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HTR-M1 Graphite – Final Summary report

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Summary

This report provides an overall summary of the work performed on graphite activities within HTR-M1 and represents Deliverable D12 of the required project outputs.

The work on graphite (contained within Work Package2 of HTR-M1) covers graphite oxidation work plus the first stage of an irradiation programme (INNOGRAPH 1A) on graphites selected from those proposed by manufacturers for the HTR core. The oxidation work addresses two areas namely: a) the oxidation behaviour of the graphites selected for the graphite irradiation programme and b) a review of past experiences on pebble coatings to address corrosion problems investigated as part of the previous German HTR programme. For the former one graphite (NBG 20) is not considered suitable for HTR application based on the available oxidation evidence to date.

The first stage of an irradiation programme has been carried out in the Petten HFR involving ~200 samples manufactured from eight different grades of graphite, as part of the HTR-M1 Project within the 5th Framework EU part-funded Programme. The samples were first of all pre-characterised, and then irradiated to a maximum of around 1/3 peak fluence at a temperature of 750^{o}C. Results from the initial measurements on ~50% of the irradiated samples have been evaluated, and for two of the grades (NBG-10 and PPEA) have been compared with the German ATR-2E graphite. The measurements suggest that these two graphites have a higher initial shrinkage rate than ATR-2E, but with turn-around expected to occur at a higher fluence.

None of the current grades can be eliminated at this stage, and it will only be when the results from the second stage of the irradiation (INNOGRAPH 1B) are available that meaningful conclusions can be made. The results however are promising and will make an important contribution to the evaluation of suitable graphites for the next generation VHTR.

The future activities to be performed within 6th Framework (RAPHAEL-IP) as a continuation of this work includes a second phase of irradiation testing at 750^{o}C and the generation of a new full curve behaviour at 950^{o}C. Further work is also planned on oxidation and micro-structural modelling.

1 Introduction

This report provides an overall summary of all the work performed on graphite activities within HTR-M1 and represents Deliverable D12 of the required project outputs.

Graphite has been used as a neutron moderator and reflector in a variety of different reactors, and there has recently been a renewal of interest in High Temperature Reactors (HTRs) in the world. Most of the available data have been obtained by irradiating small graphite samples in MTR's, however almost all the graphites previously irradiated are no longer commercially available. As a result a planned irradiation programme of some of the currently available graphites is being carried out within the HTR-M1 Project (as part of the EU 5TH Framework Programme) to allow the 'best' graphite(s) to be chosen, and to provide initial data necessary for a core design to be undertaken.

For the design of the graphite components sophisticated models (generally finite element based) are used to determine the distortions and stresses in individual components, the deformations of the graphite structure(s) as a whole, and the interactions with interfacing structures, under the range of expected operating conditions and specified fault conditions. Testing of individual components will also be required to determine their strength (failure stress) and failure mode under the expected loading conditions, and possibly the testing of arrays of components. As input to such calculations and test procedures, material properties (physical and mechanical) of the as-manufactured graphite, and the way in which these change over the lifetime of the reactor (including dimensional change behaviour of the graphite) with fast neutron irradiation are necessary for confirmation of core integrity and safety.

The work on graphite (contained within Work Package2 of HTR-M1) involves some graphite oxidation work plus the first stage of an irradiation programme (INNOGRAPH 1A) on graphites selected from those proposed by manufacturers for the HTR core. The oxidation work addresses two areas namely: a) the oxidation behaviour of the graphites selected for the graphite irradiation programme and b) a review of past experiences on pebble coatings to address corrosion problems investigated as part of the previous German HTR programme.

The HTR-M1 irradiation programme is carried out in the High Flux Reactor (HFR) at Petten with a target dose 8 dpa_g (i.e. up to 'turn round') at a temperature of 750^{o}C and focuses on the determination of key physical properties (including dimensional change) under neutron irradiation. .

The second stage of this irradiation (INNOGRAPH 1B) to full fluence is to be carried out in a follow-on programme within the 6th Framework Programme (RAPHAEL-IP) with around 1/2 of the samples replaced by new samples of the selected graphites. The samples left in will therefore be taken to full fluence, with the newly installed samples being taken to ~2/3 peak fluence.

A similar irradiation tests is also to be conducted at 950^{o}C (again within RAPHAEL-IP), initially to 1/3 peak fluence, (INNOGRAPH 2A) followed by a second stage (INNOGRAPH 2B) to full fluence (~25 dpa_g).

Some preliminary Post irradiation examination (PIE) have been carried out on the irradiated specimens as part of HTR-M1 project however the complete set of PIE measurements on the samples describing the full curve behaviour will not be available until ~2008 at the end of the full fluence irradiation. A similar set of PIE results for the 950^{o}C case will also be available at the end of the RAPAHEL-IP ~2010.

2 Activities of HTR-M1 Work Package 2 on graphite

The activities of Work Package 2 of HTR-M1 address both the selection and the testing of graphites for the HTR core. Although the final choice of graphite is based on a number of factors, the most important is the effects of fast neutron irradiation on its properties (especially dimensional change) plus resistance to oxidation. These issues are addressed within HTR-M1, the former up to a target dose of 8 dpa_graphite at 750^{o}C and the latter with respect to high temperature tests in steam and air . The specific graphite tasks involved in HTR-M1 are as follows:

- Graphite selection, procurement, specimen fabrication and testing programme
- Graphite oxidation plus coating evaluation
- Graphite irradiation and post irradiation tests
- Database and assessment of suitability

Note that a much larger dose (the peak dose for pebble bed reactor is around 25 dpa_graphite) will need to be investigated before the final selection can be made and this is catered for in the follow-on activity within RAPHAEL-IP (as mentioned above) which involves the second stage (2/3 fluence) and third stage (full fluence) definition of the property curves (at 750^{o}C) plus a similar full curve definition at a higher temperature (950^{o}C).

The following sections provide a summary of the findings for each of the task areas addressed.

3 Graphite specification and selection

Issues associated with graphite selection and specification are discussed in Ref 1-7. The graphite core is a key component that affects safety and operability of the reactor. It provides structural support, coolant channels, moderation, and shielding while operating in a high temperature helium environment. Its performance is critically dependent on the graphite properties, which are irradiation dependent (Figure 1 & 2). The most important considerations are component integrity and changes in core geometry, both of which are affected by the dimensional change (see Figure.

1). The graphite selection criteria are therefore heavily dependent upon its behaviour under HTR irradiation typical fluence conditions.

With respect to graphite selection and the qualification of the graphite and its properties the following issues need to be considered:

- Graphite specification
- As-manufactured graphite properties / impurities
- Irradiation induced changes
- Oxidation

These impact on core integrity/safety, operational and decommissioning requirements:

The initial impurity levels in the graphite(s) together with the operating history of the reactor are important both for establishing the maximum activity of the graphite at any time (required if active components are to be removed and replaced) and the total active inventory at final shut-down (which is required for decommissioning).

The graphite specification needs to be practical, realistic and compatible with the design requirements. The Specification should cover all grades to be used. Different grades of graphite can be used for different regions of the core, (e.g. cost reduction purposes) with the highest grade graphite for example used for the high dose regions (moderator and inner reflector), and lower grade graphites used for the low dose regions (outer reflector and neutron shields).

The graphite Specification should state the material property requirements. These will provide the core designer with the initial information on which to base the preliminary core design and core lifetime assessment. Note that only the non-irradiated property variations should be included in the manufacturing specification.

The most important physical and mechanical properties required for current HTR core designs are density, strength (tensile and four point bend), thermal conductivity and coefficient of thermal expansion (CTE). Those of lesser importance but still required are Young's modulus, compressive strength, specific heat capacity and elastic strain to failure. Other important properties include neutron capture cross section and chemical reactivity with respect to air, and to a lesser extent open pore volume, permeability and diffusivity (mainly because the helium coolant is non-oxidising, although there will be some oxidation over life due to impurites).

The graphite Specification should contain limits with respect to impurity levels. The most important of these, in no particular order, are sulphur, chlorine, cobalt and lithium. (Boron is also important and this is related to the neutron capture cross section.) The Specification should also contain details of the material testing and certification requirements, plus the acceptance standards

for the material (i.e. allowable sizes/location of cracks, inclusions, porosity etc.).

To support the selection of graphites for use in a future HTR it was first necessary to establish the graphites that are currently available. This was done through a series of specialist meetings and meetings with specific manufacturers (Ref 1 to 4). Discussions were held with graphite manufacturers - GrafTech (previously UCAR Carbon), SGL and Toyo Tanso, to identify suitable candiate graphites from those currently available. A number of candidates were put forward for consideration in the irradiation programme as listed in Table 1. Toyo Tanso, agreed to provide IG 110 samples (to be used as a reference) and IG 430 samples for comparison.

Of the ten potential graphite grades manufactured by GrafTech, the grades put forward for selection were PPEA, PAEA, PCEA and a super-fine grade of PCIB (PCIB-SFG). The quoted density and Young's modulus for all four graphites are similar. Of the three graphite grades put forward by SGL for selection, only two were being manufactured on a large scale. These are NBG-10 and NBG-25, and were selected. Although the third grade, NBG-20 is being developed on a lab scale as an economic alternative, it was also selected. The two graphite grades put forward by Toyo Tanso were selected (IG 110 and IG 430), The former was included as a reference graphite since data at high temperature already exist..

For the tests, five (major) grades of graphite were selected (PCIB-SFG, PCEA, NBG-10, NBG-25 and IG 110) for full testing in the experiment, two made by GrafTech, two made by SGL and one made by Toyo Tanso. These will be the main focus of the experimental work to be carried out under irradiation and oxidation conditions. It was also decided that it would be useful to test other proposed 'graphites in the irradiation experiment, included as 'minor'grades, to give an early indication of their suitability for inclusion in further follow-on experiments (for example in INNOGRAPH 1B in RAPHAEL-IP). These could be included as 'piggy-back' samples in the irradiation capsule (see Figure 9). The (minor) graphites selected were UCAR grade PPEA, SGL grade NBG-20 and Toyo Tanso grade IG-430. Table 3 gives the full range of graphites investigated in the HTR-M1 irradiation experiment.

4 Graphite procurement and machining

Nuclear grade graphite is manufactured in large blocks. The 'major' graphites to be used in the experiment were supplied by UCAR, SGL and Toyo Tanso in the block sizes as listed in Table 2. Each block should be uniquely identified with a number after its initial forming. This allows each block to be traced though the subsequent baking and impregnation stages and the graphitisation stage. The final graphitisation process is carried out in large furnaces containing many large blocks. Each graphitisation run is generally referred to as a 'Heat', which would normally be uniquely identified with a number indicating the furnace used and the run number.

The manufacturer, through knowledge and experience, should be asked to produce graphite(s) with minimum guaranteed values for particular physical and mechanical properties (e.g.. density, Young's modulus, strength and coefficient of thermal expansion). These correspond to the macroscopic properties and their specification for comparison with pre-characterisation tests to be carried out in the experimental laboratories prior to irradiation testing. Manufacturers values for the selected graphites have been reported in Ref. 8.

The manufacturer, through knowledge and experience of the raw materials (e.g. filler, binder and impregnant), and the purification process during the graphitisation stage, should also be able to meet the requirements for impurity levels. This is an important additional limitation that can have a strong impact on decommissioning. Manufacturers values for the selected graphites have been reported in Ref. 8. The determination of acceptance levels for each impurity will depend on the flux level the graphite is exposed to, and whether or not man-access to certain areas are required. In the case of the UK Advanced Gas Cooled Reactors (AGRs), (for example), it was a case of whether some of the graphite is to be removed during the operating life for property measurements. It is the manufacturers responsibility to ensure that the impurity limits given in the specification are not exceeded. With regard to testing for compliance, the sulphur, chlorine, boron, cobalt and lithium content should be measured for every Heat. For other elements, the levels can be measured less frequently (e.g. every tenth Heat).

With regard to fabrication of the test pieces, the discs/blocks supplied were sectioned and machined to produce the 150 plus samples to be included in the capsule. The samples were cylindrically shaped (diameter of 8 mm, length of 6 or 12 mm) and uniquely identified with suitable markings on a 3mm flat (see Figure 5). The samples were machined both from the central region and edge of each block to allow the initial property variation to be determined as a function of block/disc position.

The specimens were machined from the blocks in five steps:

(1) Sawing of (11 mm) square bars from centre & edge
(2) Turning of the bars into cylinders
(3) Milling of 3mm plane over length of cylinder
(4) Cutting of bars into 6 & 12mm lengths
(5) Engraving code on milled face

Note the plane is milled perpendicular to the z direction if the longitudinal direction of the bar is parallel to the xy direction. The plane is milled in an arbitrary position if the longitudinal direction of the bar is parallel to the z direction

For the 'major' grades selected, ten samples of 6 mm in length and five samples of 12 mm in length, i.e. fifteen in total, were produced for each direction (with grain and against grain) giving a nominal thirty samples for each 'major' grade. The number of samples of the 'minor' grades was dependent on the space in the irradiation rig.

5 Graphite test programme and pre-characterisation

The irradiation testing in INNOGRAPH 1A is carried out at a nominal irradiation test temperature of 750^{o}C. For each major grade there were originally 30 specimens (15 per main direction – z and xy per grade) planned, however due to improvements in the design of the irradiation test rig it was possible to include some extras. For the minor grades a maximum of 10 specimens per grade was envisaged. In addition, some special piggy-backs were included in to gain extra knowledge in the irradiation behaviour of graphite. The final test matrix is shown in Table 3.

Pre-characterisation tests are required for each sample in order to determine specific property values for each specimen before inserting into the irradiation capsule. This is necessary in order to make sure an accurate description of the influence of the irradiation is determined for each sample. Such tests are required in addition to the manufacturers nominal property values taken at the centre and edge regions of the manufactured blocks (see previous section) which provide a check on the measured pre-characterisation measurements performed by the experimental laboratories. Impurity levels are also provided by the manufacturers for each of the graphite grades to be irradiated which can be used to assess post irradiation handling and examination requirements.

Unirradiated properties were determined for each graphite from the machined samples. Figure 7 shows some property range values for the graphites. The dimensions were measured together with Young's modulus, CTE and thermal diffusivity (from which the thermal conductivity is determined). The mass of each sample was measured, which together with the dimensions, will allow the initial density to be determined. Clearly it was not possible to measure strength on the samples to be irradiated as this will involve a destructive test. However it is possible to make strength measurements on other suitable samples from a block for comparison and which should be representative of those which will be irradiated. Details of the testing methods adopted are given in Ref 8 & 9 and identical to those used for the PIE. The measurements were recorded in tabular form in an electronic spreadsheet and used as part of the graphite database (see Ref 10).

Measurement of the coefficient of thermal expansion coefficient and thermal diffusivity take one full day due to the heating and cooling of the specimen and performing these tests on all 150 to 180 specimens before the irradiation was expected to take more than 30 weeks. To save time not all samples were measured. A measuring scheme was adopted to allow the properties of non-measured specimens to be estimated from neighbouring specimens as shown in Figure 6.

All the pre-characterisation measurements, and the PIE measurements made to date (together with specific properties derived from them) are contained within Excel spreadsheets developed by NRG. These have now been transferred to individual worksheets for each graphite grade (Ref 10). These

worksheets are used to calculate the fractional changes in the properties due to irradiation and to display them in graphical form. Eventually, it is intended that they be used to produce the appropriate 'design curves'.

Details of the measuring methods and devices used are given in Ref 9. The following sections briefly provide the main features;

Dimensional changes (Fig. 15)

The dimensions of all specimens are measured before and after the irradiation. The measurements are performed with 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 known calliper and the unknown specimen. The length of the specimens is measured at six positions, the diameter at four and the diameter across the flattening at three. The time span between the measurements before and after irradiation is about two years. To prevent differences in the measurement procedure before and after irradiation the complete procedure is formalised into a working instruction

Density (Fig. 15)

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 mass is determined three times. The reported density is the measured mass divided by the determined volume.

