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Progress of Graphite Irradiation tests at higher temperature

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

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Status of the irradiation experiments on selected graphite grades in RAPHAEL-IP - ***Progress of Graphite Irradiation tests at higher temperature***

Abstract

Within the RAPHAEL-IP framework, irradiation experiments on graphite are being performed at 750 °C and 950 °C in the High Flux Reactor (HFR) in Petten. This report reviews the progress of the experiments, including the selected major and minor graphite grades, details of the specimens, and future planning for the remaining of the project.

Revisions

Rev.	Date	Short description	Author	WP Leader	SP Leader	Coordinator
01	31/01/2006	First issue	O. Wouters NRG	Derek Buckthorpe NNC	Derek Buckthorpe NNC	Edgar Bogusch, ANP

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1 Introduction

Graphite is a suitable material to be used as a neutron moderator and reflector in nuclear reactors. Graphite has been used in Advanced Gas-Cooled Reactors, Magnox reactors, RBMK's (a Russian acronym meaning "reactor cooled by water and moderated by graphite", Research Reactors, Materials Testing Reactors and High Temperature Reactors (HTR's). Recently, there has been a renewal of interest in HTR's. Two prototypes, one in Japan (HTTR) and one in China (HTR-10) are operated. The development of the Pebble Bed Modular Reactor (PBMR) in South Africa and the Gas Turbine Modular Helium Reactor (GT-MHR) are at an advance stage. The European Commission is also supporting research projects for the development of HTR technology with the aim to create the technological requirements for designing and constructing an HTR in Europe.

Much research has been done on the behaviour of graphite grades in a nuclear reactor environment, because graphite is already being used in reactors for decades. However, these graphite grades are no longer commercially available because the raw materials do not exist anymore. In addition, most data from the past are from low temperature experiments (<550 °C), whereas for HTR's the graphite temperatures will generally be higher than 550 °C. It is decided to create a database that contains the data of the materials behaviour of available graphite grades under neutron irradiation at HTR relevant temperatures, in order to be able to design a European HTR. This will allow the 'best' graphite(s) to be chosen, and provide all the data necessary to allow a full core design to be carried out.

Within the RAPHAEL-IP framework, irradiation experiments will be performed nominally at 750 °C and 950 °C in the High Flux Reactor (HFR) in Petten. In these experiments a few graphites will also be irradiated at lower temperatures of 650 °C and 850 °C respectively. The distribution of specimens will be decided prior to final assembly. Four graphites have been selected to be tested as major grades. Two of these are produced by SGL Carbon and two by Graftech. In addition a list is made of minor graphite grades to be included in the experiments with fewer samples. These will also include graphites produced by Toyo Tanso, and were supplied through an agreement with JAERI. The following material properties are measured and compared before and after irradiation: specimen dimensions, dynamic Young's modulus, coefficient of thermal expansion and thermal diffusivity).

A start has been made with the irradiation experiment at 750 °C carried out under HTR-M1 as part of the 5th Framework Programme. The remainder of the post irradiation examinations is included in the RAPHAEL-IP project.

This report gives an update on the preparations for the three planned irradiations experiments.

2 Description of experiments

2.1 Irradiation programme

Figure 1 shows schematically the expected volume change with neutron fluence at the two main irradiation temperatures. The boxes on the curves indicate the four different irradiation experiments.

