



Graphite irradiation testing for HTR technology at the High Flux Reactor in Petten

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ABSTRACT

In 2001 the Nuclear Research and Consultancy Group started a large graphite irradiation program for the development of High Temperature Reactor technology in the European framework. The irradiation experiments, containing present day available graphite grades, are performed at the High Flux Reactor in Petten. The grades are NBG-10, NBG-17, NBG-18, NBG-20, NBG-25, PCEA, PPEA, PCIB, LPEB, IG-110 and IG-430. In the fifth framework programme (2001–2004) and sixth framework programme (2005–2009) four irradiation experiments are foreseen, resulting in design curves at irradiation temperatures between 650 °C and 950 °C. The post-irradiation testing is focused on dimensional changes, dynamic Young's modulus, coefficient of thermal expansion and coefficient of thermal conductivity. The irradiation programme and preliminary results from the first irradiation experiment at 750 °C to 8 dpa will be discussed in this paper.

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1. Introduction

The European Commission is supporting research projects for the development of High Temperature Reactor (HTR) technology. The materials projects HTR-M and HTR-M1 were implemented in 2001 in the fifth framework programme, with the original aim to create the technological requirements for designing a European HTR by 2011 [1,2]. The work is continued in the sixth framework programme in the RAPHAEL Integrated Project in 2005. The RAPHAEL Integrated Project is focused on the technology for a Very High Temperature Reactor, with helium outlet temperature well above 900 °C to potentially allow system integration with a chemical hydrogen production unit. A significant part of the materials research in these projects is focused on graphite issues [3].

The core of a HTR consists of graphite blocks. Under neutron irradiation the graphite undergoes dimensional changes and its material properties significantly change [4–7]. Graphite bodies initially shrink as a function of neutron damage due to the open microstructure of graphite. After a certain dose, the so-called turn-around dose, graphite starts to swell. This swelling continues significantly after it has reached its original size, the dose at cross-over.

Much research is done on the irradiation behaviour of graphite in the past and many data are available. Unfortunately this irradiation behaviour is not the same for all graphite grades. The microstructure of graphite is very inhomogeneous and strongly depends on the raw materials and production processes. The graphite grades that were investigated before are no longer commercially available and the raw materials do not exist anymore. Moreover,

most of the data of the past are from experiments with an irradiation temperature below 550 °C, whereas for HTR conditions the temperature in the graphite components will be higher than 550 °C. The renewal of interest in HTR technology makes it necessary to perform graphite irradiation programmes of present graphite grades at HTR relevant temperatures.

It was decided to start developing a database that contains the irradiation behaviour at HTR conditions of presently available graphite grades. The aim of this database is to generate a design base for a European HTR. This paper describes the programme and gives some preliminary results.

2. Description of the irradiation programme

One irradiation experiment, called INNOGRAPH-1A, was planned at the start of the programme in 2002. The nominal irradiation temperature was chosen to be 750 °C. This is about the temperature at the peak dose in the graphite core of a typical HTR. The target irradiation damage was 8 dpa (average sample dose), that is about one third of the end of life dose (dose at cross-over) estimated at 24 dpa. A second irradiation experiment at 750 °C, called INNOGRAPH-1B will be performed in RAPHAEL and will be targeted at 16 dpa. In this experiment a selection of samples from INNOGRAPH-1A (having a starting dose of about 8 dpa) will be re-loaded after being measured. Fresh unirradiated samples will be included in INNOGRAPH-1B as well. This experiment will result in data points at 16 dpa and 24 dpa. The gaps between 8, 16 and 24 dpa will largely be covered by making use of the flux buckling in the HFR (High Flux Reactor in Petten). Due to this flux buckling the neutron flux in the centre of the irradiation capsule is the highest and this decreases gradually to about 60% at both ends.

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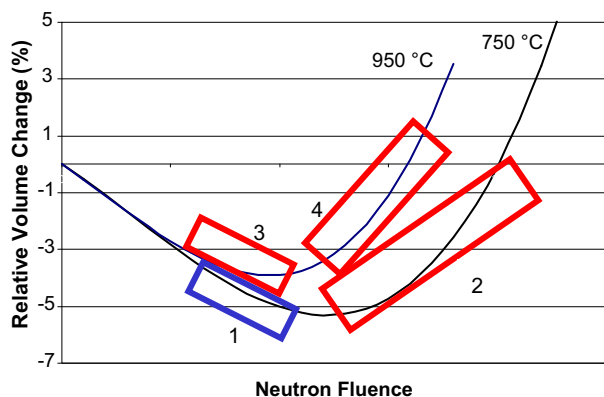


Fig. 1. Overview of the targets of the four irradiation experiments, one in HTR-M1 (in blue) and three in RAPHAEL-IP (red). 1 = INNOGRAPH-1A 8 dpa at 750 °C, 2 = INNOGRAPH-1B 16 dpa and cumulative 24 dpa at 750 °C, 3 = INNOGRAPH-2A 5 dpa at 950 °C, 4 = INNOGRAPH-2B 11 dpa and cumulative 16 dpa at 950 °C. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Two other irradiation experiments, INNOGRAPH-2A and INNOGRAPH-2B are performed at an irradiation temperature of 950 °C.

