
5. Graphite component testing
6. Graphite irradiation effects
7. Graphite irradiation induced creep
8. Graphite codes and standards development
9. Graphite behaviour models development
10. Links to existing graphite irradiation behaviour databases
11. Materials data base

Work is already ongoing in some of these areas and planned in others.

Currently within RAPHAEL only data relating to items 2, 4 and 6 above are being obtained on the selected graphites. These will be placed on SINTER. Additional data for design Rule development, as well as data on items 3, 5 and 7 will be obtained from the GIF collaboration. All the (shared) graphite data obtained will be placed on a general graphite database, the format of which is being developed by the GWG. With regard to item 8 on Codes and Standards (i.e. Graphite Design Rule) development, the main EU input is therefore from the combined activities of RAPHAEL and ASME Code developments as described earlier.

6.2 Guidelines for Composite Materials

There may be regions of the VHTR core, or specific items of the VHTR that may have to be made out of carbon-carbon (C-C) composites, because the normal operating temperatures or potential fault condition temperatures may preclude the use of metallic components. An example of this is the Control Rod. There is therefore a need to establish what candidate C-C composites are available, and to qualify them for use in the environment to which they would be subjected. This is likely to include fast neutron irradiation and/or high temperature. In addition, there is a need to develop suitable methodologies for the design of reactor components made from C-C composites. Consensus methodologies for graphite core component design are being addressed through ASME. It is also the intention to develop consensus methodologies for components designed using other ceramic materials such as C-C composites. It looks as if another ASME Sub-group will be set up to do this and will benefit from the work already carried out by the graphite Sub-group.

The following sections provide a summary of the findings from the RAPHAEL activities on development of guidelines for Composite materials. Also included is a review of the available feedback from the ASME activities and from the GIF Ceramic Working Group.

RAPHAEL Activities on Guidelines for Composites

The Scope and Planning for this activity is reported in [Ref 6-5]. The report also summarises the issues connected with the selection of composites, the requirements for irradiation testing, and the scope and planning requirements for the development of a database. Within [Ref 6-5] the calculation requirements are summarized together with available information on existing design Codes.

For Composite guidelines, an approach similar to that of graphite is proposed and reported in [Ref. 6-6]. The most important class of properties of C-C composites is their thermal properties. C-C composites have very low thermal expansion coefficients, making them dimensionally stable at a wide range of temperatures, and they have high thermal conductivity. C-C composites retain mechanical properties even at temperatures (in non-oxidizing atmospheres) above 2000°C. They are also highly resistant to thermal shock, or fracture due to rapid and extreme changes in temperature. The material properties of a C-C composite vary depending on the fibre fraction, fibre type selected, textile weave type and similar factors, and the individual properties of the fibres and matrix material. C-C composites are available as unidirectional (1D), 2-D and 3-D reinforced.

However unlike graphite, they are much less resistant to fast neutron damage and they generally oxidise more readily at temperatures between 600-700°C, especially in the presence of atomic oxygen. A protective coating (usually silicon carbide) should be considered to prevent high-temperature oxidation, adding an additional manufacturing step and additional cost to the production process. Some investigative work on oxidation of selected composites was carried out in the HTR-M Project [Ref.6-7].

For the main sections of the proposed composites code, these can follow a similar structure to the ASME graphite Code but with their content adjusted to comply with Composite material requirements as shown below:

Section 1000	Introduction
Section 2000	Materials. General requirements, material property requirements, sampling requirements, manufacturer's QA requirements, as well as examination, packaging, transportation and storage requirements.
Section 3000	Design. This section will cover general design requirements (including part classification, loading criteria), design by analysis (including both deterministic and probabilistic methods) and design criteria
Section 4000	Manufacture, testing and examination. This section will cover general and specific requirements for the manufacture of the C-C composites and components, and the subsequent testing and examination of the components
Section 5000	Installation and examination. This section will cover the general and specific requirements for site installation and examination
Section 9000	Glossary. This section will provide definitions of the appropriate terms in the code.