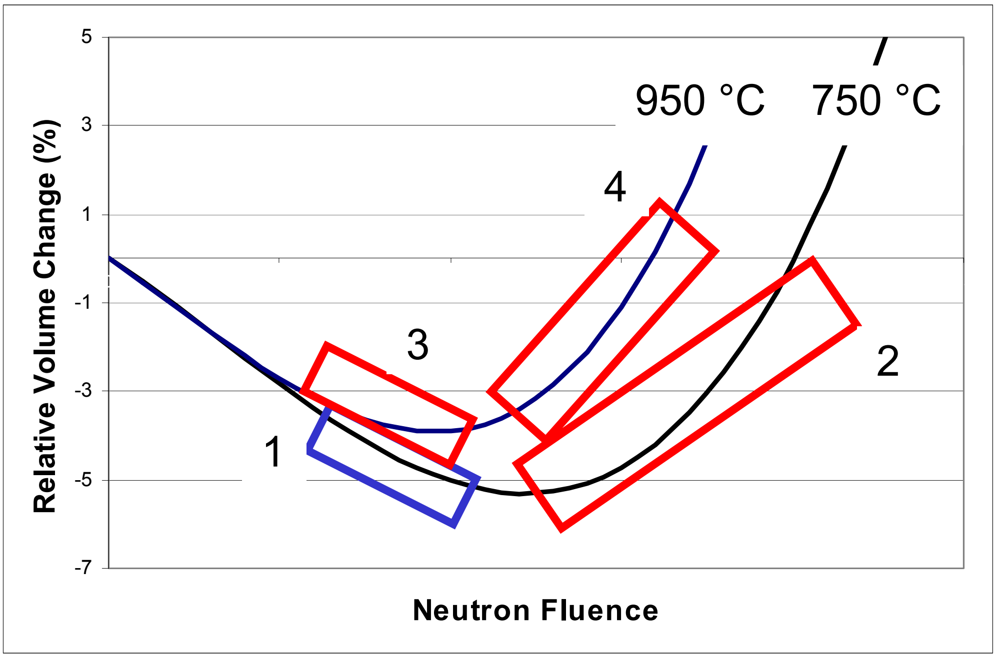


Figure 1 Schematic overview of the different irradiation experiments

Box 1, the low dose irradation at 750 °C named INNOGRAPH-1A has been performed under HTR-M1 in the 5th Framework Programme and had a target dose of 8 dpa. Box 2, named INNOGRAPH-1B is the high dose (up to ~25 dpa) experiment at the same temperature. Boxes 3 and 4, designated as INNOGRAPH-2A and INNOGRAPH-2B, represent respectively the low to medium dose and the high dose irradiation experiments at 950 °C. In order to reach the target dose in as short a time as possible, the high dose experiments (INNOGRAPH-1B/2B) will partly contain specimens, which have been irradiated in the first stage. Those specimens will determine the property changes like dimensional change beyond cross-over.

2.2 Graphite grades

The selection of the graphite grades for the RAPHAEL-IP programme is based on factors such as thermal and mechanical properties, impurity levels and availability. The four major grades, produced by SGL Carbon and Graftech are chosen in such a way that the graphites cover a variety of microstructures. This is achieved by selecting grades based on different raw materials, i.e. coal tar pitch coke or petroleum coke, and manufacturing methods, i.e. extrusion or vibro-moulding.
The minor grades include graphites from three different manufacturers, SGL Carbon, Graftech and Toyo Tanso. Among these minor grades are iso-moulded graphites.
As compared to the material selection in the HTR-M1 project, two new materials have become available from SGL: grade NBG-18 is the pre-pilot production for the PBMR project, and grade NBG-17 is similar but with a 2 times smaller filler grain that would make that grade more suitable for fine details like in prismatic blocks.The BAN grade was added mainly for scientific reasons.

The graphite grades selected are listed in Tables 1 and 2.

Table 1 Selected graphite grades: major grades

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

Table 2 Selected graphite grades: minor grades

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

Samples are taken out of the edge as well as the centre of the graphite blocks, half the pills having the *with grain* (WG) orientations parallel to the specimen axis and the other half the *against grain* (AG) orientation.

2.3 Specimen geometry

The specimen shape is cylindrical. The cylindrical specimens are flattened by milling a plane with a width of 3 mm along the length. This plane is used to engrave the specimen number and fix the orientation of the specimen. A drawing of the specimen shape is shown in figure 2. The diameter of all specimens is 8 mm and the length is either 6 mm or 12 mm. These sizes are small enough to allow a sufficient number of specimen in the irradiation rig, and big enough to perform reasonably accurate measurements. The specimens with a length of 6 mm are suitable for type of measurements (dimensions, dynamic Young's modulus, coefficient of thermal expansion and thermal diffusivity coefficient). The thermal diffusivity coefficient measurements cannot be performed on the 12 mm specimens because a length of 12 mm is too long. These samples are included to have some data points with higher measurement accuracy in mainly the dimensional change measurements.