The graphite irradiation test programme in HTR-M1 and RAPHAEL-IP are summarised in Fig. 1, on the basis of a drawing of theoretical dimensional change curves.

Additional data points at a lower temperature than the target temperature are generated by making efficient use of the HFR core. This is done by adding extra irradiation volume at the top of each irradiation capsule. At this height the neutron flux is not high enough to generate the necessary gamma heat to reach the targeted temperature with the necessary accuracy; however, it is possible to achieve a temperature that is 50–100 °C lower than the target temperature.

3. Measurement techniques

The following properties of the graphite samples are of main interest in this program:

- Change in dimensions.
- Change in dynamic Young's modulus.
- Change in coefficient of thermal expansion.
- Change in coefficient of thermal diffusivity and thermal conductivity.

All the measurements of these properties are non-destructive. This is important because the measurements have to be performed before and after each irradiation experiment.

The design of the sample is chosen in such a way that all measurements can be performed on one sample, to use the irradiation volume as efficiently as possible. The sample size is mainly based on the limitations of the space in the irradiation capsule. Therefore small samples have to be used, keeping in mind that they are large enough to measure valid material properties with sufficient accuracy.

The samples are cylinders with a diameter of 8 mm and a height of 6 mm. Some samples with a height of 12 mm are included in the first irradiation for two reasons: first to improve the accuracy of the dimensional measurements in case the differences between the samples before and after irradiation would turn out smaller than expected, and second, to get some idea of possible size effects.

Sample dimensions were measured using Mahr inductive probes (Mahr, Type P 2104 MB) with a measurement range of

4 mm and an accuracy of 0.1 µm. The measurement is a comparison of the dimensions of a well-known caliber and the specimen. The volume of each specimen is calculated based on the measured dimensions. The mass of each specimen is determined with a Mettler AT261 scale with an accuracy of 0.01 mg.

The dynamic Young's modulus was determined by measuring the velocity of sound in the sample. The velocity of sound is determined with an Agfa-Krautkramer USM 25, using a nominal frequency of 5 MHz. The set-up is calibrated using a steel sample with a known velocity of sound. The dynamic Young's modulus is calculated by multiplying the density with the velocity of sound squared.

The coefficient of thermal expansion was measured using a Netzsch dilatometer DIL402C over the temperature range 25 °C irradiation temperature. Finally, thermal diffusivity was measured using the laser flash method on a Netzsch LFA457 MicroFlash again from room temperature to the irradiation temperature. Thermal conductivity values were derived from the thermal diffusivity data by multiplication with density and specific heat.

Besides the mentioned measurements, information on the microstructure will be given from microscopy and X-ray diffraction (crystallinity, crystal sizes). X-ray tomography will give some 3D information of the pore structure. At the end of the programme, when the samples have their final irradiation damage, it is possible to measure strength of the samples by diametric compression testing.

4. Material selection

The graphite grades that have been selected for the irradiation programme have to satisfy certain criteria. The first criterion for the graphite grade selection was availability. The grade should be available in large amounts, not only at that moment but also in the near future. The second criterion is that its properties are suitable for nuclear applications (high density, low anisotropy, high purity, etc.). Another consideration for the material selection was to cover a wide range of different graphite structures by selecting grades produced from two different coke materials (Petroleum and Pitch coke material) and two manufacturing processes: extrusion and iso-moulding.

The irradiation volume in INNOGRAPH-1A was large enough to contain five grades for which full design curves can be produced and three additional grades for which only trends are investigated. The first group is called Type A grades and the second group is called Type B grades. The only difference between these two classifications is the number of samples in the experiment.

Three graphite manufacturers have been involved in the selection process: two European Union based manufactures (SGL Carbon Group and GrafTech Int. Ltd.) and one Japanese (Toyo Tanso). Eight nuclear graphite grades from the three graphite manufacturers have been selected in 2002. The selection is shown in (Table 1). IG-110, used in the HTR-10 and HTRT, is the only graphite that is

Table 1
Material selection for the HTR-M1 project, made in 2002

Grade	Manufacturer	Coke	Process	Class in HTR-M1
PCEA	GrafTech	Petroleum	Extrusion	Type A
PCIB-SFG	GrafTech	Petroleum	Iso-moulding	Type A
NGB-10	SGL	Pitch	Extrusion	Type A
NBG-25	SGL	Petroleum	Iso-moulding	Type A
IG-110	Toyo Tanso	Petroleum	Iso-moulding	Type A
PPEA	GrafTech	Pitch	Extrusion	Type B
NGB-20	SGL	Petroleum	Extrusion	Type B
IG-430	Toyo Tanso	Pitch	Iso-moulding	Type B

This selection is used for the INNOGRAPH-1A irradiation experiment.