The material data and design methodology will be contained in two appendices, the first of which contains the material data and the second one the methodologies required to carry out the design of a component.

ASME Activities on Ceramics/Composites Design Rules

To date, the ASME Sub-group has not considered the requirements for composites. It has however recognized the need to develop such requirements after the graphite design rules have been submitted. Another Sub-group with the appropriate expertise will be set up to develop the new code but it is likely that the motivation and timing of this will very much depend upon the extent and importance of the components in which composite materials are being used.

Feedback from the GIF VHTR Materials Ceramics Working Group

The Ceramics Working Group (CWG) which is attached to the GIF VHTR Materials Project Management Board (PMB) on materials has as its membership participants from the EU, France, USA, South Africa, South Korea, Switzerland and Japan. The CWG has so far met through Teleconferences and has produced a detailed collaboration plan (contained within the PA [Ref. 6-4]) which describes the coordinated research activities for composites for future (V)HTRs.
The programme covers six tasks:

1. Out of Pile components – Heat Exchangers - virgin properties
2. Out of Pile components - Insulating materials and structures -
3. In Pile Components – Control Rods -
4. In Pile components - Other internal structures
5. Codes and standard development
6. Materials data base

and within each task there are a number of sub-tasks that are considered necessary to understand the material and component behaviour:

- Design, fabrication, processing and assembling of materials
- Mechanical and physical Properties of materials (virgin materials)
- Environmental properties of material
- Behaviour Models development
- Acceptability tests

The CWG activities include aspects of evaluation, irradiation testing, fabrication and covers SiC/SiC and C-Composite materials. Currently within RAPHAEL data relating to items 2, 3 and 6 above are being obtained. Additional data for design Rule development, as well as data on items 1 and 4 is expected to be obtained from the GIF collaboration. All the (shared) composite data obtained will be placed on a general graphite database, the form and location of which is still to be agreed between the GIF partners. As with Graphite Design Rules, the main EU input for item 5 is from the combined activities of RAPHAEL and ASME Code development described in the above sections.

6.3 Codes & Standards work on Metallic Materials

R&D Recommendations to support Design data and Design rule omissions for the VHTR

This document [Ref. 6-8] addresses Research and Development (R & D) recommendations on material issues and for Design Code omissions for the VHTR and its components. The VHTR system can operate at potentially much higher temperatures than other HTGR systems and further R&D is needed to increase the coolant temperatures and to develop interfaces between the Nuclear Heat Supply System and the heat utilisation systems. The materials covered include composites, metallic and high temperature resistant alloys and ceramics. It deals with the needs and characteristics of such materials including issues that may impact on availability and manufacture.

The report considers the requirements for the main VHTR components, the requirements within the reactor (the fuel and core assemblies), the reactor block materials (control rod, core support and vessel), the heat exchanger and other power circuit components (turbine, hot gas duct, the gas circulator and valves). Existing materials and component requirements are reviewed and recommendations made on future research actions.

Key areas that remain include the development of Graphite, SiC and composite design procedures for the core and its components, high temperature materials for the IHX, vessel materials behaviour at normal operating temperatures where small levels of creep are possible, coatings for corrosion and wear resistance and needs of a large gas circulator. The overall conclusions are that a significant amount of R & D on material technology issues still remains to be done if the VHTR is to develop further to achieve its aim of providing robust highly efficient heat to a range of electricity and process heat applications and to provide the baseline nuclear unit for future large scale hydrogen generation.

Report on Creep/Fatigue (Interaction) of Mod 9Cr-1Mo Steel for vessel application – Review and Recommendations for Future Work

The objective this report [Ref. 6-9] is to provide a summary of the information available on Creep-fatigue Interaction effects for modified 9Cr1Mo steel applicable for its use as a vessel steel for the VHTR and in relation to Codes and Standards development. This is important since it has been identified that creep/fatigue evaluation procedures currently available in the ASME design Code and RCC-MR have predominantly been developed for austenitic stainless steels, and may not therefore be suitable for cyclic softening materials, such as modified 9Cr1Mo steel.