Dynamic Young's Modulus (Fig. 15)

The Dynamic Young's Modulus (DYM) is determined by measuring the velocity of sound in the sample using ASTM standard C769. 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 calibration is done before each measurement series and repeated after 20 measurements. Important points are dry contacts (using rubber heads) and constant contact force. The velocity of sound is measured by changing the selected velocity of sound to match the travel distance through the specimen thickness. The velocity of sound of each sample is determined three times. As a check on accuracy. the velocity of sound is also determined for a representative specimen thickness, which differs from the specimen thickness by 0.02 mm. Cross check measurements were also made against procedures used by BNFL.

Coefficient of Thermal expansion (Fig. 17)

This is determined using a commercial dilatometer which covers a temperature range up to 1500^{o}C, and operates in an inert environment and can be used in a glove-box and hot cell. Measurements are taken in two directions at temperature (e.g. 20^{o}C to 750^{o}C) and each day one sample is measured three times..

Thermal diffusivity (Fig. 16)

This uses the laser flash method and measurements were made for temperatures up to 750°C. The thermal conductivity (λ) is determined using the formula:

$$\lambda = \rho \, . \, C_p.a$$

where a = thermal diffusivity
ρ = density
C_p = specific heat

The samples were first of all pre-characterised, and then after the irradiation the property changes were investigated using specially installed shielded facilities and equipment. Further assessments are to be carried out whenever future PIE measurements are completed. In the near future there will be the outstanding measurements on the samples from INNOGRAPH1A, and beyond that, the PIE measurements from the second stage irradiation at 750°C (INNOGRAPH1B), and the two stages of the irradiation at 950°C (INNOGRAPH 2A and 2B).

6 Graphite oxidation evaluation

In addition to the oxidation tests performed in the HTR-M Programme (Ref. 12), FZJ carried out further investigations on oxidation characteristics of the new graphites under investigation in the HTR-M1 graphite irradiation tests. The investigations cover oxidation measurements (oxygen/air) of eight chosen structural graphites (see Table 4) at temperatures of 650 and 700°C performed in the THERA facility (Figure 3). The samples used in the THERA facility are small pieces of maximum weight of 250 mg. Measurements were carried out up to a final weight loss of 70-100%, which means in the case of 100 %, that the sample vanished because of complete oxidation. The run times ranged between ~ 12 min up to 20 h. This work was not originally scheduled for the project and has been provided by FZJ within their original resource allocation.

Most of the materials were shown to have a comparable reactivity. Most of the new graphites were similar or better than V438, an established nuclear graphite from the former German HTR programme. Within the investigated graphites there are two materials (PPEA and NBG-10), which always show the lowest rates. The others have higher rates, which are however still within an acceptable range. The exception to this was NBG 20 (see Figure 4). Note that because of the apparent difference in rates (at least a factor of 3 higher) a number of samples of NBG 20 were tested but without any obvious differences in behaviour (Ref. 12).

The graphite NBG 20 was considered unsuitable for application to the HTR. A number of possible causes were considered (graphitisation, purification, ash content) but to date no obvious reasons for the differences have been found.

It is planned to continue the explorations of NBG 20 within the 6th Framework Project RAPAHEL Project as part of the graphite investigations.

7 Graphite Coating evaluation

Work done in Germany on the development of SiC coatings for graphite materials (mainly pebbles) for protecting against oxidation from air or steam ingress under severe accident has been reviewed and reported (Ref 13). Investigations were done using the German matrix graphite A3-3 to measure corrosion rate for temperatures up to 1200^{o}C, and on coated pebbles using different graphites. Most of the coating experiments used the Japanese graphite IG 110 adopted for the structures and fuel blocks of the HTTR. Corrosion tests, and irradiation tests plus by PIE were carried out to assess coating integrity and different coating methods (Chemical vapour deposition (CVD), Paste Siliconisation (PS)). A very good corrosion resistant quality was eventually achieved with IG 110 and V438 using CVD. No damage or weight loss was observed after being irradiated and corroded and dropped from a height of 50 cm. Although the original objective, to develop a coating for A3-3 graphite, had not yet been achieved, the results obtained so far are a very positive indicator and have attracted considerable interest Internationally (from US NRC).

8 Graphite irradiation and post irradiation tests

A major part of the HTR-M1 programme is this re-establishment of graphite irradiation experimental facilities within Europe plus the completion of a first phase of irradiation tests (with PIE) on selected graphites for the VHTR. This work is being done by NRG with the use of the High Flux Reactor (HFR) at Petten. Several grades of graphite are being irradiated at a temperature of 750^{o}C up to fluence equivalent to around 8dpa (11 to 15 HFR cycles) with the second phase of the work planned to continue in 2006 as part of the VHTR-IP up to the completion of full fluence level of around 25 dpa.

For the graphite irradiation programme five major graphites were selected (supplied by three manufacturers), two iso-molded and two extruded graphites, manufactured from pitch coke and petroleum coke plus IG 110 (HTTR graphite), iso-moulded made using a petroleum coke, to provide a reference. The samples provide a range of grain sizes (1mm down to 10 µm). Additional 'piggy-back' samples are included in the irradiation capsule to extend the knowledge of other grades. The irradiation test provides a number of data point within a single test run by using the flux (buckling) distribution available in the HFR. The work involves the following stages: development of the loading rig (INNOGRAPH), pre-characterisation of the test pieces, assembly of the rig, installation of the rig in HFR, removal of the rig after irradiation and post irradiation examination (PIE) in shielded facilities. The findings from these activities are summarized in the following sections.

8.1 Details of INNOGRAPH 1A Test facility & loading of the pre-characterised specimens

Most of the previous high temperature European data on HTR graphites has been obtained using the CERAM series of irradiation devices developed at FZJ. These were multi-specimen rigs each operating at a single temperature (e.g. CERAM 56-II contained 144 cylindrical specimens 6 mm in diameter x 25 mm in length) and with re-loadable devices.

The INNOGRAPH irradiation rig has been designed and developed at NRG to be capable of allowing 2 distinct temperature levels and by using a slightly smaller specimen than CERAM, to enable upwards of 150 specimens to be loaded at any one time. The test rig based on the D85 series (German HTR-Modul progamme up to 1994), and an actual SiCSiC irradiation (2002), consists of 8 drums made of TZM steel. Each drum has 4 columns with a graphite stacking height of 60 mm. Specimen temperatures are individually monitored and maintained using eighteen thermocouples located near the centre and circumference of the drum. Small axial and radial temperature gradients are present (checked by calculation). The design is not sensitive to graphite property changes and utilises a helium environment with moisture control. Details of the test rig and drum details are shown in Figure 9.

The graphite specimens are stacked in columns with the specimens separated from the TZM drums by a graphite foil. An example of a drum and a typical loading is shown in Figure 9. The target temperature for the irradiation is 750°C and the target dose is 8 dpa_g (dpa in graphite). The irradiation test provides a number of data point within a single test run by using the flux (buckling) distribution available in the HFR capsule (Figure 8). A third order polynomial describes the behaviour and a sufficient number of points should be obtained from the two series of test measurements to describe the distribution. The device is loaded into position C7/E7 in the HFR core.

8.2 Irradiation and cooling period

Soon after loading commenced it was found that as a result of small gaps and excentricities in the assembled geometries that there were variations in the temperatures of the specimens (see Figure 8). Since each of the specimen temperatures was individually monitored and maintained and in order not lose any tests, it was decided to adopt the resulting axial variation temperatures for the experiment. This meant that 75% of specimens (upper part) are being maintained at 750^{o}C and 25% of specimens (lower part) at 670^{o}C. It is considered that there are sufficient specimens at each temperature not to impair the statistical balance of the test results.

The aim of the irradiation was to get a large spread in dose with a target of 8 dpa_g. Figure 8 shows the actual variation of the interpolated dose with the position of the specimen in the irradiation rig. The median dose achieved is 7.92 dpa_g or 6.32 x 10^{25} m^{-2} EDN, with a maximum of 9.88 dpa_g (or 7.88 x 10^{25} m^{-2} EDN) and a minimum of 2.92 dpa_g (or 2.33 x 10^{25} m^{-2} EDN). This implies that within the irradiation position used, the radial flux gradient has added a surplus of about 0.8 dpa_g on the expected peak value.

The temperature readings during the full irradiation period have not been fully evaluated as yet but will be completed during the preparations for the follow-on test. The low dose specimens (< 4 dpa_g) have been irradiated at a lower temperature, in a range between about 650 and 690 °C.

The irradiation test was achieved using 15 Loading cycles, and was followed by a period of cooling (approximately 1-2 months) before the specimens could be transferred to the shielded facilities for dismantling and examination.

8.3 Post Irradiation Examinations

The specimens were transported into concrete cells and the drums retrieved. The drums were transported to lead cells (Figure 11, 12) and the cover removed and the specimens pushed out and placed in trays. Three Glove boxes (Figure 13) are provided next to the shielded cells (see also Figure 12, 14) for the examination stages. The first Box (Box 1) is connected directly to the hot cell to make transportation to the glove boxes easy. Box 1 contains facilities for determining dimension changes, mass and Dynamic Modulus measurement (Figure 15). Box 2, which connects to Box 1 contains the equipment for measuring Thermal Diffusivity (Figure. 16). Box 3 is linked to Box 2 and contains the thermal expansion measuring equipment (Figure 17).

For the post irradiation measurements it was found that the dose rates on the graphite samples were low enough for handling within glove boxes. There was however a large variation between grades. The extra purification step performed on some NBG-10 specimens for example decreased the specimen dose rates by factor 10. The activity will be measured by gamma spectroscopy.

Dose rate after 4 months of cooling:

- At contact: 2-250 mSv/h
- At 0.1 m: 1-23 mSv/h

8.4 Results

The Irradiation experiment (with 200 graphite specimens) was been successfully performed in the High Flux Reactor at Petten with a target dose of 8 dpa_g (or 6.3×10^{25} m^{-2} EDN) at 750°C. This experiment was accompanied by the installation of state-of-the-art pre- and post-irradiation test facilities for measurement of key physical properties. A full traceability of materials and data has been implemented.

The achievement of this first phase of tests within HTR-M1 is an important milestones in the substantiation of graphite selection for a European HTR. Also the task objectives to re-establish graphite irradiation and qualification methods within Europe (while the available experience and facilities exist), has been achieved. The irradiation programme has been performed on selected graphites to determine the variations in their physical and mechanical properties up to low/medium irradiation dose. The focus has been on the determination of engineering design data:

– Dimensional change
– Dynamic Young's Modulus
– Coefficient of Thermal Expansion
– Thermal conductivity/diffusivity

As the total measurement time for the full test matrix is quite long, priority was given to the 100 highest dose specimens (Table 5) with a fuller examination taking place within the Raphael Project activities. A selection of the HTR-M1 specimens will be re-irradiated in INNOGRAPH-01B rig within the RAPHAEL project to determine the full curve.

The results after irradiation are given as the percentage of change or fractional change compared to the pre-irradiation characteristics. The results are presented in Table 6. It is important to note that the average dose of each group is different and therefore it is difficult to compare the average results with each other. In Table 7 the results are summarised per grade. Around 50% of the irradiated samples have been subjected to some PIE, having their dimensions, mass and DYM re-measured. Results are presented in graphical form for extruded and iso-moulded graphites covering dimensional change, Youngs' Modulus and volume change (Figures 18-42) for all the measured graphites.

The University of Manchester has performed pre-irradiation x-ray tomography on about 60 samples on a voluntary basis for preparation work on modelling to be performed within RAPHAEL-IP.

9 Database and assessment of suitability

All the pre-characterisation measurements and the PIE measurements made (together with specific properties derived from them) were stored within Excel spreadsheets developed at NRG. However, before the experiment got underway, an Excel worksheet was proposed (comprised of individual spreadsheets for each graphite grade) that would be used to summarise the data in a convenient way and would enable the appropriate 'design curves' to be produce. At the conclusion of the HTR-M1 programme there was a requirement to review the available measurements and to enter them into this worksheet. There was also a requirement to assess the measurements and compare them with those made on other graphites in the past at similar temperatures. These requirements are covered below.

9.1 Database (Ref. 10)

The initial task was to transfer the measurements from the NRG spreadsheet into the proposed worksheet. This would then allow the property variations to be displayed in graphical form and the appropriate 'design curves' fitted. However, it soon became apparent that a single worksheet for all eight graphites would be too large and difficult to use and so it was decided that individual worksheets should be produced for each grade. There was also a need to amend each basic spreadsheet slightly in order to facilitate data entry and the generation of graphs/curves from them.

Worksheets for all eight graphites have been produced and the spreadsheet within each has been populated with the pre-characterisation measurement data (Ref 10). This spreadsheet is split into three 'pages'. The first 'page' contains the pre-characterisation data, the second 'page' contains the PIE measurements, and the third 'page' contains the calculated percentage and fractional changes in the properties.

9.1.1 Pre-characterisation measurements

Table 8 is an example of the first page of a spreadsheet, and shows the pre-characterisation measurements for IG-110. For IG-110 a total of 30 samples were pre-characterised, 16 of which were from the centre of the block and the other 14 from the edge of the block. Of the 16 samples from the centre of the block, 8 were machined with the cylindrical axis in the AG direction and the remaining 8 were machined with the cylindrical axis in the WG direction. Of the 14 samples from the edge of the block, 7 were machined with the cylindrical axis in the AG direction and the remaining 7 were machined with the cylindrical axis in the WG direction.

Measurements were made of the initial length (Lo) and diameter (Do) of each specimen, together with the shorter diameter perpendicular to the machined flat (xo). From these the initial volume (Vo) of the specimen was calculated. The initial mass (Mo) was measured and from this and Vo the initial density (?o) was calculated. The DYM was measured along the axis only ('L' direction). The CTE of the specimen was also only measured along its axis, but over three temperature ranges (30 – 120^{o}C), (30 – 200^{o}C), and (30 – 750^{o}C). The value quoted for each is the average over the range. The thermal diffusivity (a) was measured at 27^{o}C and at 100^{o}C intervals between 100^{o}C and 700^{o}C. From each of these measurements the thermal conductivity (λ) was calculated from the thermal diffusivity using the formula $\lambda = \rho \, . \, C_p.a$, where ρ is the density and Cp is the specific heat of the specimen.

As discussed above, due to the length of time required to carry out individual CTE and thermal diffusivity measurements it was not practical to carry out these measurements on all the samples. Therefore samples that were close neighbours to those that were measured were allocated the same values.

9.1.2 Irradiated sample measurements

All the measurements carried out for the pre-characterisation are also required on the irradiated samples. At the time of writing, a total of 96 samples have been subject to measurement (~50%), but to date, it has only been possible to measure their linear dimensions, mass and DYM, and to calculate (from the first two) their volumes and density.