Table 2
Updated material selection for the RAPHAEL integrated project, made in 2005

Grade	Manufacturer	Coke	Process	Class in Raphael
PCEA	GrafTech	Petroleum	Extrusion	Type A
NGB-10	SGL	Pitch	Extrusion	Type A
PPEA	GrafTech	Pitch	Extrusion	Type A
NBG-18	SGL	Pitch	Vibro-moulding	Type A
PCIB-SFG	GrafTech	Petroleum	Iso-moulding	Type B
NBG-25	SGL	Petroleum	Iso-moulding	Type B
IG-110	Toyo Tanso	Petroleum	Iso-moulding	Type B
NGB-20	SGL	Petroleum	Extrusion	Type B
IG-430	Toyo Tanso	Pitch	Iso-moulding	Type B
NBG-17	SGL	Pitch	Vibro-moulding	Type B
LPEB	GrafTech	Needle Petroleum	Extruded	Type B

This selection is used for the INNOGRAPH-2A and INNOGRAPH-1B experiments.

presently available and from which irradiation data is available. The results from IG-110 will be the connection between this programme and experiments from the past.

At the start of the follow-up programme (RAPHAEL in 2005) the selection of 2002 is reconsidered. In 2005 new nuclear graphite grades were available, i.e. NBG-17 en NBG-18. There was also a renewed interest in BAN (LPEB) graphite. To be able to include these grades in the test matrix for the next three irradiation experiments the classification of the first eight grades had to be reassessed. The material selection of 2005 is shown in Table 2. This selection is used for the experiments INNOGRAPH-2A (started in 2006) and INNOGRAPH-1B (start foreseen in 2007). INNOGRAPH-2B foreseen in 2008 will probably have the same material selection, although there are possibilities to have some changes if new insights arise.

From the graphite blocks that have been supplied by the graphite producers, samples are taken from the centre region and edge region of the block to include the inhomogeneity of the product. A distinction between the two main directions in graphite (with grain and across grain) has been made as well.

A small amount of irradiation volume in each experiment is used to get some additional interesting information. Some samples of coke materials are included to study the irradiation behaviour of the raw materials. Also some samples of Gilsocarbon graphite, a material that is used in the advanced gas-cooled reactors in the United Kingdom. Furthermore, a few small pre-strained highly oriented pyrolytic graphite samples are irradiated.

5. Irradiation experiments

The irradiation experiments are performed in the High Flux Reactor (HFR) Petten.

The HFR is a tank-in-pool multipurpose test reactor with a thermal power of 45 MW. Its core arrangement of 9×9 positions contains 33 fuel elements, six control rods and 22 beryllium reflector elements, and provides effective 20 in-core positions. The HFR is currently operated in cycles of 27 days, with an accumulated 280–290 full power days per year.

The irradiation series 'INNOGRAPH' is designed with the experience gained from the earlier graphite irradiations and the fusion materials program. An INNOGRAPH capsule consists of a stack of eight metal drums (Fig. 2). Each drum has either three or four channels, depending on the experiment, for sample stacks. An example of the loading of a drum with four stacks (INNOGRAPH-1A) is shown in Fig. 3.

The capsules are instrumented with 24 thermocouples, placed on different axial and radial positions, to monitor the irradiation

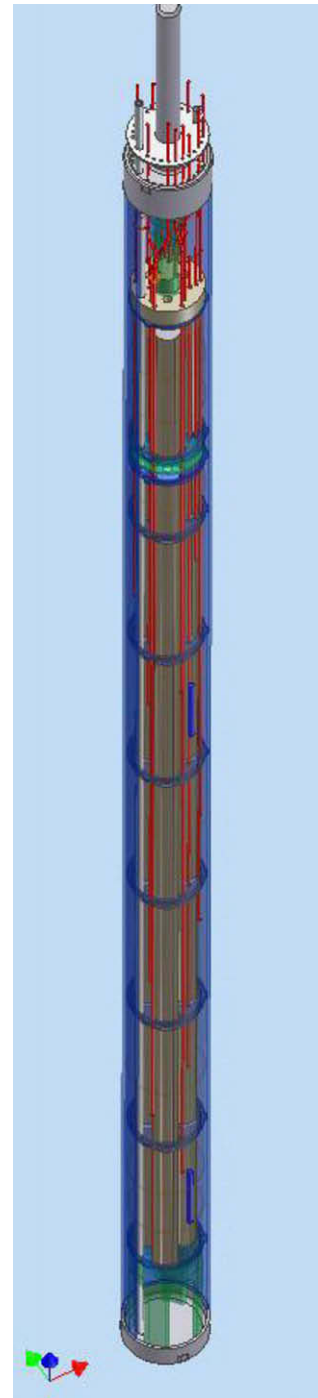


Fig. 2. Schematic drawing of a typical INNOGRAPH design.

temperature. The temperature can be controlled by changing the He/Ne gas mixture in the second containment and vertical displacement.

For purposes of neutron metrology nine activation monitor sets were prepared. Five monitor sets were placed in the central channel of the specimen holder at various vertical levels. The remaining monitor sets were placed in the northern (2), eastern (1) and southern (1) channels, useful for an indication of the radial gradients and the orientation of the experiment. The neutron fluence is measured after the experiment.

