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Progress on continuation of the 750°C graphite irradiation test

Author(s):

J.A. Vreeling (NRG)

Contributors

J B J Hegeman (NRG)
O Wouters (NRG)

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





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Progress on continuation of the 750°C graphite irradiation test

Abstract

Reloading pre-irradiated graphite samples are foreseen in Raphael IP. The reason for this is to get graphite property information at different levels of irradiation damage. Two experiments are needed per irradiation temperature. The first irradiation experiment with pre-irradiated samples is INNOGRAPH-1B. The challenge of loading radioactive samples in an irradiation experiment is that the loading and assembling has to be performed in a shielded environment. This report describes the progress and plans for designing, loading and assembling INNOGRAPH-1B.

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**Designing, loading and
assembling an irradiation
experiment with radioactive
samples**

*DML3.1: Progress on continuation of
the 750°C graphite irradiation test*

J.A. Vreeling
J.B.J. Hegeman
O. Wouters

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Summary

Reloading of pre-irradiated graphite samples is foreseen in Raphael IP. The reason for this is to get graphite property information at different levels of irradiation damage. Two experiments are needed per irradiation temperature. The first irradiation experiment with pre-irradiated samples is INNOGRAPH-1B. The challenge of loading radioactive samples in an irradiation experiment is that the loading and assembling has to be performed in a shielded environment. This report describes the progress and plans for designing, loading and assembling INNOGRAPH-1B.

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.

It is decided in 2001 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 (SP-Materials development), irradiation experiments will be performed at 750°C and 950°C in the High Flux Reactor (HFR) in Petten. In every experiment a few graphites will also be irradiated at the lower temperature of 680°C and 850°C respectively. A start has been made with the irradiation experiment at 750°C carried out under HTR-M1 as part of the 5th Framework Programme.

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. 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.

Data points at different levels of irradiation damage are needed to get useful design curves (material property as a function of irradiation damage). The dose range within one irradiation experiment is ~65%-100% of the peak dose, due to flux difference between the centre and ends of the irradiation capsule, the so-called flux-buckling. The maximum dose of interest is 25 dpa (estimated end-of life dose). One irradiation experiment only gives information between ~16 and 25 dpa, which is not sufficient to get a design curve. Therefore it is decided to have two irradiation experiments to produce these design curves. The first experiment is targeted at one third of end of life dose, and the second experiment, that contains fresh unirradiated samples and irradiated samples from the first experiment, is targeted at two thirds of end of life dose. In this way information will be generated at 1/3, 2/3 en 3/3 of end of life dose. Data points between these dose levels will be obtained by making use of the flux buckling.

It is common practice to perform the loading and assembling of an irradiation experiment on a table in a normal lab. However, in this case radioactive samples are loaded in the second experiment and radiation protection measures have to be taken. Loading the samples, assembling the irradiation capsule and loading the experiment in the HFR is therefore complicated because most work has to be done without having direct contact with the experiment. The design, loading and assembling of INNOGRAPH-1B (the second stage experiment at 750°C) is described in this report.

2 INNOGRAPH-1B

2.1 Design

The design is based on the first irradiation experiment INNOGRAPH-1A (750°C, first phase).

There are two important points that are different for the second phase experiment. The first one is that the loading should be performed in a lead-shielded cell. The design should therefore be kept simple. The design of INNOGRAPH-1A (a stack of eight drums of a Molybdenum alloy, having four vertical holes in which the graphite samples are loaded) is already based on future reloading, and therefore can be used for INNOGRAPH-1B.

The second point is that this experiment contains samples that will be irradiated to end of life dose. At these dose levels the samples could swell and therefore the samples should have sufficient space. In INNOGRAPH-1A the samples were only shrinking. The diameters of the holes in the drums of INNOGRAPH-1B need to be larger than INNOGRAPH-1A, resulting in space for three stacks of graphite samples instead of four.

The maximum stack height of INNOGRAPH-1B is 400 mm. This is based on the height over which the target irradiation temperature (750°C) can be reached. The stack consists of six drums of 60 mm and one drum of 30 mm in height. It is decided to place one extra drum of 60 mm on top of this with a lower target temperature (680°C). This drum is positioned outside the 400 mm, but at this vertical position there is still a neutron flux high enough to reach the target temperature. However, the temperature variation in this drum will be higher. The two temperature zones are separated by a thermal barrier.