The second 'page' of the spreadsheet within each of the worksheets has been populated with the PIE measurement data. Table 9 is an example of such a 'page', and shows the PIE measurements for IG-110. As well as the measurements, it is also important to know the fluence seen by each sample. This is expressed in the Table in two ways, firstly in terms of displacements

per atom (dpa) and secondly in terms of Equivalent DIDO Nickel Dose (EDND). It can be seen that the fluence reached ranges from 6.85 to 9.84 dpa, or 53.9 to 77.4 in terms of EDND (10^{20} n.cm^{-2}). The mid range is 8.35 dpa (65.7×10^{20} n.cm^{-2} EDND), which is ~1/3 of the target full fluence of 25 dpa (200×10^{20} n.cm^{-2} EDND),

9.1.3 Changes due to Irradiation

The properties of interest to the core designer all vary with irradiation fluence and that this variation also depends on the irradiation temperature. The linear dimensional and volumetric changes that occur are traditionally given in terms of fractional changes, expressed as a percentage. The variation is normally expressed as a ratio so that the it can be used as a multiplying factor (f) for the initial modulus (E_o) to give the irradiated modulus (E_i) at the required fluence/temperature ($E_i = f \, . \, E_o$). In order to calculate the changes, the pre-characterisation measurements and the post-irradiation measurements are entered into the appropriate locations the first and second 'pages' of the spreadsheet respectively. The variations are calculated in the required manner in the third 'page' of the spreadsheet. Table 10 is an example of such a 'page', and shows the calculated changes for IG-110. This is the 'page' used to generate the graphical plots and is therefore made 'stand-alone' by also providing information on the location and orientation of the samples as well as the fluence. The production of the graphical plots and the assessment of the behaviour of the different graphites is discussed below.

9.2 Initial assessment of the graphites (Ref. 11)

The measurements made for the effects of irradiation on the eight graphites have been assessed against values obtained from other graphites in the past at similar temperatures. A preliminary comparison has been carried out against the graphite used in the German HTRs, namely ATR-2E (see Fig. 43-46). The following sections summarise the preliminary findings for the main property changes measured.

9.2.1 Dimensional Change

The dimensional change behaviour of a graphite is arguably the most important selection criterion from a designer's point of view. Differential shrinkage strains within components give rise to stresses that could ultimately lead to cracks developing. The 'ideal' graphite would have a very small shrinkage rate, a moderately high fluence at which turn-around occurs, and a high fluence at which it goes into positive growth (i.e. original dimensions/volume attained).

Although only around half the samples have been measured, a preliminary assessment of the dimensional change behaviour of the graphites tested can be made. It has been found that there is a significant difference in the shrinkage behaviour, with one graphite showing a particularly high shrinkage rate and another with a particularly low shrinkage rate. This large variation was to be expected, given the range of graphites tested. It must be remembered however that the fluence attained so far is only ~ 1/3 of the

target peak fluence, and none of the graphites appear to have gone through turn-around although some are clearly approaching it. Therefore a low initial shrinkage rate, although appearing to be a good sign, will be of little use if there is an early turn-around and change to positive growth. This will not be known until the measurements have been carried out at the end of the next irradiation stage within the RAPHAEL –IP (INNOGRAPH 1B). At the end of this irradiation, some of the samples will have reached ~2/3 and the remainder will have reached the peak fluence. The former should allow the fluence at which turn-around occurs to be accurately determined, and the latter should allow the fluence at which the initial dimensions/volume are attained to be determined.]

(i) SGL Graphites

These cover both petroleum and pitch coke, and both extrusion and iso-moulding.

NBG-10 (Fig. 18 &19)

This is a pitch coke extruded graphite. The shrinkage in the with grain (WG) direction is slightly larger (at ~2.0%) than in the against grain (AG) direction (at ~1.5%) at the maximum fluence (of ~9 dpa). This is as expected. The scatter on the data is not too large. There is no evidence of turn-around at the peak fluence reached. The volume change behaviour appears to be decreasing linearly with fluence, reaching ~4.5% at the maximum fluence, and the scatter is less than for the individual dimensions.

NBG-20 (Fig. 20 & 21)

This is a petroleum coke extruded graphite. The shrinkage in the WG direction is slightly larger (at ~2.5%) than for NBG-10 at the maximum fluence. However, in the AG direction there is a very large difference, both in comparison with the WG direction, and with NBG-10. The dimensional change in the AG direction is very small being <0.5% at the maximum fluence. It is clear that this graphite exhibits a high level of anisotropy which is not expected based on the relatively low anisotropy calculated from the CTE values in the WG and AG directions. The scatter on the data is not too large, and is broadly the same as that for NBG-10. There is no evidence of turn-around at the maximum fluence reached. The volume change behaviour shows that the rate of change of volume is decreasing with fluence, suggesting it is approaching turn-around. The volume change at the maximum fluence is ~2.5%, which is much less than for NBG-10. The scatter is again less than for the individual dimensions.

NBG-25 (Fig. 23 & 24)

This is a petroleum coke iso-moulded graphite The shrinkage in the WG direction is lower than the other two at the maximum fluence. However, the scatter on the data is much larger. The dimensional change in the AG

direction is much less than in the WG direction, and is somewhere in between that for the other two (NBG) graphites. The scatter on the data is also less. This graphite also exhibits a high level of anisotropy, which is again not apparent from the CTE measurements in the two directions. The volume change behaviour appears to be decreasing linearly with fluence, reaching ~ 4.2% at the maximum fluence, which is similar to NBG-10. The scatter is less than for the individual dimensions.

(ii) Graf Tech Graphites

These cover both petroleum and pitch coke, and both extrusion and iso-moulding.

PCEA (Fig. 25 & 26)

This is a petroleum coke, extruded graphite. The shrinkage in the WG direction is slightly larger (at ~2.5%) than in the AG direction (at ~2.0%) at the maximum fluence (of ~10 dpa). This is as expected. The anisotropy is similar to that for NBG-10, but the shrinkage is higher by ~0.5% in each direction. The scatter on the data is not too large. There is some evidence that the graphite is approaching turn-around at the maximum fluence reached. The volume change behaviour shows it decreasing with fluence, suggesting it is approaching turn-around. The volume change at the maximum fluence is ~6.3%, which is much higher than for NBG-10. The scatter is again less than for the individual dimensions.

PPEA (Fig. 27 & 28)

This is a pitch coke equivalent of PCEA (and also the equivalent grade to NBG-10). The shrinkage in the WG direction is larger (at ~2.0%) than in the AG direction (at ~1.4%) at the maximum fluence (of ~10 dpa). The anisotropy is similar to that for PCEA, but the shrinkage rate is lower. It is very similar to the behaviour of NBG-10 (which would be expected as it is the equivalent grade). The scatter on the data is not large, and arguably less than for NBG-10 and PCEA. The volume change behaviour suggests a rate of change of volume decreasing slightly with fluence. The volume change at the maximum fluence is ~4.5%, which is much less than for PCEA, but very similar to NBG-10 (as expected). The scatter is again less than for the individual dimensions.

PCIB-SFG (Fig. 29 & 30)

This is a petroleum coke, iso-moulded graphite. The shrinkage in the WG direction is lower than the other two GrafTech grades at the maximum fluence (of ~10 dpa). In the WG direction it is generally <1.0%, and in the AG direction <0.5%. However, the scatter on the WG data is quite large. Although the shrinkage rate is low, which is desirable, it appears that turn-around is happening in the AG direction at the maximum fluence reached. This can only be confirmed when the INNOGRAPH 1B data have been

assessed. The volume change behaviour suggests that the rate of change of volume is decreasing slightly with fluence. The volume change at the maximum fluence is only~1.8%, which is much less than for extruded graphites, and even less than that for the other iso-moulded graphite (NBG-25), which reached ~4%. This difference is likely to be the effect of the much smaller grain size used. PCIB is a super-fine grain graphite whereas all the others are medium grain. The scatter on volume is again less than for the individual dimensions.

(iii) Toyo Tanso Graphites

The Toyo Tanso grades cover both petroleum and pitch coke, but both are iso-moulded.

IG-110 (Fig. 31 & 32)

This is a petroleum coke graphite. The shrinkage in the WG direction (at ~2.0%) is again larger than in the AG direction (at ~1.3%) at the maximum fluence (of ~10 dpa). The scatter on the data is quite large, which is consistent with the other two iso-moulded graphites. Although the curve for the AG direction does not show it, there is a possibility that turn-around is occurring. This can only be confirmed when the INNOGRAPH 1B data have been assessed. The volume change behaviour suggests that the rate of change of volume with fluence is still linear at the maximum fluence. The volume change at the maximum fluence is ~5.0%, which is slightly higher than for NBG-25, but much larger than for PCIB. The scatter is very much less than for the individual dimensions.

IG-430 (Fig 33 & 34)

This is a pitch coke graphite. The shrinkage in the WG direction has a very high scatter, ranging from 0% to ~1.5% at the maximum fluence (of ~10 dpa). In the AG direction, most of the samples show a slight growth of up to 0.4%, which has only arisen for some AG samples in NBG-20. The scatter on data is not as large as in the WG direction. The graphite has a high level of anisotropy. The volume change behaviour shows a very large scatter in the data. This is the only graphite of the eight to have such a high scatter. The reason for this is not clear at the moment.

9.3 Dynamic Youngs' Modulus

The variation in DYM for the eight graphites (see Figure. 35 to 42) show an increase in DYM, but by different amounts, and the data show a fair amount of scatter. The behaviour is exactly as would be expected and is typical of the changes observed in all graphites previously irradiated. It is known that all graphites show a significant increase at fairly low fluences. This is attributed to the pinning of mobile dislocations in the lattice structure. The DYM increases by a factor of ~1.6. (In the UK this is referred to as the 'pinning' term). At progressively higher fluences there is a further, but more gradual, increase in DYM attributed to structural changes within the crystals.

(In the UK this is referred to as the 'structure' term.). The data obtained do not show the increases happening at low fluences, and so the true curve has not been fitted. It is possible however to construct the low fluence part of the curve from existing data on previously irradiated graphites.

The DYM of all the extruded graphites have increased by a factor of around 2.4 at the maximum fluence, whereas all the iso-moulded graphites have increased by a factor of between 2.8 and 3.0. (It should be noted that the strength of a graphite also increases with fluence in a similar way to the DYM. The factorial increase is however less than that for the DYM)

9.4 Comparison with Previous Graphites

The only graphite with freely available data that can be used to make comparisons with at the present time is ATR-2E. (Most of the data on other graphites previously irradiated are subject to restrictions, although these might be lifted in the future). Fortunately data for ATR-2E are available at 750^{o}C for both linear dimensional changes and DYM (see Fig. 43-46). ATR-2E was a pitch coke, medium grain extruded graphite and so is closely related to NBG-10 and PPEA. For this reason, the comparison has been limited to these two graphites only.

The comparisons in dimensional change behaviour for NBG-10 and PPEA suggest that they have higher shrinkage rates than ATR-2E although turn-around will occur at a higher fluence. NBG-10 and PPEA also show less anisotropy but increased scatter. For DYM the behaviour is very similar over the fluence range covered. Fortunately data exist for ATR-2E at the lower fluences, and due to the similarity in behaviour of all other graphites in this region, it can be assumed with reasonable confidence that the behaviour of NBG-10 and PPEA will also be similar.

9.5 Conclusions

It was observed that some of graphites tested show similarities in dimensional change behaviour, whereas others show significantly different behaviour. It was evident that the extruded graphites shrink at a faster rate than the iso-moulded graphites in both directions. However, the iso-moulded graphites exhibit a higher anisotropy and also have a higher scatter in the data. Although some of the graphites exhibit similarities up to the maximum fluence achieved, experience has shown that it is unlikely that each will exhibit similar behaviour beyond turn-around. This will only be known after the INNOGRAPH 1B results have been assessed. It is therefore not possible at the present time to rule out any of the graphites. The advantage of the lower shrinkage rates evident for the iso-moulded graphites are likely to be outweighed by a lower fluence at turn-around, a much higher anisotropy, and higher scatter in the data. The DYM variations are as expected and completely consistent with those for previously irradiated graphites.

The comparisons in dimensional change behaviour for NBG-10 and PPEA with a previously irradiated graphite, ATR-2E, has shown that both NBG-10 and PPEA have higher shrinkage rates than ATR-2E, although turn-around

will occur at a higher fluence. NBG-10 and PPEA also show less anisotropy but more scatter. The DYM behaviour, however, is very similar over the fluence range covered.

Further assessments will be carried out whenever future PIE measurements are completed. In the near future there will be the outstanding measurements on the samples from INNOGRAPH1A, and beyond that, the PIE measurements from the second stage irradiation at 750^{o}C (INNOGRAPH1B), and the two stages of the irradiation at 950^{o}C (INNOGRAPH 2A and 2B).

10 Overall Conclusions

The need for reliable material data and properties is a key issue in the development of HTR technology and is especially important for those areas where safety considerations are upper-most. The work done within the HTR-M1 project on graphite behaviour provides a firm platform on which to develop the future HTR technological basis for graphite selection and development both within Europe and the International community of Generation IV. The work done provides a first step in establishing the requirements and behaviour for some of the new graphite materials that will be needed for the new generation of HTR and VHTR reactors being considered for future development.

The first stage of an irradiation programme has been carried out in the Petten HFR involving ~200 samples manufactured from eight different grades of graphite. The samples were first of all pre-characterised, and then irradiated to a maximum of around 1/3 peak fluence at a temperature of 750^{o}C. For the initial pre-characterisation of the samples their initial dimensions, mass, dynamic Young's modulus (DYM), coefficient of thermal expansion (CTE) and thermal diffusivity were measured. (The latter is used to calculate thermal conductivity.) Over 50% of the irradiated samples have been subjected to some PIE, having their dimensions, mass and DYM re-measured and an initial assessment of the graphite behaviour and data has been performed.

Some of graphites tested show similarities in dimensional change behaviour, whereas others show significantly different behaviour. Comparisons with previously irradiated graphite, ATR-2E, for some of the major graphites has shown higher shrinkage rates than ATR-2E, although its likely that turn-around will occur at a higher fluence. It was evident also that the extruded graphites shrink at a faster rate than the iso-moulded graphites in both directions. However, the iso-moulded graphites exhibit a higher anisotropy and also have a higher scatter in the data. Although some of the graphites exhibit similarities up to the maximum fluence achieved, experience has shown that it is unlikely that each will exhibit similar behaviour beyond turn-around. This will only be known after the INNOGRAPH 1B results have been assessed. It is therefore not possible at the present time to rule out any of the graphites. The advantage of the lower shrinkage rates evident for the iso-moulded graphites are likely to be outweighed by a lower fluence at turn-around, a much higher anisotropy, and higher scatter in the data. The DYM

variations are as expected and completely consistent with those for previously irradiated graphites.

Further assessments will be carried out whenever future PIE measurements are completed. In the near future there will be the outstanding measurements on the samples from INNOGRAPH1A, and beyond that, the PIE measurements from the second stage irradiation at 750^{o}C (INNOGRAPH1B), and the two stages of the irradiation at 950^{o}C (INNOGRAPH 2A and 2B).

Oxidation work on the selected graphites suggest that most of the materials have a comparable reactivity. Most of the new graphites were similar or better than V438, an established nuclear graphite from the former German HTR programme. Within the investigated graphites two of the major graphites (PPEA and NBG-10) always showed the lowest rates with the others still within an acceptable range. The exception to this was NBG 20 which was a rate at least a factor of 3 higher. A number of samples of NBG 20 were tested to investigate possible causes (graphitisation, purification, ash content) but to date no obvious reasons for the differences have been found. At this stage in the evaluation therefore the graphite NBG 20 should be considered unsuitable for application to the HTR. It is planned to continue the explorations of NBG 20 within the 6th Framework Project RAPAHEL Project as part of the graphite investigations.

Work done in Germany on the development of SiC coatings for graphite materials (mainly pebbles) for protecting against oxidation from air or steam ingress under severe accident has been reviewed and reported. Investigations were done using the matrix German graphite A3-3 to measure corrosion rate for temperatures up to 1200^{o}C, and on coated pebbles using different graphites. A very good corrosion resistant quality was eventually achieved with IG 110 and V438 using Chemical vapour deposition (CVD). Although the original objective, to develop a coating for A3-3 graphite, had not yet been achieved, the results obtained so far are a very positive indicator and have attracted considerable interest internationally

11 Future work within RAPHAEL-IP

To continue with the remaining measurements on the samples subjected to PIE and update the data base worksheets, producing a further report that assesses the complete data set.