It is important to keep the level of radioactivity of the graphite samples as low as reasonably possible, to make the handling of the



Fig. 3. A metal drum with four stacks to load the graphite specimens.

samples more practical after the irradiation experiment (post-irradiation examinations and reloading in next irradiation experiment) of the samples. Besides extra purifications steps that were made by the manufacturers, some measures are taken in the design. Contact between the metal drum and graphite samples is avoided by placing a graphite foil between them. Contamination of the graphite samples by the drum material is therefore not possible. For the same reason the inner containment is purged with high purity Helium.

The design of the INNOGRAPH capsules is kept as simple as possible to allow loading and assembling in a lead shielded hot cell.

INNOGRAPH-1A consists of eight drums all having a height of 60 mm. There are four stacks for samples in each drum. The irradiation temperature in the lower six drums was 750 °C, the irradiation temperature in the upper two drums 650 °C.

The second irradiation capsule at 750 °C (INNOGRAPH-1B) will contain samples that are irradiated to a dose above cross-over, i.e. the diameter of the samples might be larger than the original 8 mm. To make space for this potential swelling, the diameter of the sample stacks in the drums is larger in this capsule. Therefore, there is only space for three columns of samples in INNOGRAPH-1B. This capsule consists of seven drums (six drums of 60 mm and one drum with a height of 30 mm) that will be targeted at 750 °C. An extra drum at the top, having a height of 60 mm is designed to have a temperature of 650 °C. The high vertical position of this upper drum makes it not possible to reach 750 °C, because of the low gamma heating at this position. The extra drum is separated from the other drums by a thermal barrier.

INNOGRAPH-2A, the experiment at 950 °C up to medium dose, also has three columns for the samples. To reach 950 °C more mass is needed in the experiment than the mass needed for an irradiation temperature of 750 °C. Therefore, the relative high temperature is the reason that in this experiment the number of stacks is limited to three. Furthermore INNOGRAPH-2A has the same drum configuration as INNOGRAPH-1B: six drums of 60 mm and one drum of 30 mm targeted at 950 °C. An extra drum of 60 mm is placed on top. In this drum the temperature is targeted at 850 °C. Again the two temperature zones are separated by a thermal barrier.

The final design of INNOGRAPH-2B, which is foreseen to start in 2008 is not finished at the moment this paper is written, but is likely to resemble that of INNOGRAPH-2A.

6. Results INNOGRAPH-1A (preliminary)

The first INNOGRAPH irradiation experiment, INNOGRAPH-1A was started in February 2004 and was finished in April 2005. In this section a first glance of the results are presented.

6.1. Irradiation performance

The fluence that an individual sample receives depends on its vertical position in the experiment. The neutron flux gradually decreases when the distance from the centre line of the core increases. The fluence is also different in each sample channel, because one channel is closer to the fuel position in the core. Since the material properties change with increasing neutron dose and corresponding irradiation damage, it is important to know the exact damage suffered by the individual graphite specimens. The neutron fluence of each sample can be determined from the data obtained from nine activation monitor sets placed throughout the capsule [8].

INNOGRAPH-1A has been irradiated for 14 HFR cycles. The effective irradiation time, total full power days, was 324 days.

The average results of the fluence measurements are summarised in Table 3. The uncertainty in the fluence numbers is <8%, the uncertainty in the damage is <15%.

The average irradiation damage in the experiment (8.2 dpa at 750 °C) is close to the targeted 8 dpa. The spread in the dose levels is shown in Table 4. A dpa range from 5.7 to 9.9 is covered by this irradiation experiment. This range is very useful to obtain the property changes as a function of irradiation damage.

The irradiation temperature of each sample can be obtained from the 24 thermocouples in INNOGRAPH-1A. During the irradiation experiment the data of the thermocouples are stored every minute. The thermocouple readings are used in the thermo-mechanical models to calculate the individual sample temperatures [9,10]. The detailed analysis of the individual sample temperature is in progress and will be finished in the first quarter of 2007.

The average temperatures per drum over the total irradiation time are shown in Table 5. The target irradiation temperature of 750 °C is reached in drums 1–6. The irradiation temperature in the upper two drums is about 650 °C.

Table 3

Average sample fluences and irradiation damages in INNOGRAPH-1A

	Fluence Φ , $E > 0.1$ MeV (10^{25} m $^{-2}$)	Fluence Φ , $E > 1.0$ MeV (10^{25} m $^{-2}$)	dpa_graphite
Sample average in 750 °C zone	11.5	5.2	8.2
Sample average in 650 °C zone	7.7	3.5	5.5

Table 4

Irradiation damage levels in INNOGRAPH-1A

	Drums 1–6: 750 °C zone	Drums 7–8: 650 °C zone
Average sample damage	8.3	5.5
Maximal sample damage	9.9	7.7
Minimal sample damage	5.7	2.9
Minimal damage/maximal damage	0.58	0.38

Table 5

Preliminary irradiation temperatures in INNOGRAPH-1A

Drum	$T_{irr, average}$ (°C)	$T_{irr, standard deviation}$ (°C)
Drum-1	640	40
Drum-2	660	40
Drum-3	720	30
Drum-4	750	20
Drum-5	750	20
Drum-6	750	20
Drum-7	730	10
Drum-8	740	20

The variation in temperature over the irradiation time is shown by the standard deviation. This variation in the upper two drums is higher than the variation in the other drums. This is because the temperature is controlled by keeping the irradiation temperature in the 750 °C zone as close to 750 °C as possible.