Modified 9Cr1Mo steel has been considered for application in VHTR components: pressure vessel, cross vessel and core support, for normal operating temperatures up to 500ºC. The advantage of modified 9Cr1Mo steel over conventional steel materials is its low thermal expansion coefficient and high thermal conductivity. It has good weldability and microstructural stability over long periods of exposure to high temperatures, and is expected to have better behaviour in Helium environments compared to conventional LWR ferritic steels.

The report considers the available creep and fatigue properties in existing Codes and Standards and recent ASME reports in which creep-fatigue evaluations have been performed and future needs in terms of R & D and other materials information has been highlighted. Modified 9Cr-1Mo steel vessel experience is very limited with respect to thick sections. Recommendations have been made with respect to material specifications, end of life properties and a section included on qualification issues for the vessel.

Specific recommendations arising from this review include:

Creep-fatigue procedures:

- Investigate Cyclic softening and symmetrisation effects
- Stress levels at the beginning of the dwell
- Treatment of relaxation
- Definition of creep-fatigue interaction diagram
- Application of Ductility Exhaustion methods

Creep-Fatigue test information

- Tests at temperatures below 550^{o}C
- Tests with longer hold times
- Characterisation of cyclic softening and effects of ageing
- Investigate effects of reactor environment
- Tests with effects of post weld heat treatment
- Tests on (cross) welds

- The above tests should be performed on products corresponding to the future procurement specifications for the VHTR.

Other tests and evaluations

- Development of Creep strain accumulation laws (in ASME)
- High cycle Fatigue Tests (load controlled)
- Establish end-of-life fracture properties
- Feasibility study for a prototype forged shell in Modified 9Cr-1Mo steel
- Properties over the temperature range covered by operation and accident conditions (400-650°C) and timescales.
- Low temperature toughness of Modified 9Cr-1Mo base material and weld metal.
- High temperature tensile and creep properties of Modified 9Cr-1Mo base material and weld metal.
- Availability of welding processes and welding consumables for Modified 9Cr-1Mo steel.

Within RAPHAEL creep tests are being performed at 450^{o}C, also ageing however a large number of other issues remain and need to be considered within Generation IV and future EU Framework Programmes.

Prioritising of short comings in National & International Design Codes

This report [Ref. 6-10] reviews and evaluates currently available national and international codes and procedures to be used in design of HTR's and VHTR's. The focus of the report is on the applicability of the present codes and standards (mainly ASME) and the identification of key omissions and short-comings in order to make recommendations on future actions for updating such Codes for application to the HTR/ VHTR conditions.

Key shortcomings include those for the vessel (application of Mod 9Cr 1Mo steel), for the IHX (lack of materials information for IN 617 and Haynes 230), weldment and dissimilar weld (where applicable) fatigue and creep-fatigue behaviour at representative HTR and VHTR temperatures for the above materials. Also needed are Design Rules for Graphites and Composite and Ceramic materials. A summary of omissions and gaps in current codes and standards is given for reference.

Outline rules /guidelines for Nickel based Alloys

The report [Ref. 6-11] deals with a review of possible alloy materials for high temperature application and an outline of a Design Code procedure that will need to be developed for such materials for the Codes and Standards application. Mechanisms such as corrosion – erosion -

helium effects are considered and the needs for off-normal situations. The outline needs consider the temperature limits and materials used, the Draft Code Case for IN 617, the mechanical results needed for IHX application and elastic and inelastic rules and procedures are required for meeting strain limits and fatigue and creep-fatigue criteria.

The key issues/changes that need to be considered include:

- Inelastic analysis and therefore the development of applicable constitutive equations above 650°C for certain materials
- Rules and properties applicable to the environment and fluid (helium + impurities).
- Fracture analyses to justify the material after significant toughness reduction
- Rules drafted to cover HTR piping, pumps and valves.
- Alloy 617 bolting is not presently included; also effect of cold work on properties
- Design assessments may be limited to a design life of 100,000h or less.
- Tertiary creep (has been) eliminated as a basis for definition of St in draft of IN617 Rules
- Smt rather than So may be used for components with short lives
- Level D limits in Appendix F have been modified for plastic instability/collapse)
- For certain materials (Alloy 617) simplified ratchetting rules may only be applicable at 650°C and below.