The second stage of this irradiation to full fluence (INNOGRAPH 1B) is to be started in 2006. Around 1/2 of the samples will be replaced by new samples of the selected graphites. These will therefore be taken to full fluence, with the newly installed samples being taken to ~2/3 peak fluence. The new samples are being pre-characterised (within RAPHAEL-IP) and the data will be added to the worksheets when available. The complete set of PIE measurements on the samples at the end of the INNOGRAPH 1B irradiation will not be available until ~2008. The further report will then be produced that assesses the complete data set up to the peak fluence.

A similar irradiation at 950^{o}C is also to be carried out, initially to 1/3 peak fluence, in the RAPHAEL-IP. This is referred to as INNOGRAPH 2A. Pre-characterisation measurements on the prepared samples will be available during 2006. The worksheets will be updated to incorporate these data. The initial PIE measurements are expected to be available from mid 2007, and a report will be produced to assess the results. The second irradiation stage to peak fluence at this temperature (referred to as INNOGRAPH 2B) will be undertaken between 2007 and 2009 and the full set of PIE measurements on the samples at the end of this irradiation will not be available until ~2010. A further report will be produced at this stage.

Corrosion resistance at higher temperature is one of the most critical issues affecting the selection of graphite materials and it is proposed to carry out further testing (started in HTR-M1) on the range of currently selected grades to cover VHTR operation. It is proposed also within RAPHAEL-IP to investigate the oxidation characteristics of the remaining graphites to be introduced into the irradiation experiments at VHTR relevant temperatures This covers selected SGL and UCAR graphites developed since the start of the HTR-M1 programme. Two of these graphites (NBG-17 & NBG-18) have been selected for the industrial reactor developments of PBMR (South Africa) and ANTARES (France).

A further test is also planned to investigate the effects of reheating a pre-irradiated graphite sample to higher temperatures (to simulate accident conditions) to measure the expected recovery in thermal conductivity. Such an experiment has not been performed before and will give an indication of any potential benefit to safety assessments in this area.

For the longer term development of graphite and to minimise the extent of future irradiation testing, work will also be carried out on investigation of microscopic modelling methods for prediction and assessment of graphite behaviour making use of micrographs and tomography images from virgin and irradiated graphite used in the HTR-M1 FP5 project.

Design & Construction Codes demonstrate the quality of design, manufacture, inspection and construction and provide the criteria and procedures needed to ensure a safe and a consistent engineering approach. There are currently no agreed Codes & Standards accepted for use with the modular HTR. There is also a need to develop guidelines and procedures for application and assessment of new (and existing) materials at temperatures relevant to the VHTR. It is important to define such rules and guidelines early enough to give good orientations on the test programmes and make sure that important aspects, required later, are not missed in these programmes. For graphite, design guidelines and curves are to be constructed for the specific graphite grades examined in the HTR-M1 & RAPHAEL irradiation tests.

12 References

Ref **Title**

Ref	Title
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2	M. W. Davies Minutes of meeting with graphite manufacturer UCAR on 26th. March 2002, Notre Dame, France Report No. HTR-M1-02/05-M-2-0-3
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11	M. Davies HTR-M1 Report on preliminary irradiation testing of selected graphites for HTR – Assessment of the results and suitability Report No. HTR-M1 05/12 D 2 4 41
12	K Kuhn Comparison of oxidation behaviour of 8 graphites Report No. HTR-M1 04/11 D 2 0 27
13	B. Schroder Graphite with SiC coatings Report HTR-M1 03/07 2 5 14

Table 1 Graphites proposed by Manufacturers

Supplier	Grade	Coke type	Forming method
GrafTech	PPEA and PPEB	Pitch	Extrusion
GrafTech	PAEA and PAEB	Pitch	Extrusion
GrafTech	PCEA and PCEB	Petroleum	Extrusion
GrafTech	PCMA and PCMB	Petroleum	Moulding
GrafTech	PCIB and PCIC	Petroleum	Iso-moulding
SGL	NBG-10 (previously P3XA2N)	Pitch	Extrusion
SGL	NBG-25 (previously R6810)	Petroleum	Iso-moulding
SGL	NBG-20	Petroleum	Extrusion
Toyo Tanso	IG-110	Petroleum	Iso-moulding
Toyo Tanso	IG-430	Pitch	Iso-moulding

Table 2 Details of Graphite Block sizes provided by the manufacturers

Major Grades	Procurement sizes
PCIB-SFG	three discs with diameter of 265 mm and height of 45 mm
PCEA	three half-discs with original diameter of 465 mm and height of 45 mm
NBG-10	block of dimensions 305 x 320 x 95 mm
NBG-25	block of dimensions 520 x 200 x 130 mm
IG-110	block of dimensions 400 x 200 x 100 mm
Minor Grades	Procurement sizes
PPEA	three discs with diameter of 265 mm and height of 45 mm
NBG-20	four discs with diameter of 110 and height of 296 mm
IG-430	two blocks of dimensions 400 x 200 x 50 mm

Table 3 Number of specimens per grade that were included in INNOGRAPH-1A

Supplier	Grade	Total per grade	Orientation	Total per orientation	Centre		Edge	
					6 mm	12 mm	6 mm	12 mm
SGL-Carbon	NBG-10	32	AG	17	7	3	5	2
			WG	15	5	3	5	2
	NBG-25	30	AG	15	5	3	5	2
			WG	15	5	3	5	2
	NBG-20	14	AG	7	5	2	-	-
			WG	7	5	2	-	-
Toyo Tanso	IG-110	30	AG	15	5	3	5	2
			WG	15	5	3	5	2
	IG-430	18	AG	9	5	2	2	-
			WG	9	5	2	2	-
Graftech	PCEA	30	AG	15	5	3	5	2
			WG	15	5	3	5	2
	PCIB	30	AG	15	5	3	5	2
			WG	15	5	3	5	2
	PPEA	14	AG	7	5	2	-	-
			WG	7	5	2	-	-

Table 4 Graphites investigated in FZJ Oxidation Tests

Manufacturer	Graphite	Apparent Density [g/cm^3]
UCAR	PCEA	1,82
	PCIB	1,85
	PPEA	1,84
SGL	NBG-10	1,81
	NBG-20	1,78
	NBG-25	1,81
TOYO TANSO	IG 110	1,77
	IG 430	1,82

Table 5 Number of high dose specimens per grade, including dose range

Supplier	Grade	Total per grade	Dose (dpa_g)	Orient.	Total per orientation	Centre		Edge	
						6 mm	12 mm	6 mm	12 mm
SGL-Carbon	NBG-10	14	5.93 - 9.15	AG	7	4	1	2	-
				WG	7	3	1	3	-
	NBG-25	14	6.31 - 9.84	AG	7	4	1	2	-
				WG	7	3	1	3	-
	NBG-20	8	6.60 - 9.14	AG	4	4	-	-	-
				WG	4	4	-	-	-
Toyo Tanso	IG-110	14	6.68 - 9.84	AG	7	4	1	2	-
				WG	7	3	1	3	-
	IG-430	8	6.00 - 9.63	AG	4	3	-	1	-
				WG	4	3	-	1	-
Graftech	PCEA	14	6.66 - 9.78	AG	7	3	1	3	-
				WG	7	3	1	3	-
	PCIB	14	6.16 - 9.87	AG	7	3	1	3	-
				WG	7	3	1	3	-
	PPEA	11	5.73 - 9.17	AG	6	5	1	-	-
				WG	5	4	1	-	-

Table 6 Average results of high dose specimens per group after irradiation

			Nr. of specimens	Dose (dpa_g)	? x (%)	? d (%)	? l (%)	? vol. (%)	? ? (%)	? E (%)
NBG-10	CENTRE	AG	5	7.99	-1.5	-1.2	-1.4	-4.0	4.0	99
		WG	4	8.24	-1.3	-1.2	-1.8	-4.2	4.2	109
	EDGE	AG	2	8.46	-1.5	-1.4	-1.2	-3.9	4.0	105
		WG	3	7.58	-1.0	-1.1	-1.7	-3.8	3.8	100
NBG-25	CENTRE	AG	5	8.41	-1.7	-1.7	-0.6	-3.9	4.1	168
		WG	4	7.91	-0.4	-1.1	-1.8	-3.7	3.8	159
	EDGE	AG	2	8.67	-1.6	-1.4	-0.7	-3.6	3.8	149
		WG	3	8.72	-0.5	-1.1	-1.9	-3.8	4.0	143
NBG-20	CENTRE	AG	4	8.31	-0.4	-0.2	-1.7	-2.2	1.9	103
		WG	4	8.17	-0.2	0.1	-2.3	-2.3	2.1	104
IG-110	CENTRE	AG	5	8.36	-1.7	-1.8	-1.0	-4.4	4.9	137
		WG	4	8.53	-1.3	-1.4	-2.0	-4.6	4.8	133
	EDGE	AG	2	8.54	-1.5	-1.8	-1.2	-4.7	4.8	145
		WG	3	8.08	-1.1	-1.2	-2.0	-4.4	4.5	125
IG-430	CENTRE	AG	3	7.96	-1.0	-0.9	0.2	-1.6	1.6	159
		WG	3	8.20	0.1	-0.5	-1.2	-2.0	1.9	145
	EDGE	AG	1	7.75	0.2	-0.1	-0.3	-0.3	0.3	139
		WG	1	8.42	-0.1	-0.1	-1.4	-1.5	1.5	140
PCEA	CENTRE	AG	4	8.51	-2.1	-1.9	-2.0	-5.7	6.0	112
		WG	4	8.51	-1.7	-1.8	-2.4	-5.8	6.1	118
	EDGE	AG	3	7.99	-2.2	-2.0	-1.8	-5.7	6.0	113
		WG	3	7.86	-1.7	-1.7	-2.3	-5.6	5.9	111
PCIB	CENTRE	AG	4	8.47	-0.8	-0.6	-0.3	-1.6	1.7	144
		WG	4	8.55	-0.3	-0.5	-0.7	-1.6	1.6	145
	EDGE	AG	3	8.90	-0.7	-0.7	-0.2	-1.6	1.6	145
		WG	3	8.79	-0.2	-0.4	-1.0	-1.7	1.7	141
PPEA	CENTRE	AG	6	7.85	-1.6	-1.3	-1.2	-4.0	4.1	105
		WG	5	8.05	-1.3	-1.2	-1.8	-4.2	4.3	107

Table 7 Average results of high dose specimens per grade after irradiation.

?x is the flattening of the specimen, ?d the diameter, ?l the length and ?vol. the volume

	Nr. of specimens	Dose (dpa_g)	? x (%)	? d (%)	? l (%)	?vol. (%)	? ? (%)	? E (%)
NBG-10	14	8.0	-1.3	-1.2	-1.6	-4.0	4.0	102.8
NBG-25	14	8.4	-1.1	-1.4	-1.2	-3.8	3.9	157.4
NBG-20	8	8.2	-0.3	-0.1	-2.0	-2.3	2.0	103.7
IG-110	14	8.4	-1.4	-1.6	-1.5	-4.5	4.8	134.7
IG-430	8	8.1	-0.3	-0.5	-0.6	-1.6	1.5	148.9
PCEA	14	8.3	-1.9	-1.8	-2.1	-5.7	6.0	114.0
PCIB	14	8.7	-0.5	-0.5	-0.6	-1.6	1.6	144.1
PPEA	11	7.9	-1.5	-1.3	-1.5	-4.1	4.2	105.6

Table 8 Pre-characterisation measurements for IG-110

Graphite materials property database

Graphite: IG-110
Manufacturer: Toyo tanso
Coke: Petroleum
Grain size: Fine
Process: Iso-moulded

Unirradiated properties

Spec code	Block region	Orient-ation	Length L_o (mm)	Diam D_o (mm)	Diam x (mm)	Vol V_o (mm^3)	Mass Mo (g)	Density po (g/cm^3)	DYM E_o (GPa)	CTE α_o ($x10^{-6}/K$) 30-120	CTE 30-750	CTE 30-200	Thermal conductivity (W/m/K) 27°C	100°C	200°C	300°C	400°C	500°C	600°C	700°C
T004	CENTRE	AG	11.928	7.978	7.777	592.2	1.04036	1.757	9.2	3.35	4.52	3.55								
T005	CENTRE	AG	11.909	7.982	7.716	589.9	1.03910	1.762	9.1	3.57	4.68	3.79								
T006	CENTRE	AG	11.908	7.976	7.726	589.5	1.03201	1.751	9.3	3.51	4.55	3.68								
T007	CENTRE	AG	5.968	7.987	7.719	295.9	0.51890	1.754	9.1	3.26	4.50	3.48	117	111	102	86	82	72	70	62
T008	CENTRE	AG	5.933	7.986	7.729	294.3	0.51267	1.742	8.7	3.37	4.55	3.60	114	107	94	85	79	68	66	59
T009	CENTRE	AG	5.918	7.983	7.716	293.2	0.51242	1.748	8.7	3.35	4.57	3.58	116	109	99	85	81	72	68	61
T010	CENTRE	AG	5.837	7.986	7.725	289.5	0.50757	1.754	8.7	3.37	4.55	3.60	114	107	94	85	79	68	66	59
T011	CENTRE	AG	6.012	7.989	7.716	298.2	0.52359	1.756	8.8	3.45	4.65	3.69	116	106	96	84	79	71	67	60
T022	CENTRE	WG	5.928	7.982	7.631	292.1	0.51685	1.769	10.5	2.95	4.14	3.14	130	123	110	97	90	80	74	71
T023	CENTRE	WG	5.948	7.984	7.636	293.3	0.51998	1.773	10.8	2.96	4.15	3.16	129	122	109	96	87	80	74	71
T024	CENTRE	WG	5.935	7.989	7.633	292.8	0.52074	1.778	10.2	2.97	4.17	3.19	128	121	108	95	85	79	74	70
T025	CENTRE	WG	5.978	7.984	7.759	296.9	0.52684	1.774	11.1	2.93	4.14	3.15	130	125	112	99	89	83	76	72
T026	CENTRE	WG	5.876	7.982	7.631	289.5	0.51752	1.788	10.7	2.90	4.12	3.12	132	129	115	102	93	86	78	74
T027	CENTRE	WG	12.073	7.994	7.710	599.1	1.05841	1.767	11.3	3.20	4.25	3.38								
T029	CENTRE	WG	11.915	7.998	7.709	591.7	1.04984	1.774	11.2	3.02	4.12	3.20								
T031	CENTRE	WG	11.954	7.984	7.760	594.3	1.05234	1.771	10.6	3.09	4.03	3.12								
T035	EDGE	AG	11.949	7.989	7.722	592.8	1.04861	1.769	9.5	3.05	4.54	3.28								
T036	EDGE	AG	12.014	7.995	7.770	598.4	1.05726	1.767	9.4	3.54	4.57	3.76								
T037	EDGE	AG	5.957	7.993	7.719	295.7	0.52301	1.769	9.4	3.35	4.52	3.57	121	109	100	88	83	73	70	63
T039	EDGE	AG	6.044	7.979	7.712	299.1	0.52945	1.770	9.3	3.35	4.52	3.57	121	109	100	88	83	73	70	63
T041	EDGE	AG	5.907	7.979	7.715	292.4	0.51697	1.768	9.4	3.35	4.53	3.57	120	110	100	88	82	74	70	63
T043	EDGE	AG	5.998	7.978	7.714	296.8	0.52557	1.771	9.2	3.36	4.55	3.57	120	112	100	88	82	75	70	63
T045	EDGE	AG	5.886	7.983	7.716	291.5	0.51556	1.768	9.4	3.36	4.55	3.57	120	112	100	88	82	75	70	63
T047	EDGE	WG	5.970	7.949	7.689	293.3	0.51869	1.768	10.7	2.94	4.12	3.15	134	123	112	98	90	83	76	72
T048	EDGE	WG	5.895	7.991	7.706	292.3	0.51797	1.772	10.6	2.94	4.12	3.15	134	123	112	98	90	83	76	72
T049	EDGE	WG	5.907	7.994	7.710	293.1	0.51919	1.771	10.5	2.94	4.12	3.15	134	123	112	98	90	83	76	72