The design of the next INNOGRAPH experiment is adjusted with the aim to lower this standard deviation in the extra drum, although the temperature control will always be focussed on the other seven drums only.

The post-irradiation experiments that are presented in this paper are from samples of drums 1–6. The nominal irradiation temperature of these samples is taken in this paper to be 750 °C, i.e. the average temperature in these drums. Sample to sample variations of the irradiation temperatures will be taken into account once the individual irradiation temperature is obtained.

6.2. Post-irradiation examinations

There are eight ‘modern’ grades irradiated in INNOGRAPH-1A (see Table 1). The results in this section are from the samples from drums 1 to 6, i.e. the drums in which the irradiation temperature is controlled to be 750 °C. The grades in this section are kept anonymous because the results are not public yet. The results from Gilso-carbon graphite (included as an extra grade) are presented elsewhere in this Journal [11].

The relative length change as a function of dose of the extruded graphite grades are shown in Fig. 4. The samples are all in shrinkage as expected at this dose. The maximum shrinkage is between –2.0% and –2.7%. In the with-grain direction the shrinkage is larger than in the across-grain direction. The anisotropy in the length change is comparable for all four grades. There is no distinct difference between the results from the samples from the edge and samples from the centre of the graphite blocks.

The relative length change as a function of dose of the iso-moulded graphite grades are shown in Fig. 5. The maximum shrinkage is between –1.1% and –2.4%. The shrinkage is larger in the with-

grain direction than in the across-grain direction and again there is no distinct difference between the results from the samples from the edge and samples from the centre of the graphite blocks. It is eye-catching that the anisotropy in shrinkage behaviour of the iso-moulded grades is much larger than the anisotropy behaviour of the extruded grades.

With just the results from INNOGRAPH-1A it is not possible to draw many conclusions. The results of INNOGRAPH-1B will show where the points of turn-around and cross-over are. The dose at these points as well as the anisotropy in the dimensional change behaviour will tell more about the irradiation behaviour of the different grades.

The change in the Dynamic Young’s modulus (E) of two extruded and two iso-moulded grades are shown in Figs. 6 and 7 by plotting the modulus after irradiation (E) divided by the initial Young’s modulus (E_0). At a low dose, where unfortunately no data points are available, E increases, which has been ascribed to pinning of dislocations in the basal plane. After this pinning a plateau value is reached, before a second rise in modulus occurs. This second rise is linked to internal stresses caused by dimensional changes. The data from INNOGRAPH-1A are in a dose range between 6 and 10 dpa, and are showing the second rise in the modulus curve.

The modulus as a function of dose is comparable for the extruded graphite grades. The behaviour is isotropic. The same holds for the iso-moulded grades; however, the relative change in modulus is slightly higher.

The coefficient of thermal expansion (CTE) is also changing during irradiation. The CTE as a function of irradiation damage is shown in Fig. 8. The CTE is plotted for two grades, one extruded and one iso-moulded, at two different temperature intervals (CTE_{30–120°C} and CTE_{30–750°C}). The CTE is gradually decreasing in the neutron dose range of INNOGRAPH-1A. This decrease varies between 20% and 60%. The anisotropy in the CTE for the two directions remains. Grades that have a higher initial CTE show the largest drop in CTE upon irradiation.