The eight drums are placed in a containment tube. The graphite samples are physically separated from the TZM drum by placing graphite foil between the samples and the drum material.

The experiment will be instrumented by 24 thermocouples, positioned at different vertical and radial positions, to measure the irradiation temperature. The experiment contains also nine activation monitor sets to measure the neutron dose.

The first containment will be flushed with helium. The temperature will be controlled with a He/Ne gas mixture in the second containment.

The INNOGRAPH-1B experiment is shown in figure 1.

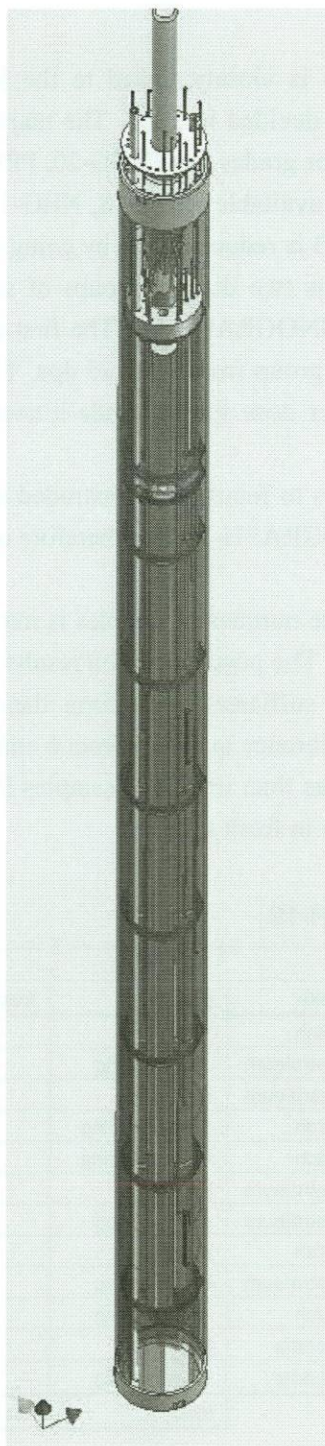


figure 1 Schematic drawing of INNOGRAPH-1B. The lower seven drums are targeted at an irradiation temperature of 750°C. The drum at the top is an extra drum, targeted at 680°C. In each drum three stacks of graphite samples are present

2.2 Loading

The loading of INNOGRAPH-1B is closely linked to the loading of INNOGRAPH-1A. The loading of INNOGRAPH-1A was decided in 2002. The major grades were: NBG-10, NBG-25, PCEA, PCIB and IG-110. The minor grades were NBG-20, PPEA and IG-430.

In the year 2005 new grades were available (NGB-18, NBG-17, LPIB and LPEB). On the other hand the space in INNOGRAPH-1B is reduced 25 % by going back from four to three columns. In addition INNOGRAPH-1B contains two distinct groups of samples: those that are unirradiated and specimens pre-irradiated in INNOGRAPH-1A. The first group is needed to get information from 10 to 16 dpa, and the second group from 18 to 25 dpa. These three arguments: more grades, less space and the need for a larger dose spread, made it necessary to reconsider the loading of INNOGRAPH-1B.

It was decided with project partners to focus on the extruded and fibro-moulded grades. The new grades were not irradiated in INNOGRAPH-1A and therefore only fresh specimens are loaded.

The loading is given in table 1. The number of samples is maximised by reducing the number of specimens with a length of 12 mm. The post irradiation results of INNOGRAPH-1A show that the accuracy of the measurements is sufficient to perform them on 6 mm samples. There is no evidence found that there was a difference in results from 6 and 12 mm samples.

There are more unirradiated samples than irradiated samples in the test matrix. The reason is that the four new grades only contribute in fresh samples.

table 1 Loading of INNOGRAPH-1B

Grade	supplier	status	coke	proces	no. of specimens		
					fresh	irradiated	total
NBG-10	SGL	Major	Pitch	Extrusion	15	17	32
NBG-25	SGL	Minor	Petroleum	iso-moulding	6	5	12
NBG-20	SGL	Minor	Petroleum	Extrusion	0	4	4
NBG-18	SGL	Major	Pitch	fibro-moulding	14	0	14
NBG-17	SGL	Minor	Pitch	fibro-moulding	14	0	14
PCEA	UCAR	Major	Petroleum	Extrusion	13	16	30
PCIB	UCAR	Minor	Petroleum	iso-moulding	6	7	12
PPEA	UCAR	Major	Pitch	Extrusion	14	13	27
IG-110	Toyo	Minor	Petroleum	iso-moulding	6	6	12
IG-430	Toyo	Minor	Pitch	iso-moulding	6	6	12
LPEB	UCAR	Minor	Needle	Extrusion	6	0	6
LPIB	UCAR	Minor	Needle	iso-moulding	2	0	2
total					103	74	177