Table 9 Post-irradiation measurements for IG-110

Graphite materials property database

Graphite:	IG-110
Manufacturer:	Toyo tanso
Coke:	Petroleum
Grain size:	Fine
Process:	Iso-moulded

Irrad temp (°C)	Fluence		Irradiated properties																
	DPA	EDND (10E20)	Length L_i (mm)	Diam D_i (mm)	Diam x_i (mm)	Vol V_i (mm^3)	Mass M_i (g)	Density ρ_i (g/cm^3)	DYM E_i (GPa)	CTE α_i ($x10^{-6}/K$)			Thermal conductivity (W/m/K)						
										30-120	30-750	30-200	27°C	100°C	200°C	300°C	400°C	500°C	600°C	700°C
750	7.73	60.8	11.7906	7.8506	7.6296	566.19	1.04038	1.8375	21.40											
750	0.00	0.0																		
750	0.00	0.0																		
750	6.85	53.9	5.9104	7.8778	7.6288	285.36	0.51834	1.8164	18.54											
750	7.71	60.7	5.8713	7.8568	7.6065	281.93	0.51211	1.8164	18.14											
750	9.84	77.4	5.8699	7.8066	7.5738	278.53	0.51242	1.8398	22.85											
750	0.00	0.0																		
750	9.68	76.2	5.9645	7.816	7.5689	283.47	0.5321	1.8771	24.60											
750	6.68	52.6	5.8438	7.8927	7.5714	281.98	0.51638	1.8313	20.63											
750	9.10	71.6	5.8202	7.8757	7.5514	279.56	0.52003	1.8602	27.87											
750	0.00	0.0																		
750	9.73	76.6	5.8344	7.8708	7.5936	280.72	0.52674	1.8764	28.95											
750	0.00	0.0																		
750	8.60	67.7	11.8466	7.8731	7.613	570.91	1.05766	1.8526	24.91											
750	0.00	0.0																		
750	0.00	0.0																		
750	0.00	0.0																		
750	0.00	0.0																		
750	7.99	62.9	5.8855	7.8547	7.6082	282.52	0.52292	1.8509	22.05											
750	9.09	71.5	5.9687	7.8233	7.5965	284.53	0.52905	1.8594	23.90											
750	0.00	0.0																		
750	0.00	0.0																		
750	0.00	0.0																		
750	7.87	61.9	5.8473	7.8494	7.6055	280.34	0.51847	1.8494	24.42											
750	9.12	71.8	5.7605	7.8788	7.6142	277.94	0.51723	1.8609	26.36											
750	0.00	0.0																		

Table 10 Fractional property changes for IG-110

Graphite materials property database								Graphite:	IG-110														
								Manufacturer:	Toyo tanso														
								Coke:	Petroleum														
								Grain size:	Fine														
								Process:	Iso-moulded														
				Fractional change																			
Location	Orientation	Fluence		Thermal conductivity																			
		DPA	EDND (10E20)	Length L (%)	Diam D (%)	Diam x (%)	Vol V (%)	Mass M	Density ρ	DYM E (Gpa)	CTE 30-120	CTE 30-750	CTE 30-200	27°C	100°C	200°C	300°C	400°C	500°C	600°C	700°C		
CENT	AG	7.73	60.8	-1.1478	-1.5932	-1.8941	-4.3905	1.9E-05	0.04594	2.33575													
CENT	AG	0.00	0.0																				
CENT	AG	0.00	0.0																				
CENT	AG	6.85	53.9	-0.9646	-1.3619	-1.166	-3.5593	-0.0011	0.03576	2.04624													
CENT	AG	7.71	60.7	-1.0461	-1.6123	-1.5807	-4.1982	-0.0011	0.04266	2.07835													
CENT	AG	9.84	77.4	-0.8158	-2.2105	-1.8459	-4.997	-2E-16	0.05264	2.61332													
CENT	AG	0.00	0.0																				
CENT	AG	9.68	76.2	-0.797	-2.1605	-1.9009	-4.9259	0.01625	0.06891	2.78274													
CENT	WG	6.68	52.6	-1.4279	-1.1235	-0.781	-3.4631	-0.0009	0.03495	1.96152													
CENT	WG	9.10	71.6	-2.156	-1.3611	-1.1014	-4.6773	9E-05	0.04918	2.57512													
CENT	WG	0.00	0.0																				
CENT	WG	9.73	76.6	-2.404	-1.4232	-2.1288	-5.4594	-0.0002	0.05755	2.59676													
CENT	WG	0.00	0.0																				
CENT	WG	8.60	67.7	-1.8747	-1.5096	-1.2585	-4.7058	-0.0007	0.04864	2.19934													
CENT	WG	0.00	0.0																				
CENT	WG	0.00	0.0																				
CENT	AG	0.00	0.0																				
EDGE	AG	0.00	0.0																				
EDGE	AG	7.99	62.9	-1.1989	-1.7255	-1.4303	-4.4531	-0.0002	0.04643	2.34635													
EDGE	AG	9.09	71.5	-1.2508	-1.9553	-1.4926	-4.8806	-0.0007	0.05053	2.56371													
EDGE	AG	0.00	0.0																				
EDGE	AG	0.00	0.0																				
EDGE	AG	0.00	0.0																				
EDGE	WG	7.87	61.9	-2.0525	-1.2579	-1.0851	-4.4312	-0.0004	0.04591	2.27349													
EDGE	WG	9.12	71.8	-2.2739	-1.4076	-1.1853	-4.9093	-0.0014	0.0501	2.48352													
EDGE	WG	0.00	0.0																				

Figure 1 Effect of Irradiatio on Dimensional Change for ATR-2E German Graphite

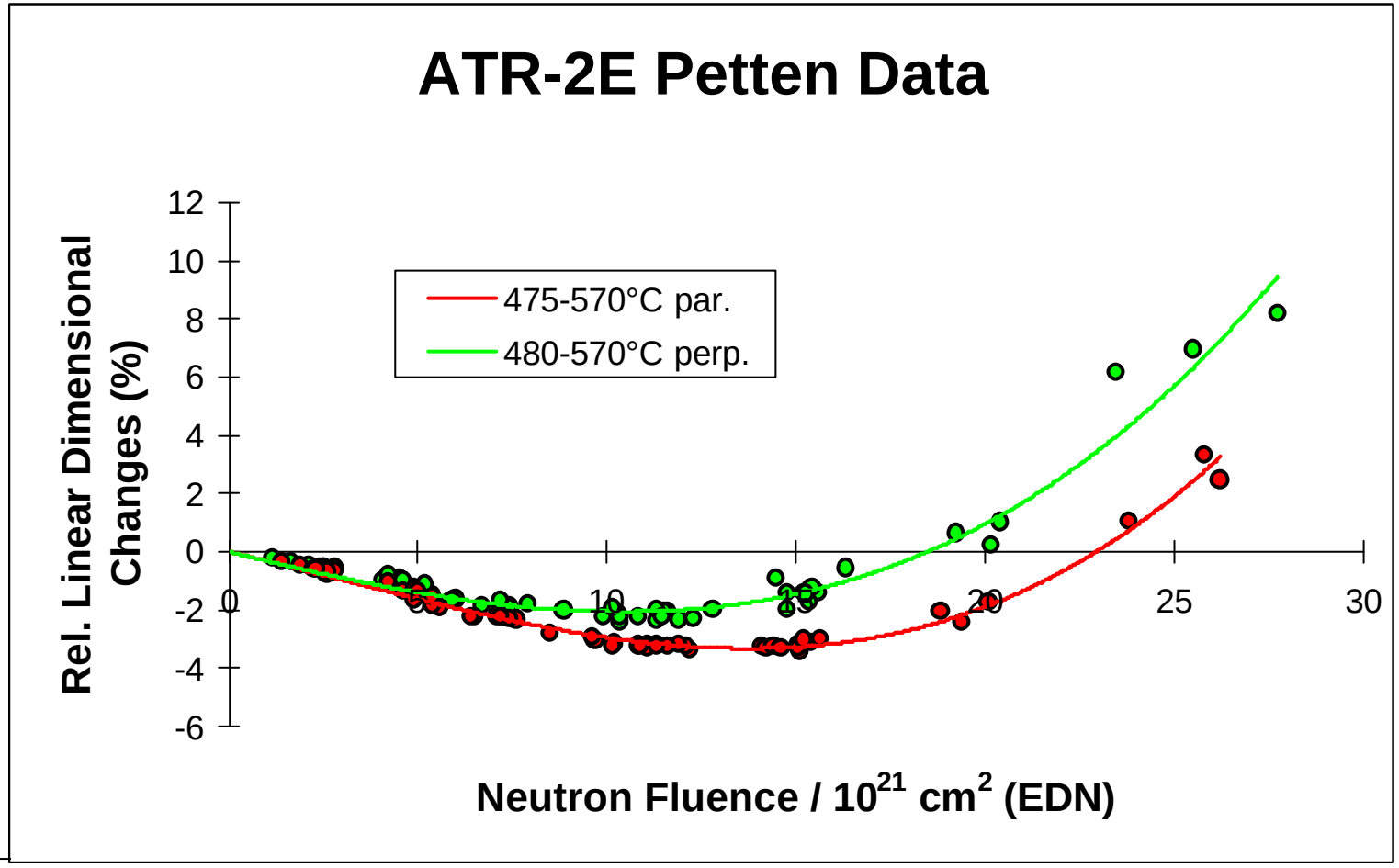


Figure 2 Relative Young's modulus changes for ATR-2E graphite

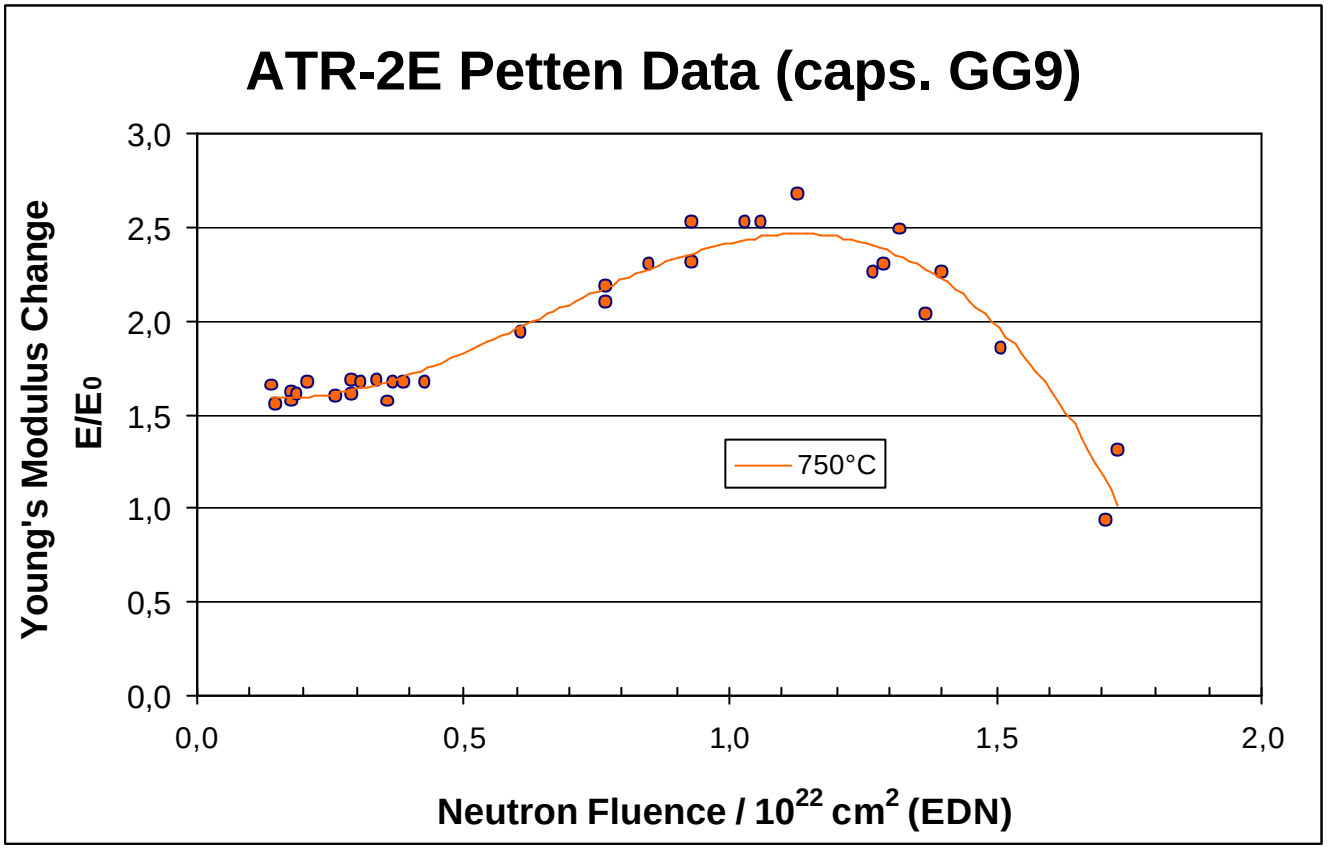
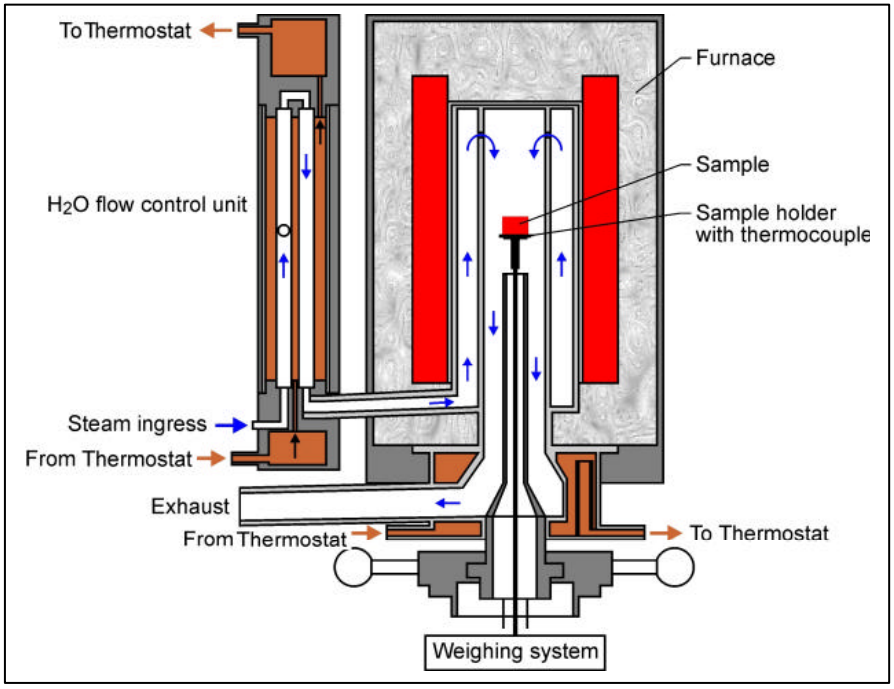
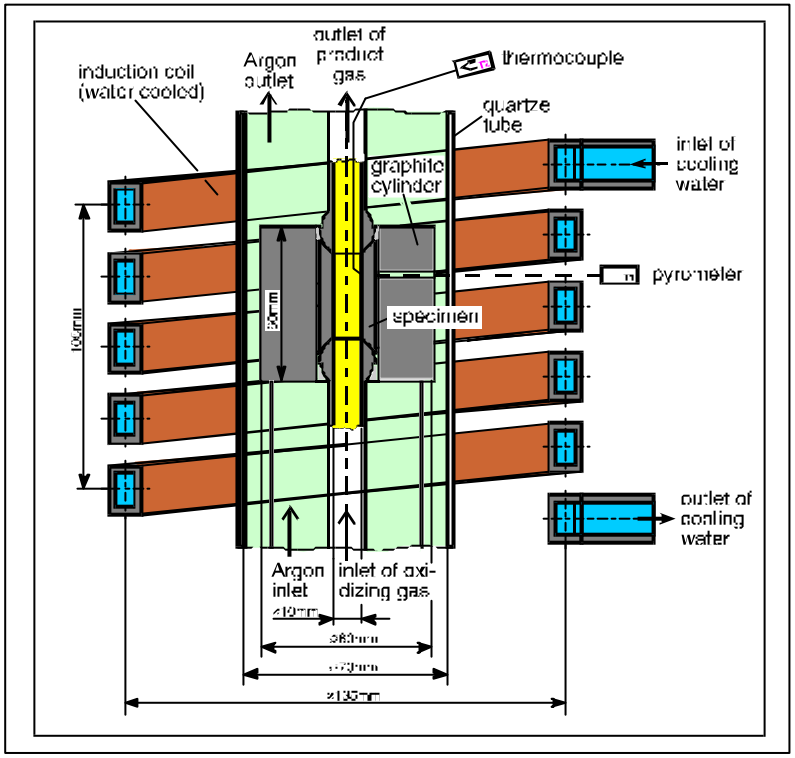