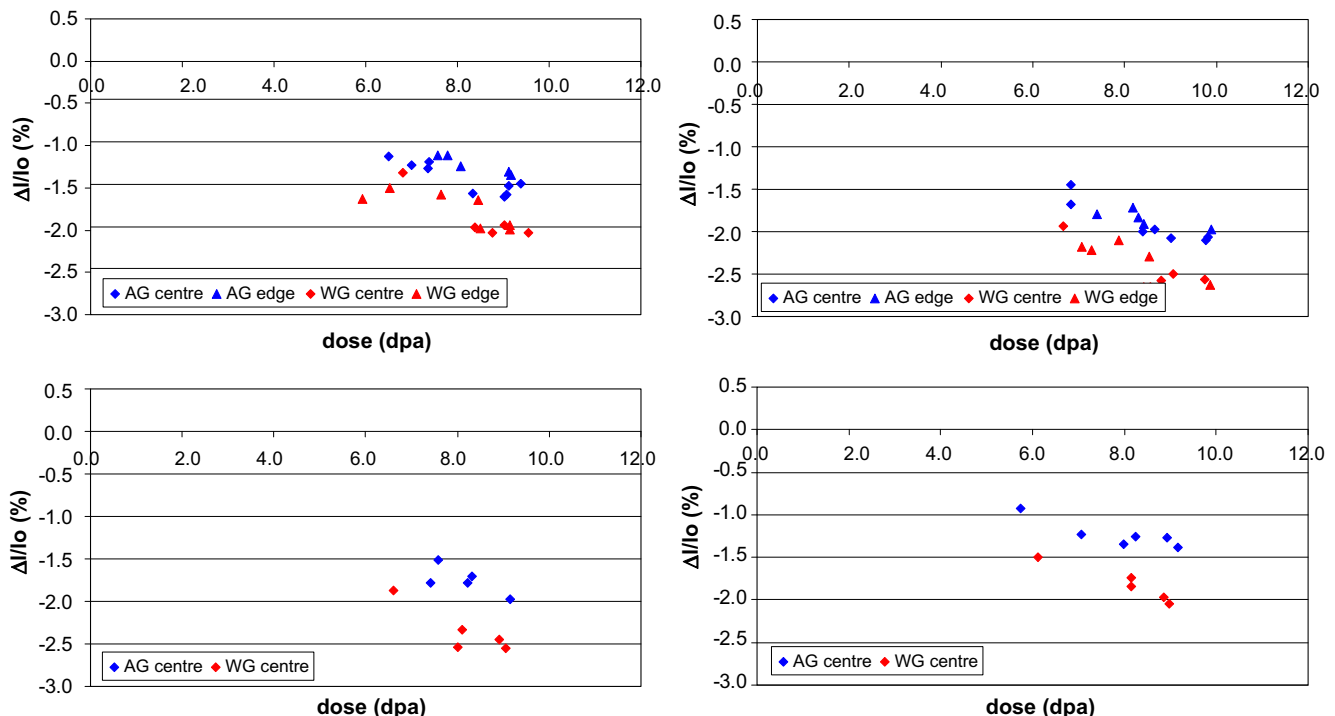


Fig. 4. Relative length change of extruded graphite grades from INNOGRAPH-1A. The irradiation temperature is 750 °C.

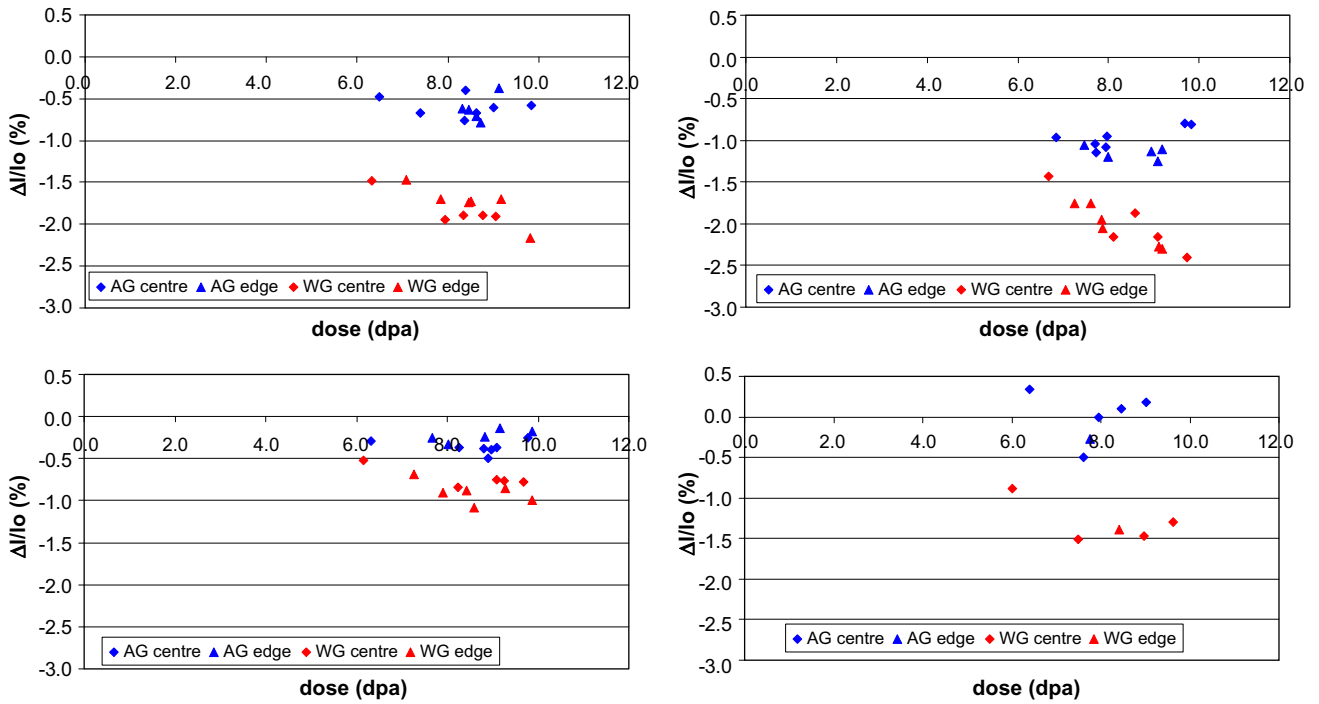


Fig. 5. Relative length change of iso-moulded graphite grades from INNOGRAPH-1A. The irradiation temperature is 750 °C.