The loading in the irradiation capsule is based on making clever use of the flux-buckling and starting dose of the samples. The vertical position of the individual samples is chosen by predicting the final dose after the irradiation experiment, based on the INNOGRAPH-1A experience. The optimised dose spread in the 750°C temperature zone is shown in figure 2. The two groups of samples (fresh and irradiated) are still distinguishable, however the gap is minimised. The irradiation damage between 5 and 10 dpa is covered by INNOGRAPH-1A.

Besides the 12 grades some piggy-backs samples are loaded: 9 Gilsocarbon samples and 6 samples containing coke material are added. The complete loading matrix is shown in table 2.

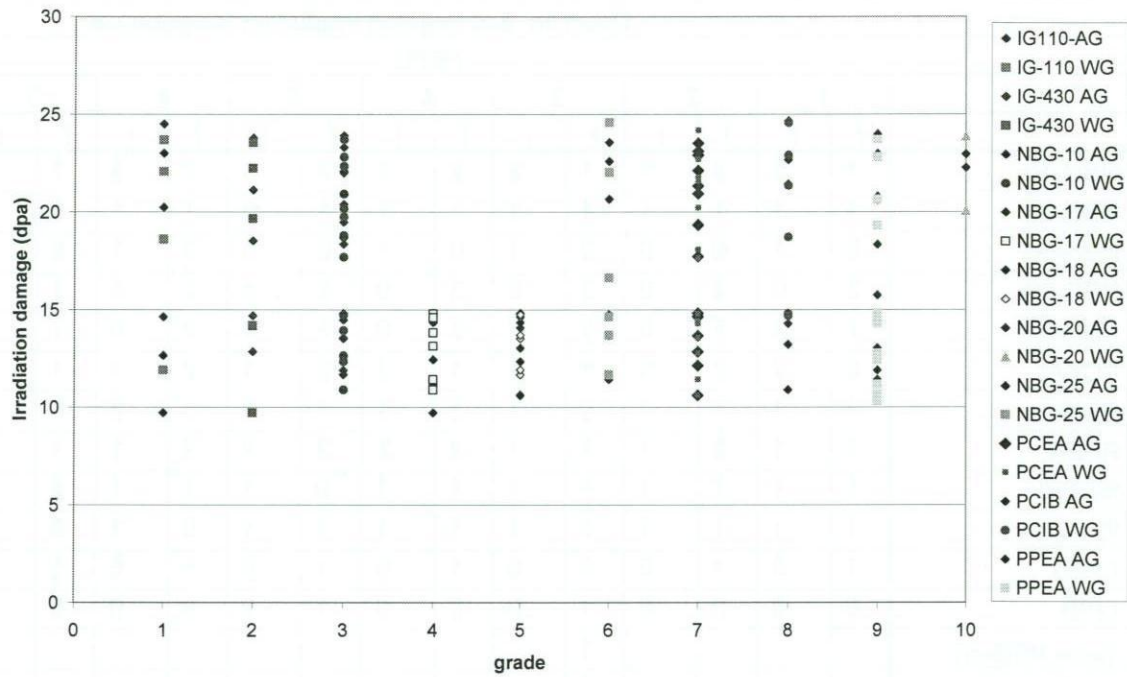


figure 2 Expected dose spread in after INNOGRAPH-1B irradiation, estimated by flux-buckling and starting dose