Figure 3 Schematics of THERA & INDEX Test Facilities used in the Graphite Oxidation Work

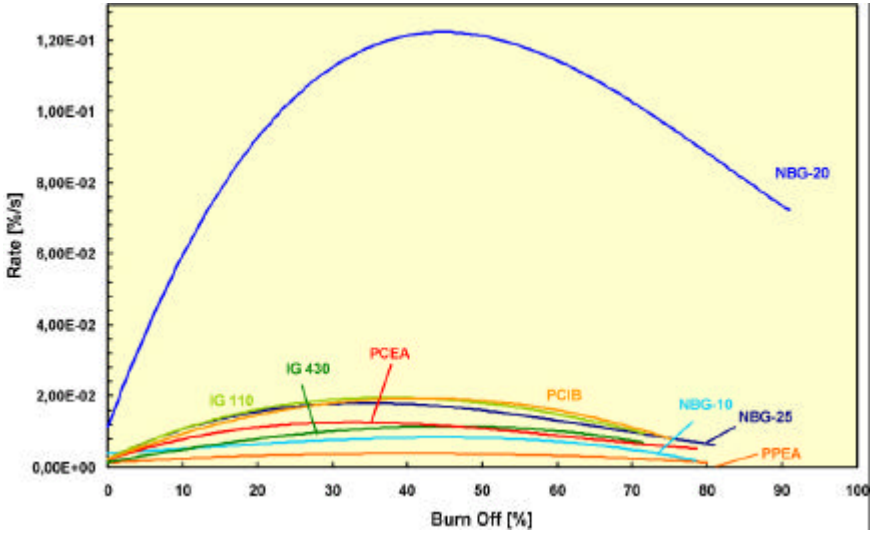




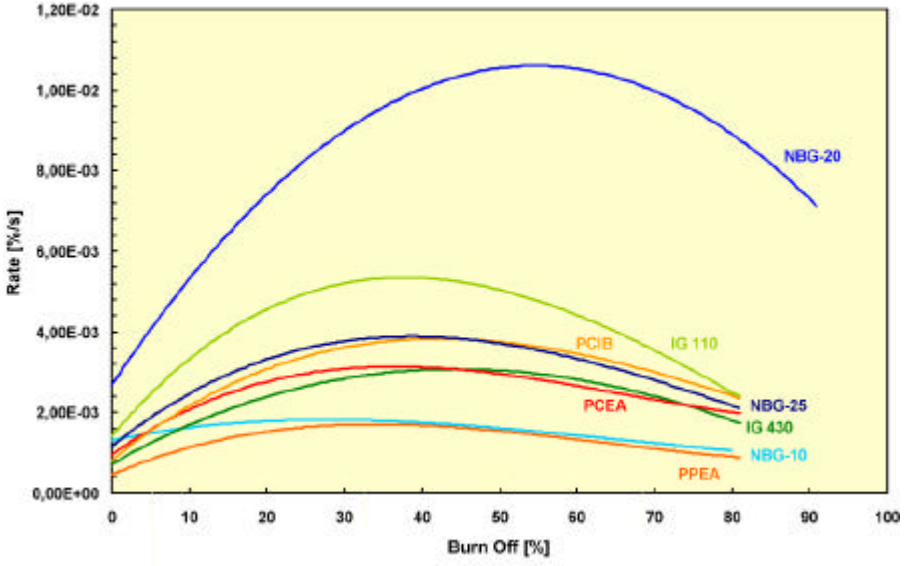
Schematic of THERA test facility

Schematic of INDEX test facility

Figure 4 Oxidation Test Results in Air & Oxygen



Rate versus burn off curves for oxidation in O_2 at 700 °C



Rate versus burn off curves for oxidation in air at 700 °C

Figure 5 Specimen Details

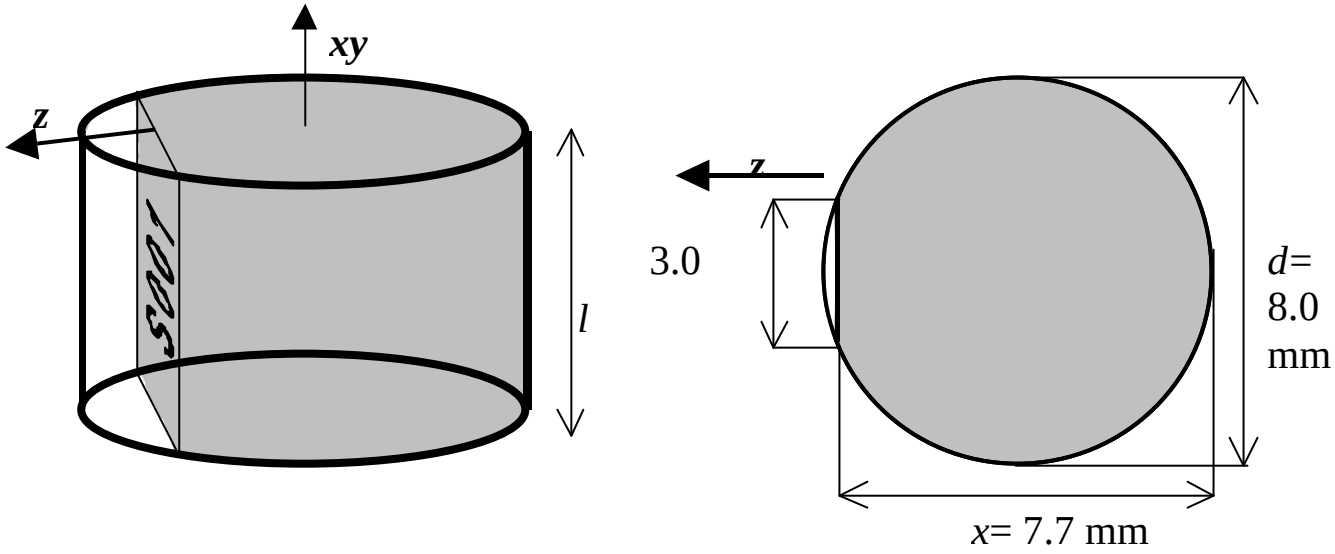


Figure 6 Specimen selection scheme for CTE measurements

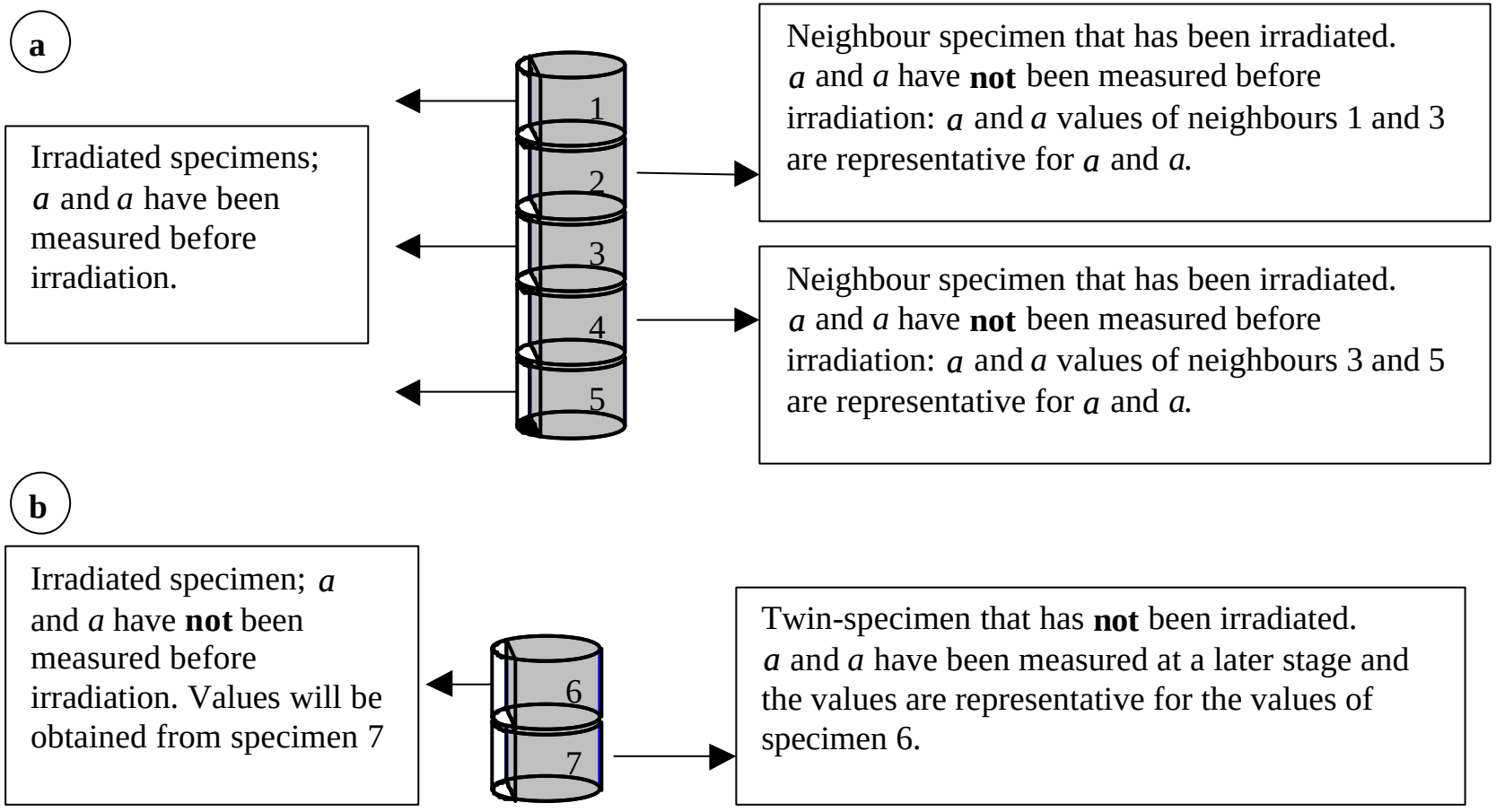


Figure 7 Pre Irradiation properties of Graphites

Property ranges before irradiation

		Min	Max
Density	g/cm^3	1.713	1.862
DYM	GPa	8.2	12.4
$CTE_{30\text{-}120°C}$	10^{-6} 1/K	2.8	4.7
$CTE_{30\text{-}750°C}$	10^{-6} 1/K	4.0	5.8
λ (27°C)	W/mK	114	169
λ (700°C)	W/mK	59	87