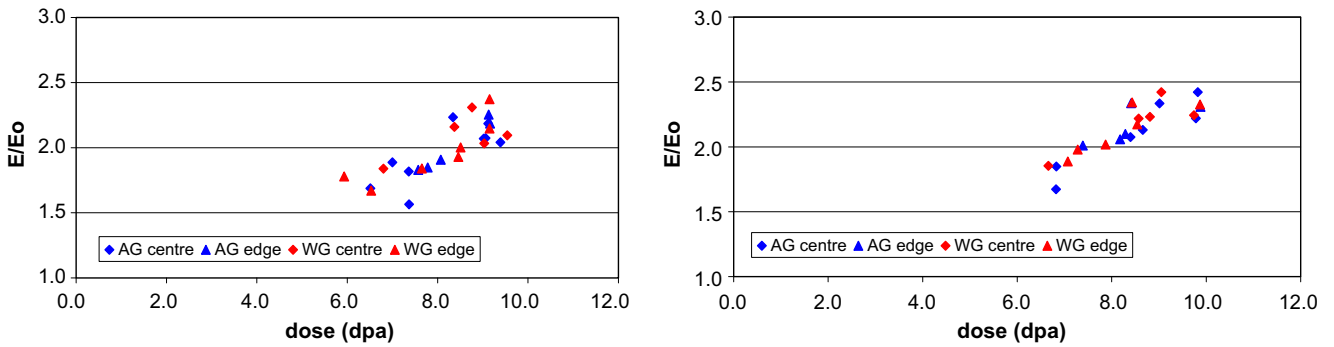


Fig. 6. Change in dynamic Young's of two extruded graphite grades from INNOGRAPH-1A. The irradiation temperature is 750 °C.

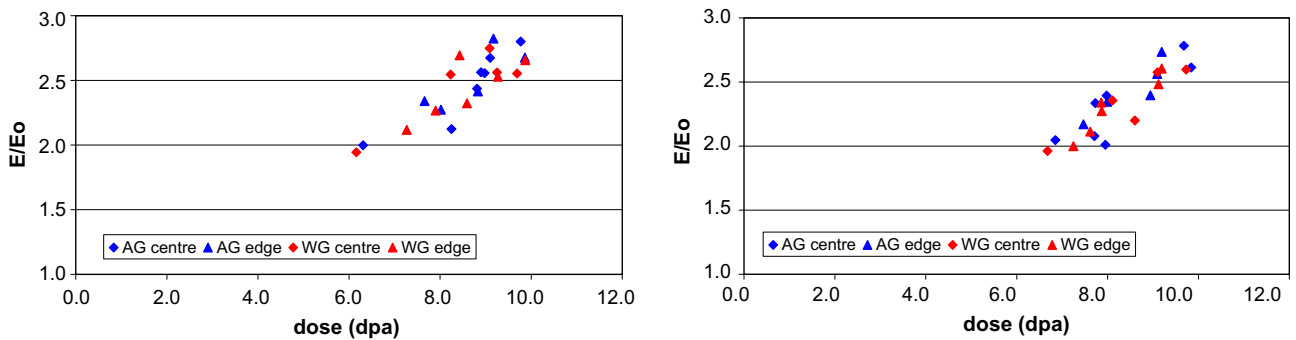


Fig. 7. Change in dynamic Young's of two iso-moulded graphite grades from INNOGRAPH-1A. The irradiation temperature is 750 °C.

The coefficient of thermal conductivity collapses due to irradiation damage. The thermal conductivity is determined by phonon scattering. During irradiation the crystal lattice of the graphite is damaged and therefore the phonon scattering increases, leading to a decrease in thermal conductivity. Two examples, one extruded

graphite and one iso-moulded graphite are shown in Fig. 9. It should be noted that the coefficient of thermal diffusivity is measured and that the thermal conductivity is obtained by multiplying the thermal diffusivity with density and specific heat. The specific heat of graphite is assumed to be the same before and after

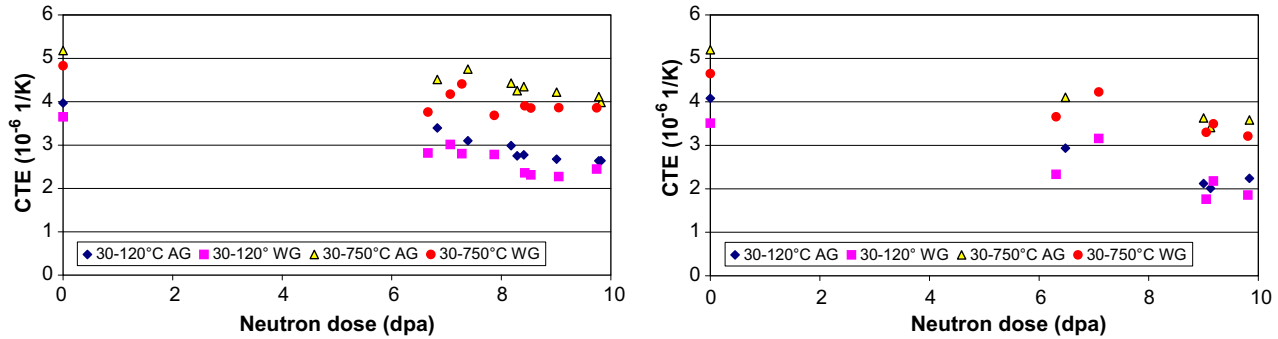


Fig. 8. Change in coefficient of thermal expansion of a typical extruded (left) and typical iso-moulded graphite (right) from INNOGRAPH-1A. The irradiation temperature is 750 °C.