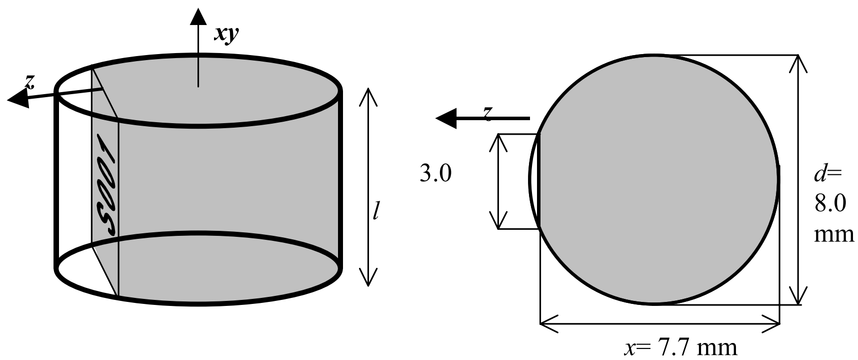


Figure 2 The specimen geometry. The figure on the left-hand side exaggeratedly shows the flatting of the cylinder. The milled plane is perpendicular to the z-direction when the axial direction of the cylinders in the xy-direction. The specimen number is engraved on the plane. The figure on the right-hand side is a top-view of the specimen. The important dimensions are indicated.

2.4 Irradiation rig

The irradiation rig used for INNOGRAPH-1A consists of eight drums made of TZM, a Ti-Zr-Mb alloy, that allows effective use of the in-reactor gamma-heating. Each drum contained four stacks of graphite pills as shown in figure 3. To account for the swelling expected in INNOGRAPH-1B/2B and in order to achieve the correct temperature of 950 °C in INNOGRAPH-2A/2B, the number of stacks per drum has to be lowered to three, thereby reducing the total number of specimens per experiment to about 150. The desired temperature can be achieved in total in seven drums. It is possible to design one extra drum at the top of the rig, where 950˚C cannot be achieved. To make full use of this possibility, the decision is made to isolate this drum by means of a heat shield and design the drum such as to obtain a temperature exactly 100 °C lower than in the other seven drums.

Figure 3 TZM drum and its loading that consists of four columns of graphite specimens (INNOGRAPH-01A).

2.5 Test matrix

Table 3 and 4 list the number of specimens for each graphite grade for the INNOGRAPH-1B and INNOGRAPH-2A experiments. The final number of specimens will depend on the final design of the irradiation rigs. In table 3 only the fresh samples are listed. The experiment will be complemented with samples, already irradiated in INNOGRAPH-1A, to reach the target dose of 25 dpa in as short a time as possible. Which specimens will be re-irradiated, will depend partly on the results from the post-irradiation experiments and will be decided in the near future.

Table 3 Number of fresh specimens per grade selected for inclusion in INNOGRAPH-1B

Grade	Manufacturer	Status	Coke	Process	Estimated no. of specimens
NBG-10	SGL	Major	Pitch	Extrusion	14
NBG-25	SGL	Minor	Petroleum	Iso-moulding	6
NBG-18	SGL	Major	Pitch	Fibro-moulding	14
NBG-17	SGL	Minor	Pitch	Fibro-moulding	6
PCEA	Graftech	Major	Petroleum	Extrusion	14
PCIB	Graftech	Minor	Petroleum	Iso-moulding	6
PPEA	Graftech	Major	Pitch	Extrusion	14
BAN	Graftech	Minor	Needle	Extrusion	6
IG-110	Toyo Tanso	Minor	Pitch	Iso-moulding	6
IG-430	Toyo Tanso	Minor	Petroleum	Iso-moulding	6

Table 4 Number of specimens per grade selected for inclusion in INNOGRAPH-2A

Grade	Manufacturer	Status	Coke	Process	Estimated no. of specimens
NBG-10	SGL	Major	Pitch	Extrusion	28
NBG-25	SGL	Minor	Petroleum	Iso-moulding	12
NBG-18	SGL	Major	Pitch	Fibro-moulding	28
NBG-17	SGL	Minor	Pitch	Fibro-moulding	14
PCEA	Graftech	Major	Petroleum	Extrusion	28
PCIB	Graftech	Minor	Petroleum	Iso-moulding	16
PPEA	Graftech	Major	Pitch	Extrusion	28
BAN	Graftech	Minor	Needle	Extrusion	6
IG-110	Toyo Tanso	Minor	Pitch	Iso-moulding	8
IG-430	Toyo Tanso	Minor	Petroleum	Iso-moulding	6