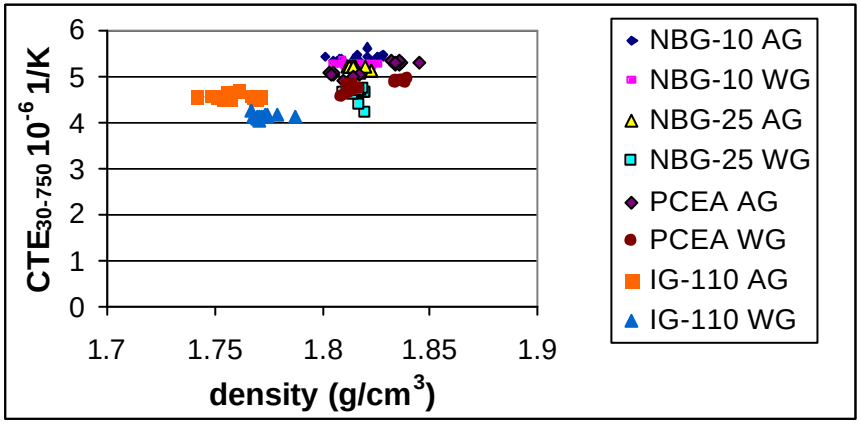


Figure 8 Dose variation of specimens with position in INNOGRAPH-01A

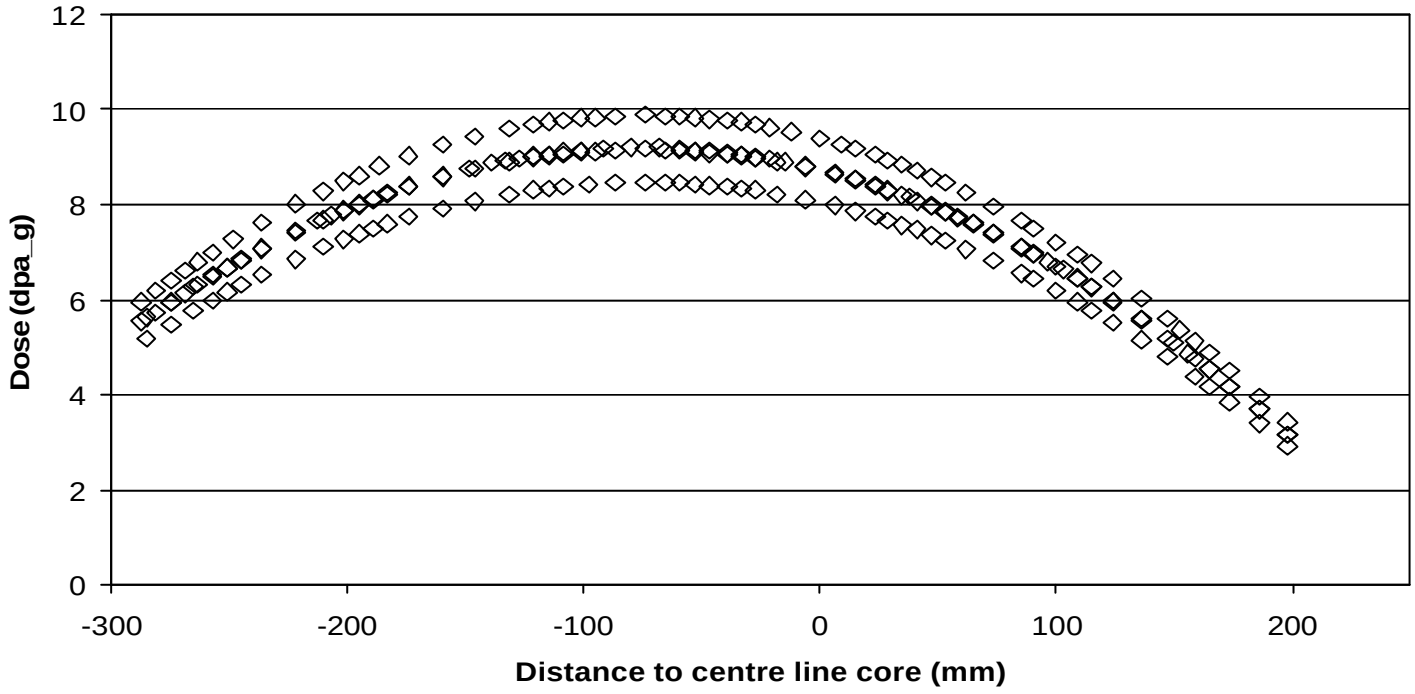
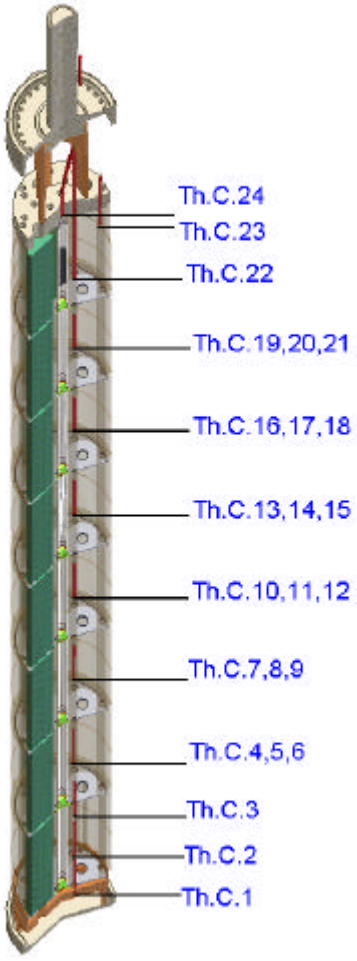
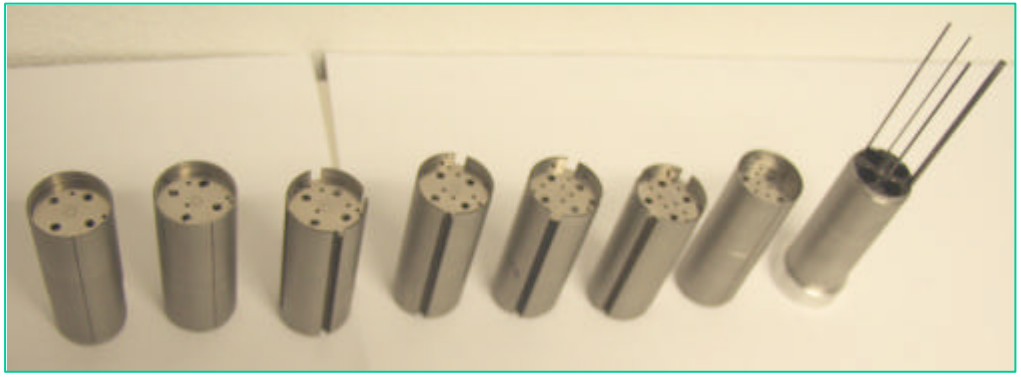


Figure 9 Details of INNOGRAPH A1 Test Facility





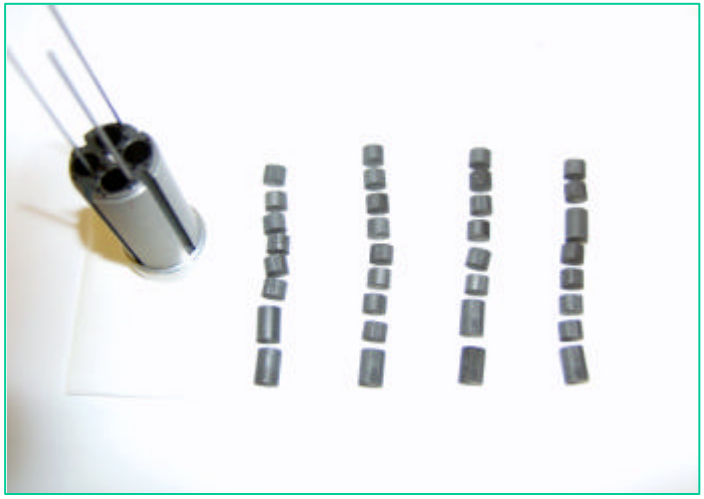
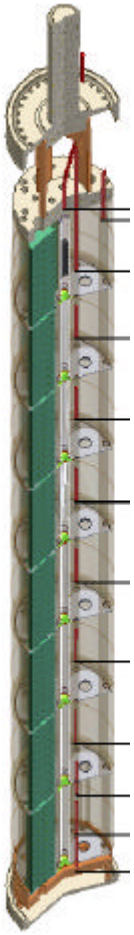
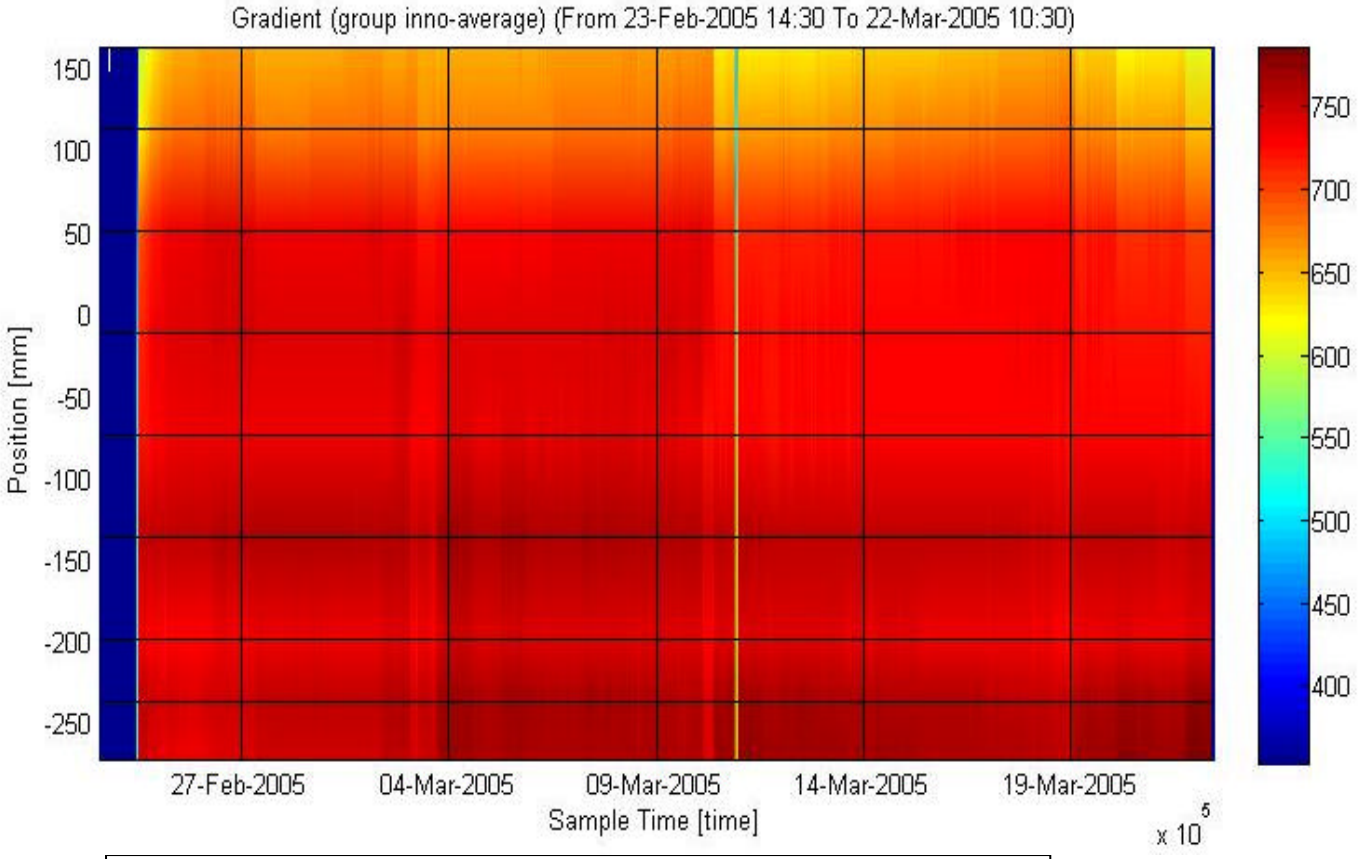


Figure 10 INNOGRAPH 1A Temperature Performance

- **6 drums at ~750°C**
- **2 drums at ~ 670°C**





Every Thermocouple reading is stored and available for analysis

Figure 11 Layout of Shielded Facilities

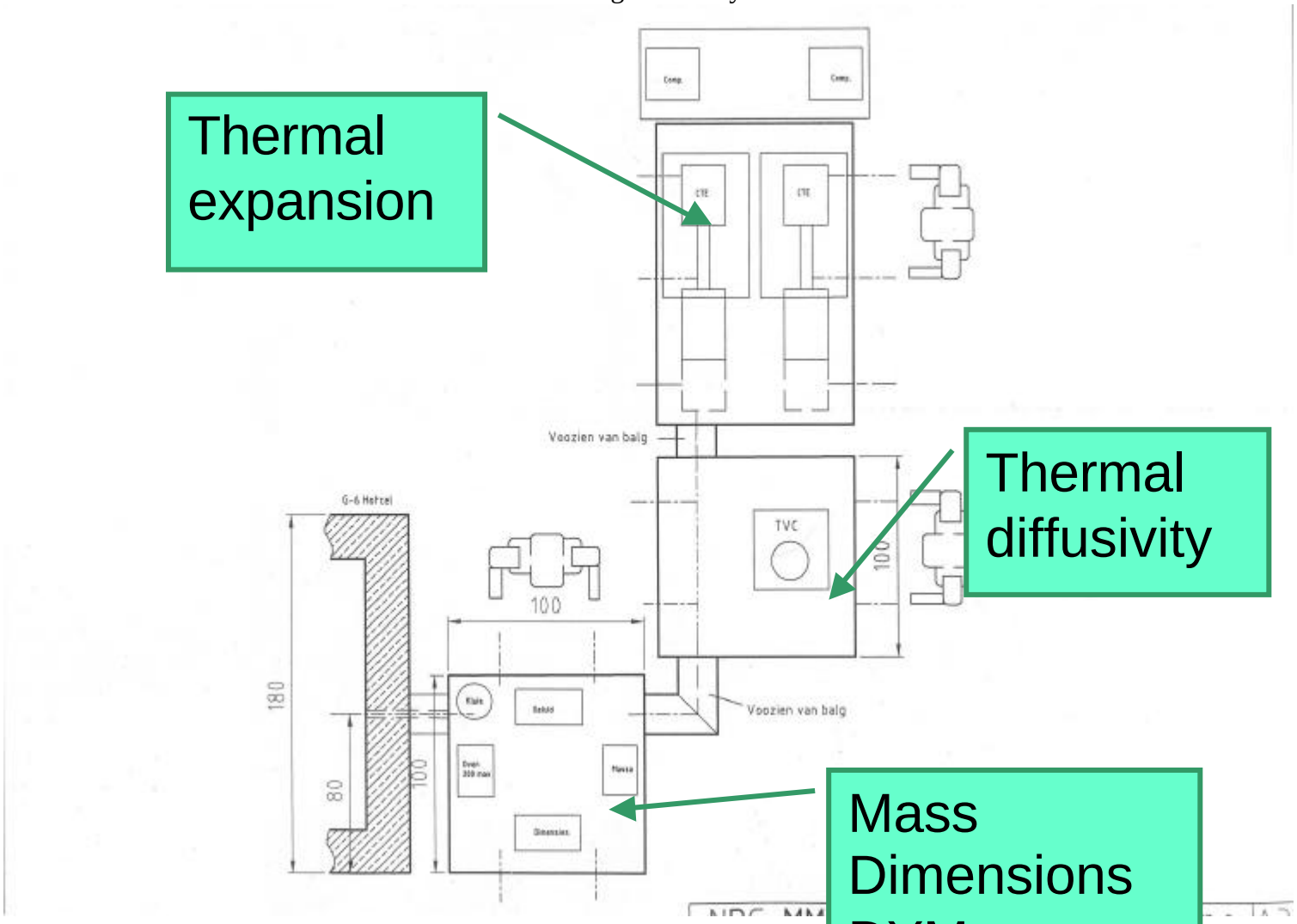
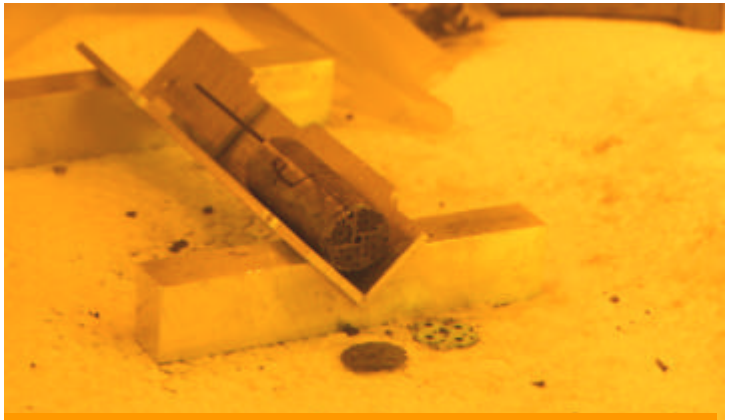


Figure 12 Retrieving Specimens in Lead Cells

Drum



Removing cover

Ready to push specimens out

200 specimens!