table 2 Loading of INNOGRAPH-1B per drum

Drum no. and targeted irradiation temperature																
750°C															680°C	
	1		2		3		4		5		6		7		8	
	f	i	f	i	f	i	f	i	f	i	f	i	f	i	f	i
NBG-10	1	3	3	1	1	2	2	2	3	3	2	3	1	2	2	1
NBG-25	1	1	1	1	1	1	1	1	1	0	1	1	0	0	0	0
NBG-20	0	1	0	0	0	1	0	1	0	0	0	1	0	0	0	0
NBG-18	2	0	2	0	2	0	2	0	2	0	2	0	1	0	1	0
NBG-17	2	0	2	0	2	0	2	0	2	0	2	0	1	0	1	0
PCEA	2	2	2	3	1	2	1	2	3	1	2	3	1	2	1	1
PCIB	1	1	1	1	0	1	2	1	1	1	0	2	0	0	1	0
PPEA	3	1	2	1	2	1	3	2	2	2	2	1	1	2	0	3
IG-110	1	1	1	1	0	1	1	1	0	1	1	1	0	0	2	0
IG-430	1	1	1	1	0	1	1	1	1	1	0	1	0	0	2	0
LPEB	1	0	1	0	0	0	1	0	1	0	1	0	0	0	1	0
LPIB	0	0	0	0	1	0	0	0	1	0	0	0	0	0	0	0
Coke NBG-10					1										1	
Coke UCAR					1										1	
Coke NBG-25					1										1	
Gilso	1	0	1	0	1	0	0	0	1	0	1	0	1	0	3	0

3 Loading and assembling an irradiation capsule containing radioactive samples

In INNOGRAPH-1B 74 irradiated samples from INNOGRAPH-1A are loaded. This means that measures have to be taken to protect the environment and the people working with or around the experiment from radiation. The total activity of the dominant nuclides, based on random checks of the active samples, is given in table 3.

table 3 Expected activity in INNOGRAPH-1B during loading and assembling

Nuclide	Activity (MBq)
Mn-54	3,55
Co-58	0,02
Co-60	3,37
Ag-110m	0,95
Cs-134	0,60

The loading and assembling of INNOGRAPH-1B is divided in two parts. The first part is handling with the specimens and pre-assembling of the rig in a lead shielded hot cell. The second part is final assembling of the experiment, while the active part of the experiment is shielded in a container.

3.1 Loading in hot cell

The samples are loaded in the drums in a lead shielded cell by using manipulators. The walls of the sample columns are covered with a graphite foil to protect the samples from contamination from the TZM alloy. The first step is loading the samples in the columns (figure 3).

The top of the drum is covered by graphite foil after the drum is filled, again to protect the samples from contamination (figure 4). A metal plate is placed on top of the foil. Finally, the drums are closed by fixing a screw (figure 5).

The drums will be placed over the thermocouples wires and gas tube onto the pre-assembled top end of the active part of the experiment, after all eight drums are filled. Once all thermocouples and drums are in place the outer containment tube will slide over all drums.