3 Progress report

3.1 INNOGRAPH-1B

INNOGRAPH-1B consists of approximately 80 irradiated and 80 fresh specimens. The post-irradiation experiments of INNOGRAPH-1A are ongoing. The dimensional measurements are approximately for 75% completed. The same holds for the dynamic Young's modulus measurements. All masses are measured. Measurement of the thermal properties will start in February 2006.
The fresh specimens are made and the measurements of the dimensions and the thermal diffusivity are ongoing. Measurements of the coefficient of thermal expansion are finished.

The capsule design for the irradiation experiments is nearly finished. Calculations are made to determine the exact drum shape to obtain a homogeneous temperature throughout the capsule, with the exception of the final drum, which will be at a temperature of 650 °C.

Irradiation is scheduled to start in August 2006.

3.2 INNOGRAPH-2A

For INNOGRAPH-2A all pre-irradiation characterisations have been performed, with the small exception of the BAN samples, since this graphite has been received only recently.

The rig design is finished. Thermal analysis has shown that seven drums will reach the target temperature of 950 °C and one isolated drum will be maintained at a temperature of 850 °C.

Irradiation is scheduled to start in May 2006.

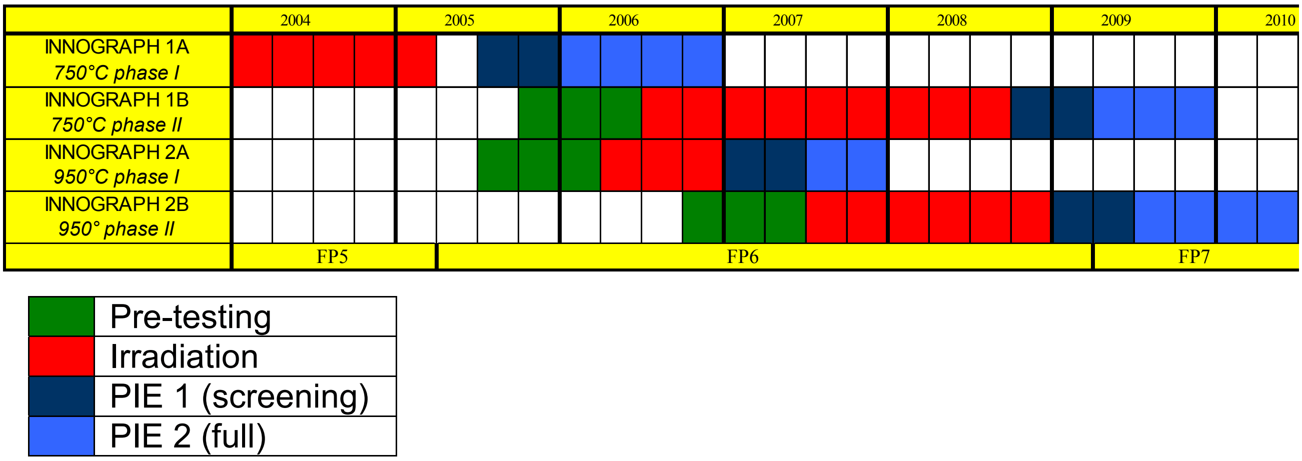
3.3 Microstructural development and modelling

The current work connects to the development of understanding the irradiation behaviour of graphites, and supports the initiatives to model that behaviour. These considerations and associated actions are not detailed here, but will appear in the revision 2 of this report.

4 Planning

In table 5, the planning for the coming years of the programme is shown. Pre-testing for the experiments described in this report are nearing completion. The indicated duration of the irradiation may change if the response of the graphites under irradiation is faster or slower than expected. The first post-irradiation experiments are indicated as being a first screening. A fast analysis of the data may be important in the preparation of following experiments e.g. to be able to make an early selection of the graphites.

Table 5 Planning of the different INNOGRAPH experiments



5 Concluding remarks

The activities in RAPHAEL IP are performed with financial support from the European Commission and the Netherlands Ministry of Economic Affairs. They are being considered within the EU contribution to the Generation-4 International Forum.