The report references presentations made at the gif VHTR materials Project Management Board on the subject.

6.4 Database developments within ODIN

The Materials Database (Mat-DB) has been established within the Petten ODIN Portal and was chosen by RAPHAEL partners as the preferred place for storage of the results from the RAPHAEL materials programme. Mat-DB provides a web-interface for data content, data entry, data retrieval and analysis routines and already contains the experimental alloys data from the fifth Framework HTR-M project [Ref. 6-12]. MAT- DB holds information on experimentally measured materials data from many European R&D activities including mechanical and thermo-physical properties of engineering alloys at low, elevated and high temperatures for base materials and joints and irradiation materials testing results for both fusion and fission applications. It also holds information on thermal barrier coatings for gas turbines and mechanical properties testing on corroded specimens.

The work carried out on the implementation and development of Mat-DB database for the RAPHAEL project is part of a voluntary database support activity provided by JRC

A graphite database structure has been implemented in MAT-DB [Ref. 6-13]. The database structure has been agreed with the graphite partners and has been oriented from existing HTR-M/M1 datasheets. A CMC database structure has also been set up in MAT-DB in agreement with the composites partners. Both these structures provide the means for electronic storage of the irradiated and non-irradiated results form the RAPHAEL and EXTREMAT projects.

The description and the procedure for accessing Mat-DB is given in [Ref. 6-14] and an on-line procedure available for entering RAPHAEL data.

6.5 Support to SP CT

An important part of the SP4 ML activities is to provide materials support to the RAPHAEL components work performed in SP5 CT. This has included the provision of materials information on graphite (tribology tests) and metallic materials for the Gas Circulator and IHX. In relation to Codes and Standards issues, materials design information has been provide on IN 617 [Ref. 6-15] to support the finite element analysis work being performed on the IHX.

6.6 Overall Conclusions on Design Codes

The activities on Design Codes within SP CT and a summary of the findings within WP4 ON Codes and Standards are reported [Ref. 6-16]. The work completed for high temperature metals within Work package 4 of RAPAHEL SP4 ML includes:

Development of Graphite Guidelines

Within Europe, for the VHTR, it is proposed to house the graphite data and design methodology in a similarly developed compendium to that used for the UK and steps to achieve this have been outlined. The results from the RAPHAEL experiments will be used as a basis for the property sections. The main sections however would be similar to the ASME code which are currently under developed, by an ASME Project Subgroup.

Development of Composite Guidelines

The main sections of the proposed composites guidelines, are to follow a similar structure to the ASME graphite Code but with their content adjusted to comply with Composite material requirements

Codes and Standards for Metallic materials

Research and Development (R & D) recommendations have been made for material issues and for Design Code omissions for the VHTR and its components.

A report on Creep/Fatigue (Interaction) of Mod 9Cr-1Mo Steel for vessel application has been produced including recommendations for future work

Outline rules /guidelines for Nickel based Alloys have been reviewed and recommendations made for further work