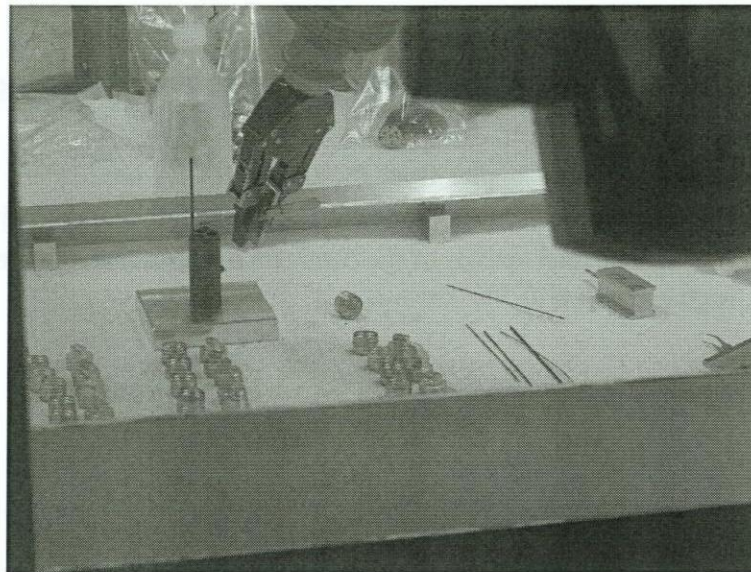


figure 3 Loading graphite samples in a drum

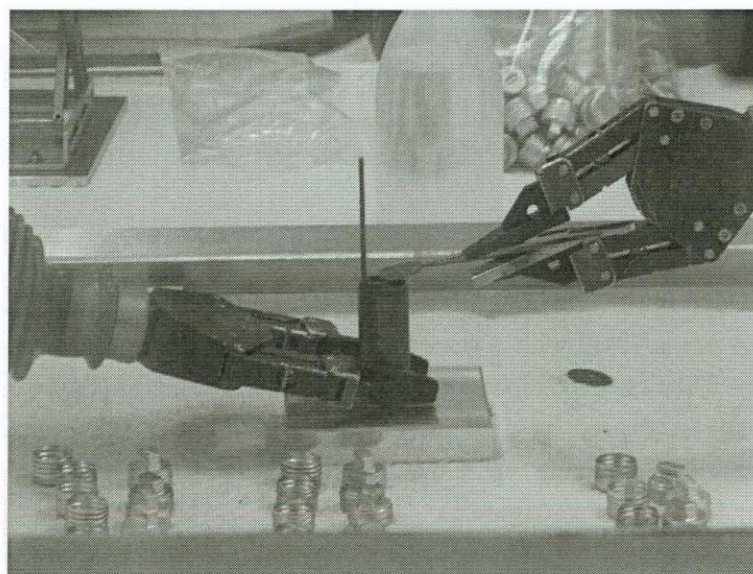


figure 4 Covering the drum by graphite foil and metal plate



figure 5 Closing the drum by screwing

3.2 Out-posting of the experiment

Once the pre-assembling work is done the experiment will be removed from the hot-cell (out-posting). In the back-wall of the hot-cell a so-called syntacs port is available. At the backside of the hot cell an extended syntacs container will be attached to the syntacs port. In this container a shielding module is placed. The shielding material is a tungsten alloy. The wall-thickness of the container is 1,65 mm. Conservative calculations show that the effective dose will be $10 \mu\text{Sv/h}$ at 10 cm and $0,4 \mu\text{Sv/h}$ at 1 m, which is allowable.

The experiment is placed in the shielding module in the syntacs container. The bundles of thermocouple wires and gas tube are placed in the syntacs container as well. After the syntacs container is closed it can be retracted from the hot cell. During the posting process radiation measurements will be performed to verify that the levels are within the limits.

3.3 Final assembling out-side the hot cell

Once the syntacs drum is disconnected it can be opened after which the shielding module with the sample holder will be taken out. Before taking it out, the radiation levels will be measured again. This will be done on the surface and front end of the shielding module after removal.

The shielding module will be placed on a construction that allows the module with the experiment to be rotated in axial direction in order to be able to weld the top lid of the sample holder to the containment tube. The person doing the welding will not place his hand directly on the shielding module in order to turn it during the welding phase, but can use the centering rings instead. Nevertheless finger dosimeters for monitoring the exposure are required. After welding of the containment tube an additional temporary shielding section can be applied to cover the front end to shield the radiation gap leaks through the thermocouples.

Next the extension to the upper section of the experiment will be welded to the rod that is part of the top lid of the containment. This is approximately 25 cm from the top lid of the experiment.

Again the experiment will be rotated by hand during welding. However this will be further away from the containment.

With the lower section well shielded and further away, the bundle of thermocouples will be stretched out and guided along the extension rod, through the biological shield and soldered to the 50 pins connector. The same goes for the gas tubes to which the gas couplings will be welded.

Once assembly of the total experiment is completed, tests will be performed on overall dimension, gas tightness and resistance of the thermocouples.

When the experiment is approved, the extension tube of the shielding module will be placed as well as the transport head. The experiment is now ready to be transported to the HFR facility.

3.4 Transport and x-ray inspection

The fully closed shielding module can be picked up by the standard HFR transport company and moved to the NDO facility for x-ray inspection of the containment weld. In order to do so the shielding module can be opened just behind the lower section, right where the weld is located, by disconnecting the extension tube. After the inspection is done, the tube will be reconnected to close the module. Next the module can be further transported to the HFR.

Once inside the HFR the crane can be used to carefully lift the module with the experiment into vertical position. The module will be moved to the pool and lowered into the water. Via the input device of the DM-cell the module will be moved into the DM-cell. The experiment will be taken out of the shielding module and placed in a leg of a standard irradiation TRIO capsule.

4 Outlook

Today's planning shows that the loading and assembling of INNOGRAPH-1B is finished by the end of 2006. The start of the irradiation experiment is foreseen in January/February 2007.

The start of INNOGRAPH-1B will be an important milestone in the graphite irradiation programme. It will confirm the idea that having the possibility of re-irradiating graphite samples will make it possible to produce full design curves in two irradiation experiments per irradiation temperature.

All developments made for the loading and assembling of INNOGRAPH-1B will be used for INNOGRAPH-2B, the 2nd phase irradiation experiment at 950°C in 2008.