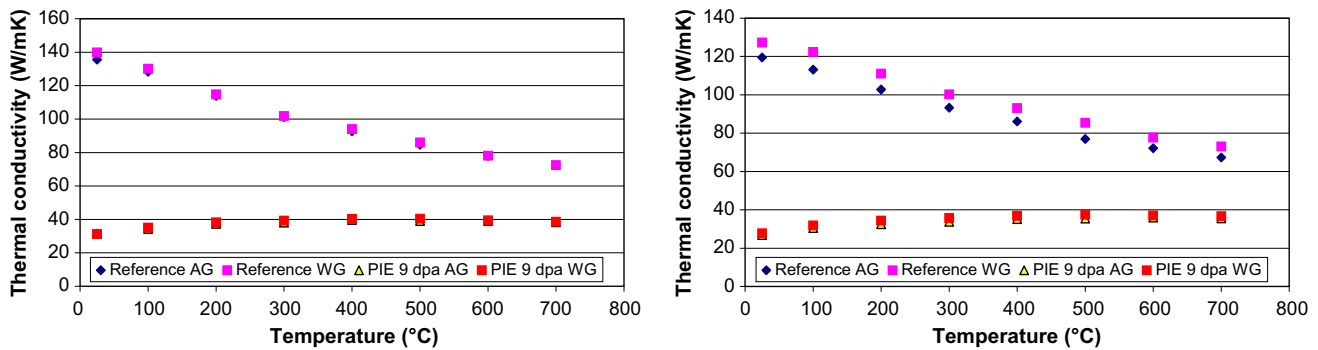


Fig. 9. The coefficient of thermal conductivity as a function of temperature of a typical extruded (left) and typical iso-moulded (right) graphite, before irradiation (reference) and after (at 9 dpa). It is assumed the specific heat of graphite is not changed during irradiation. The irradiation temperature is 750 °C.

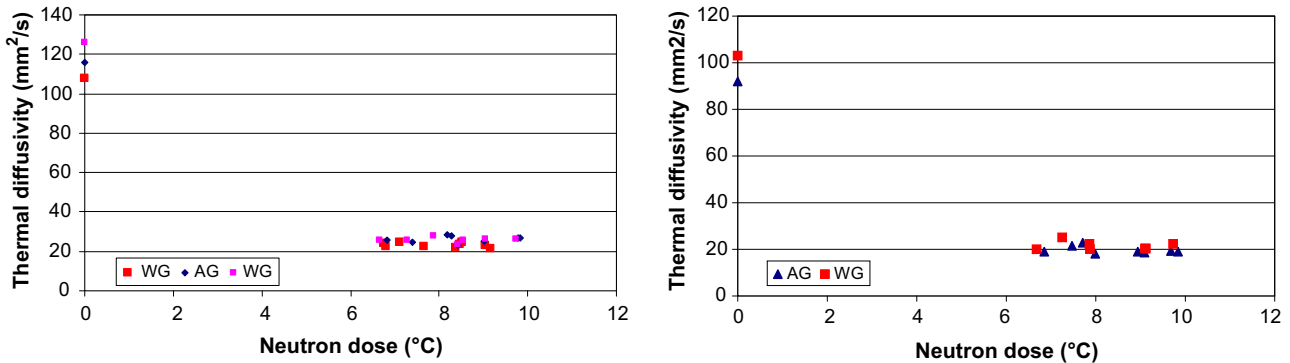


Fig. 10. The coefficient of thermal conductivity at room temperature as a function of neutron dose of two extruded grades (left) and a typical iso-moulded (right) graphite. The irradiation temperature is 750 °C.

irradiation, the values for the specific heat as a function of temperature are taken from ASTM C781-96 [12].

The coefficient of thermal conductivity as a function of neutron dose is shown in Fig. 10. After 6 dpa the coefficient of thermal conductivity is decreased to about 20% of its original value. Between 6 and 10 dpa no further change is observed.

7. Conclusions

The European R&D programme on nuclear graphite grades is focused on the irradiation of presently available grades, with emphasis on screening and obtaining reasonable design curves, allowing EU HTR design by ~2011. A wide range of graphite grades, having

different raw materials and produced by different production methods are irradiated at 750 °C and 950 °C in two successive stages up to high neutron dose. The irradiation behaviour is studied by measuring dimensional changes, change in dynamic Young's modulus, coefficient of thermal expansion, and coefficient of thermal conductivity at different neutron dose levels. In addition, microstructural investigation will be performed as well as strength measurements.

At the end of 2006 (the moment this paper has been written) one experiment (first stage at 750 °C) is finished, one experiment is in irradiation (first stage at 950 °C), one experiment is being assembled (second stage at 750 °C) to start in the beginning of 2008 and the fourth experiment (second stage at 950 °C) is planned in 2008.

The results of the first irradiation experiment (at 750 °C up to medium neutron dose) look promising. Conclusions on irradiation behaviour of the different graphite grades can only be drawn later in the programme, when the high dose behaviour is known.

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