6.7 References on Design Codes

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6-3 M W Davies: Development of Design Data and Guidelines or Graphites – Status Report. RAPHAEL-0710-S-ML4-3
6-4 GIF VHTR Materials Project Arrangement, RAPHAEL-0904-S-ML0-x
6-5 M W Davies: Development of Design Data and Guidelines for Composites – Scope and Planning, RAPHAEL-0606-D-ML4-1
6-6 M W Davies: Development of Design Data and Guidelines for Carbon Composites – Status Report. RAPHAEL-0710-S-ML4-4
6-7 D Buckthorpe: HTR-M Final Report - HTR-M 04/12 P 0 0 105
6-8 J Ali & D Buckthorpe: R&D Recommendations to sup[port design data and design rule omissions for the VHTR: RAPHAEL-0611-S-ML4-6
6-9 D Buckthorpe: Report on Creep/Fatigue (Interaction) of Mod 9Cr-1Mo Steel for vessel application – Review and Recommendations for Future Work: RAPHAEL-0704-S-ML4-7
6-10 J Ali & D Buckthorpe: Prioritising of short comings in National & International Design Codes: RAPHAEL-0611-S-ML4-8
6-11 J Ali & D Buckthorpe: Rules /guidelines for Nickel based Alloys – Outline: RAPHAEL-0804-S-ML4-5
6-12 H Over: Development of MAT-DB Database within Raphael – scope of work: RAPHAEL-0603-S-ML4-1
6-13 H Over: Development of MAT-DB Database within Raphael – Progress Report: RAPHAEL-0704-S-ML4-2
6-14 H Over: Development of MAT-DB Database within Raphael: RAPHAEL-0804-S-ML4-5
6-15 B-C Friedrich, J Ali: Material datasheet of Alloy 617(NiCr 23 Co 12 Mo): RAPHAEL-0710-S-ML2-1
6-16 D Buckthorpe, M Davies, Synthesis of results within WP4 on Design Rules, RAPHAEL 0904 D ML4.6

7 Overall Summary of SP4 Activities in Support of its Work Programme

7.1 Project Management, Education and Communication

The work described in sections 2 to 6 summarise the technical results and achievements produced within SP4 materials work packages of RAPHAEL. This section summarises the management, Education and Communication actions which have taken place in order to achieve the work.

Summary of Meetings

Technical meetings and six monthly progress meetings have taken place [Refs. 7-1 to 7-20] organised to bring graphite/ composite and metals specialists together on successive days. Management exchanges have also taken place at Executive Board meetings and Annual Steering Committee Meetings and presentations given on work progress.

Collaboration activities

Exchanges have taken place with EXTREMAT-IP on materials for the VHTR and have been summarised in [Ref. 7-21 to 7-23]. The primary interests for VHTR have been on protection barriers, irradiation resistant materials, and bonding technologies. The main exchange activities have been for the development of a composite material for the control rod, for advanced metallic materials developments for the IHX and core support (including ODS material) and for diffusion barrier requirements particularly for hydrogen generation. The EXTREMAT collaboration has provided an opportunity for new composite materials to be tested at representative irradiation conditions in the HFR. Other areas of exchange on ODS and TiAl alloys have benefited material investigations on the VHTR IHX and core support structures. The potential for utilising tritium barrier developments being made for fusion for VHTR hydrogen production application has also been highlighted and SiC material for the VHTR IHX and GFR core. The findings and conclusions from the collaborative exchanges between RAPHAEL and EXTREMAT have been discussed at RAPHAEL progress meetings and presented at RAPHAEL Steering Committee Meetings.

Exchanges have also taken place with the GCFR STREP on materials and components work within RAPHEL providing GCFR STREP deliverable D15-GCFR on materials requirements and material R&D requirements [Ref. 7-24].

Education

Support has been given to the third of the three Eurocourses organised by RAPHAEL in which a presentation was given on the materials investigation for the VHTR Reactor Pressure Vessel

Communication

Several papers have been presented at international conferences including the HTR conferences (HTR2002 to HTR2010). The submitted papers have been loaded on to the SINTER website [Ref. 7-25 to 7-33].

7.2 References on Project Management, Education and Communication

7-1 D Buckthorpe, Minutes of Raphael-IP Materials SP Kick-off Meeting, RAPHAEL-05/04-M-ML0-1

7-2 D Buckthorpe, Minutes of 1st progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL-05/10-M-ML3-1

7-3 D Buckthorpe, Minutes of 1st progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL-05/10-M-ML1-1

7-4 D Buckthorpe, Minutes of 2nd progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL-0605-M-ML3-2

7-5 D Buckthorpe, Minutes of 2nd progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL-0605-M-ML1-2

7-6 D Buckthorpe, Minutes of 3rd progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL-0611-M-ML1-3

7-7 D Buckthorpe, Minutes of 3rd progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL-0611-M-ML3-3

7-8 D Buckthorpe, Minutes of 4th Progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL 0706 M ML1.4

7-9 D Buckthorpe, Minutes of 4th progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL 0706 M ML3.4

7-10 D Buckthorpe, Minutes of 5th Progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL 0710 M ML1.5

7-11 D Buckthorpe, Minutes of 5th Progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL 0710 M ML3.5

7-12 D Buckthorpe, Minutes of 6th Progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL 0806 M ML1.6

7-13 D Buckthorpe, Minutes of 6th Progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL 0806-M-ML3.6

7-14 D Buckthorpe, Minutes of 7th Progress Meeting on Materials Development – Graphite & Composite Materials, RAPHAEL 0902-M-ML3.7

7-15 D Buckthorpe, Minutes of 7th Progress Meeting on Materials Development – Vessel and High Temperature Materials, RAPHAEL 0902 M ML1.7

7-16 D Buckthorpe, Minutes of 8th Progress Meeting on Materials Development –

Graphite Materials,RAPHAEL-1002-M-ML3-8
7-17 I Hugon, Specialist Meeting on Materials for Control Rod, RAPHAEL-06/09-M-ML2-2
7-18 D Buckthorpe, Brief notes of a 1st specialist meeting on VHTR IHX materials and components, RAPHAEL-0609-M-ML2-3
7-19 D Buckthorpe, Brief notes of a 2nd specialist meeting on VHTR IHX materials, RAPHAEL-0703 M-ML2.4
7-20 J Hegeman, Brief notes of a 1st Specialist Meeting on VHTR Vessel materials, RAPHAEL 0701 M ML1.8
7-21 D Buckthorpe, Joint Meeting with EXTREMAT-IP to discuss VHTR materials needs and requirements, RAPHAEL-06/03-M-ML2-1
7-22 D Buckthorpe, Minutes of a link meeting with EXTREMAT-IP on Fission- Fusion materials, RAPHAEL-0702-M-ML0-2
7-23 D Buckthorpe, et al., Report on RAPHAEL /EXTREMAT-IP exchanges, RAPHAEL 0912 S ML0.13
7-24 D Buckthorpe, Specific Materials Requirements and Material R&D Requirements for GFR Materials, (GCFR-DEL-015), RAPHAEL 0912 S ML0.12
7-25 D Buckthorpe, et.al RAPHAEL Project: Materials development for the VHTR – results from HTR-M & HTR-M1 projects plus future RAPHAEL programme, RAPHAEL 0610 S ML0.1
7-26 D Buckthorpe, RAPHAEL: Materials and Components highlights from the HTR FP5 and FP6 Programmes, RAPHAEL 0705 S ML0.2
7-27 D Buckthorpe, Material requirements for the very high temperature reactor – results and progress within the RAPHAEL-IP, RAPHAEL 0706 S ML0.3
7-28 D Buckthorpe, Materials for the Very High Temperature Reactor – results and progress within the Fifth and Sixth Framework Programmes – Special Issue – Journal of Advanced Material Research, RAPHAEL 0806 S ML0.4
7-29 D Buckthorpe, Materials for the Very High Temperature Reactor – results and progress within the Fifth and Sixth Framework Programmes – EXTREMAT Conference Poster Presentation, RAPHAEL 0806 S ML0.5
7-30 J Ali, D Buckthorpe, et al., Comparative study of predictive Finite Element methods for mechanical properties in 2D woven Carbon/Carbon Composites, RAPHAEL 0812-S-ML0.6
7-31 D Buckthorpe, E Breuil, HTR2008 - RAPHAEL: Achievements within the VHTR Materials and Components programmes, RAPHAEL 0812-S-ML0.7
7-32 D Buckthorpe, et al. Material Challenges of the new Advanced Gas Cooled Systems, RAPHAEL 0908 S ML0.11
7-33 D Buckthorpe, Jean-Sebastian Genot, RAPHAEL: Synthesis of Achievements on Materials and Components and Future Direction, RAPHAEL 1004 S ML0.14