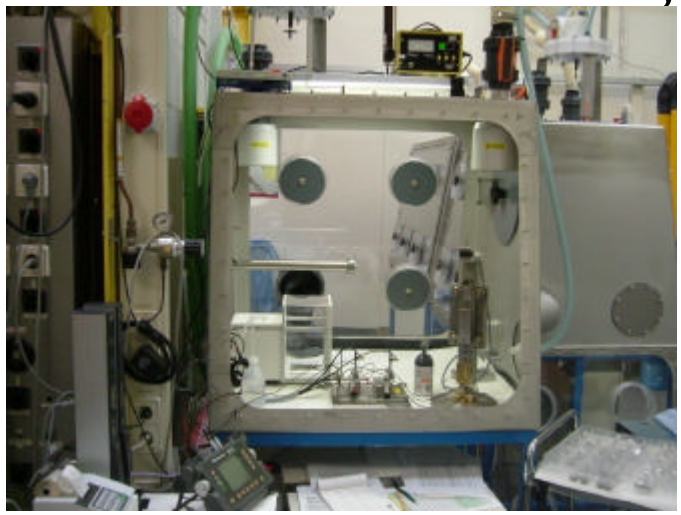
Figure 13 Glove Boxes

Figure 14 Post Irradiation Examination

Post Irradiation Examination

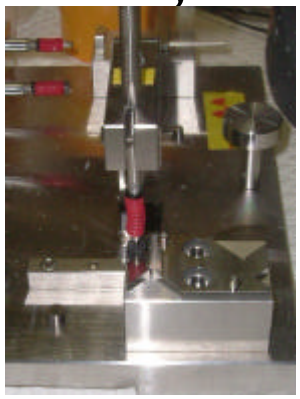
Figure 15 Measurement Facilities in Box 1

BOX1:Dimensions, Mass, DYM

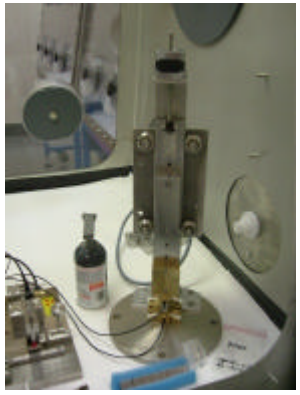


BOX1

BOX 1 is connected to hot cell, sample transport between hot cell and glove box is easy



Dimensions



DYM

Figure 16 Measurement Facilities in Box 2

BOX2:Thermal Diffusivity

NRG invested in new equipment:

NETZSCH LFA 457 MicroFlash™

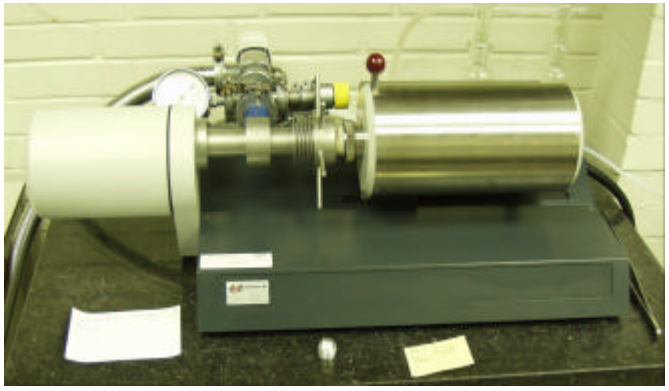
- RT- 1100ºC
- DIN and ASTM standards
- 30 specimens per week can be measured.
- Made suitable for usage in glove box and hot cell

Figure 17 Measurement Facilities in Box 3

BOX3:Thermal Expansion

In BOX3 two dilatometers are installed

Netzsch DIL 402 C



- RT- 1600ºC
- Inert environment (He)
- DIN and ASTM standards
- Made suitable for usage in glove box and hot cell

Figure 18 Volume change – effect of Irradiation

PIE INNOGRAPH 1A

Volume change:

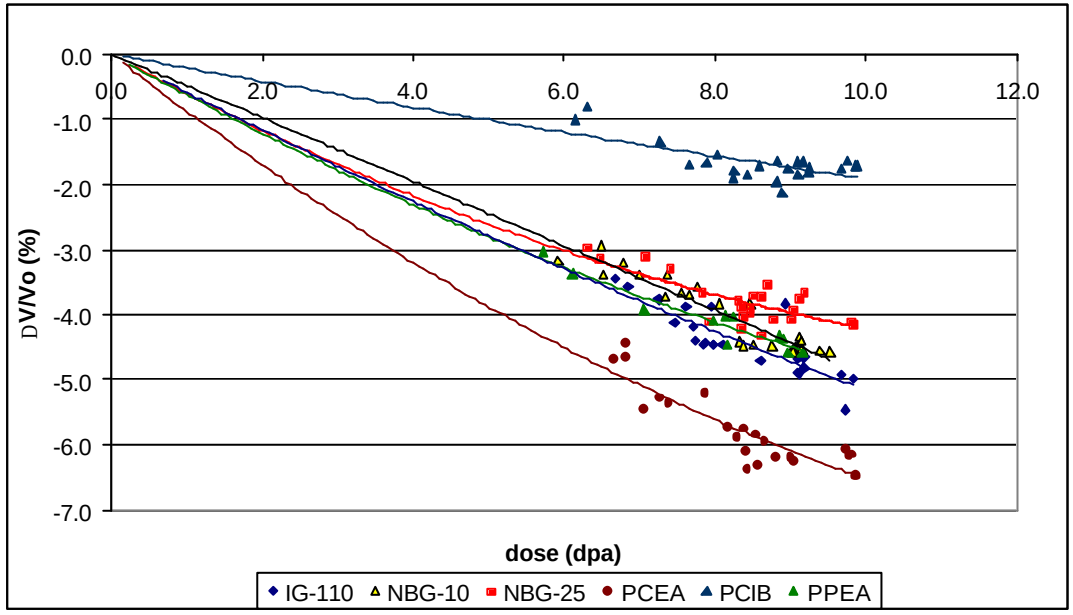


Figure 19 Dimensional change behaviour for NBG-10 (WG & AG)

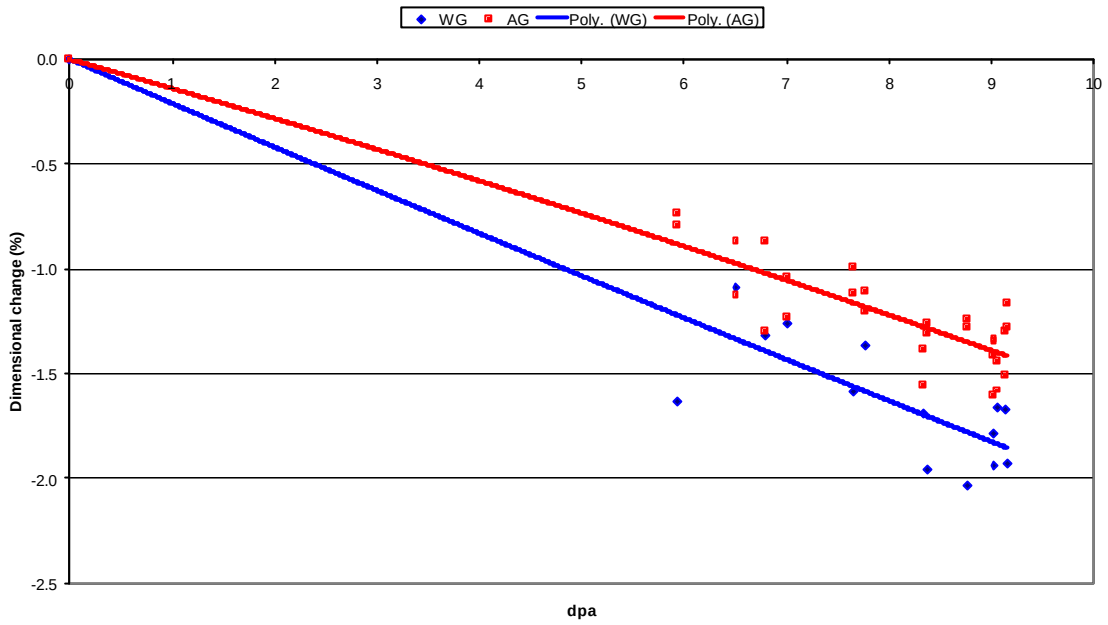


Figure 20 Volume change behaviour for NBG-10 (WG & AG)

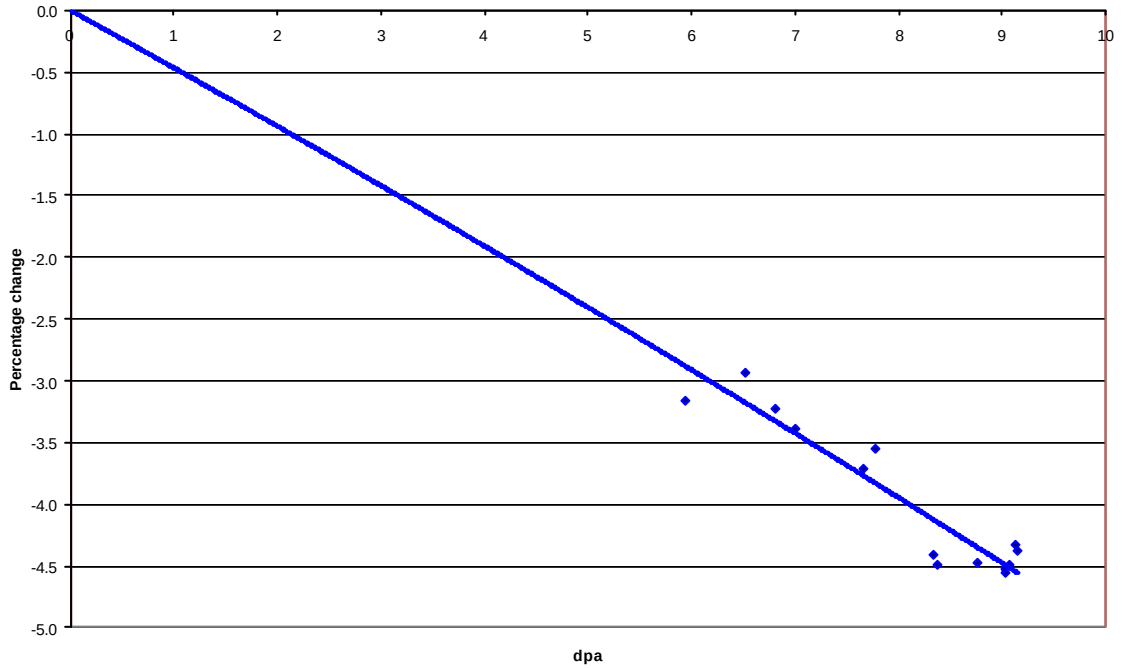


Figure 21 Dimensional change behaviour for NBG-20 (WG & AG)

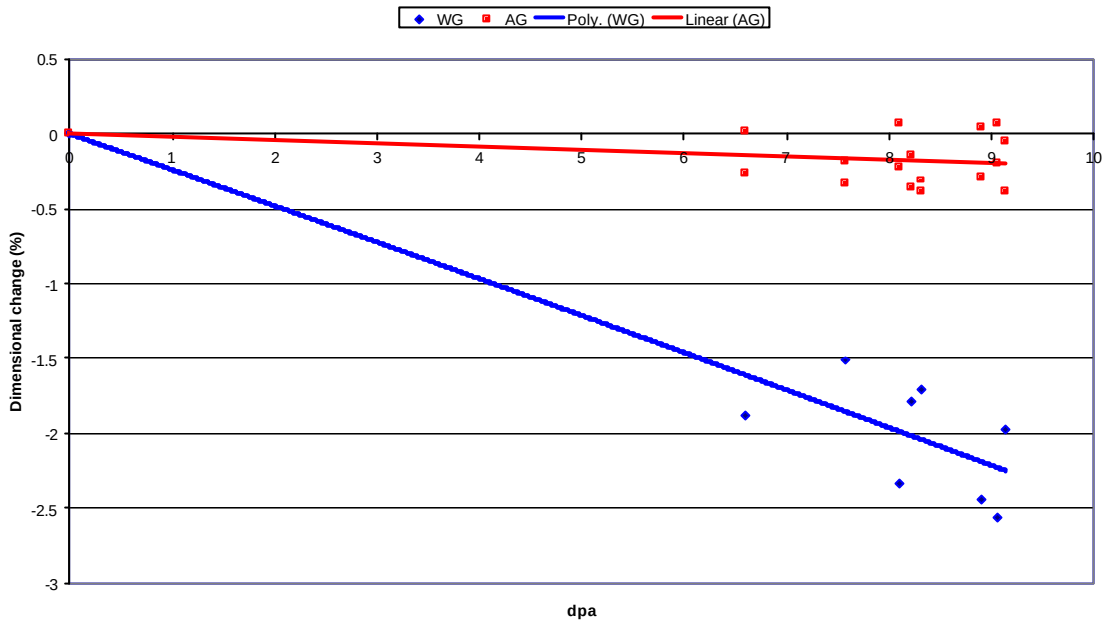


Figure 22 Volume change behaviour for NBG-20 (WG & AG)

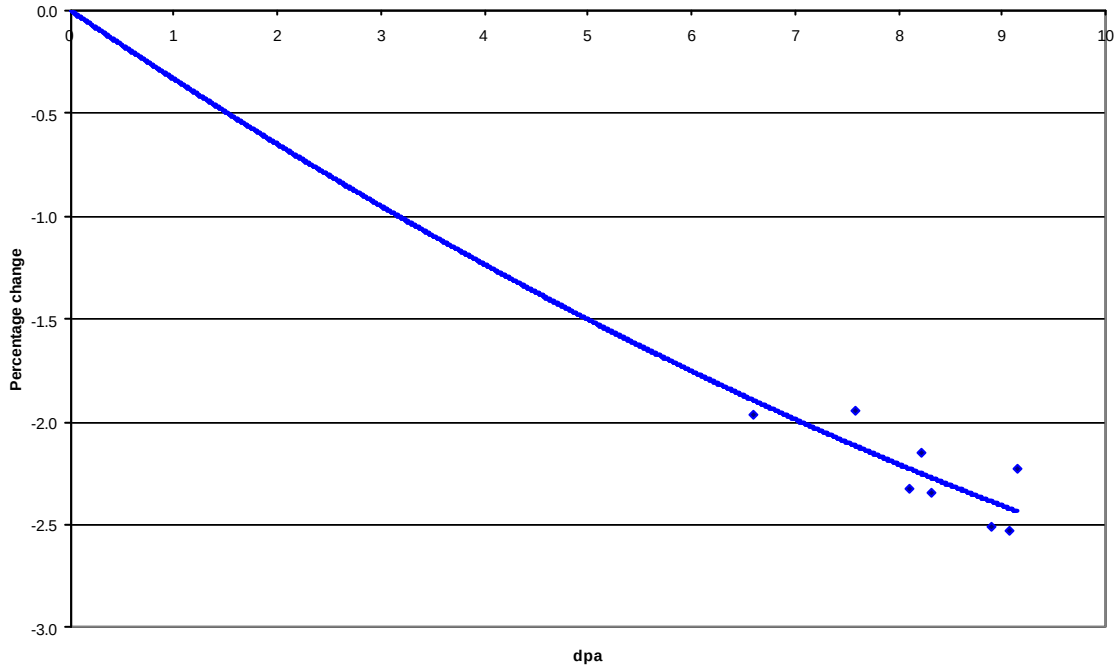


Figure 23 Dimensional change behaviour for NBG-25 (WG & AG)

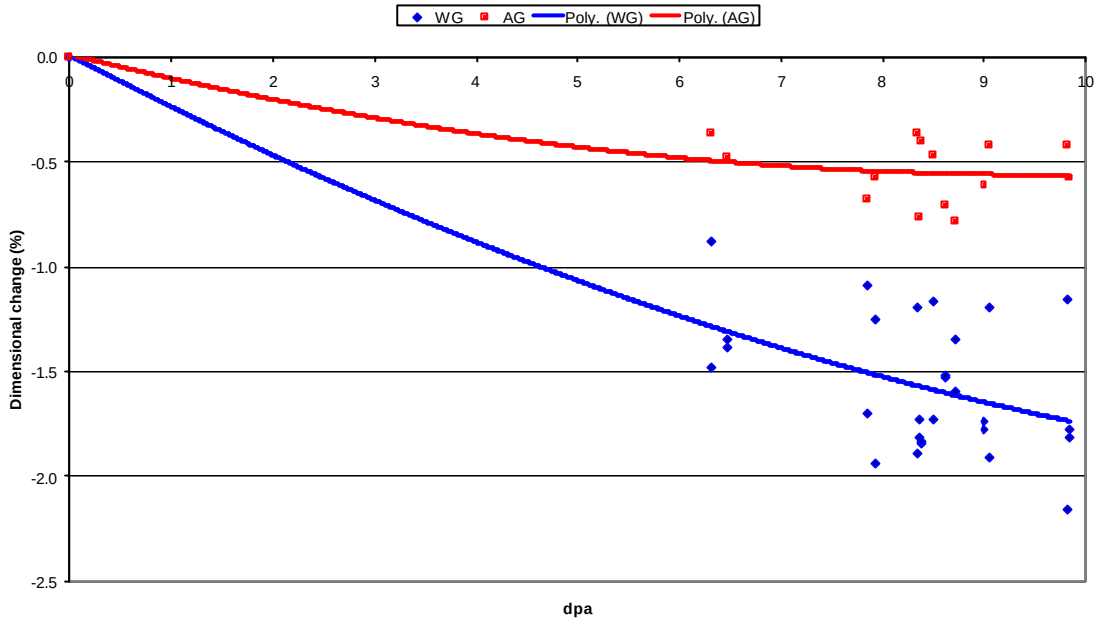


Figure 24 Volume change behaviour for NBG-25 (WG & AG)

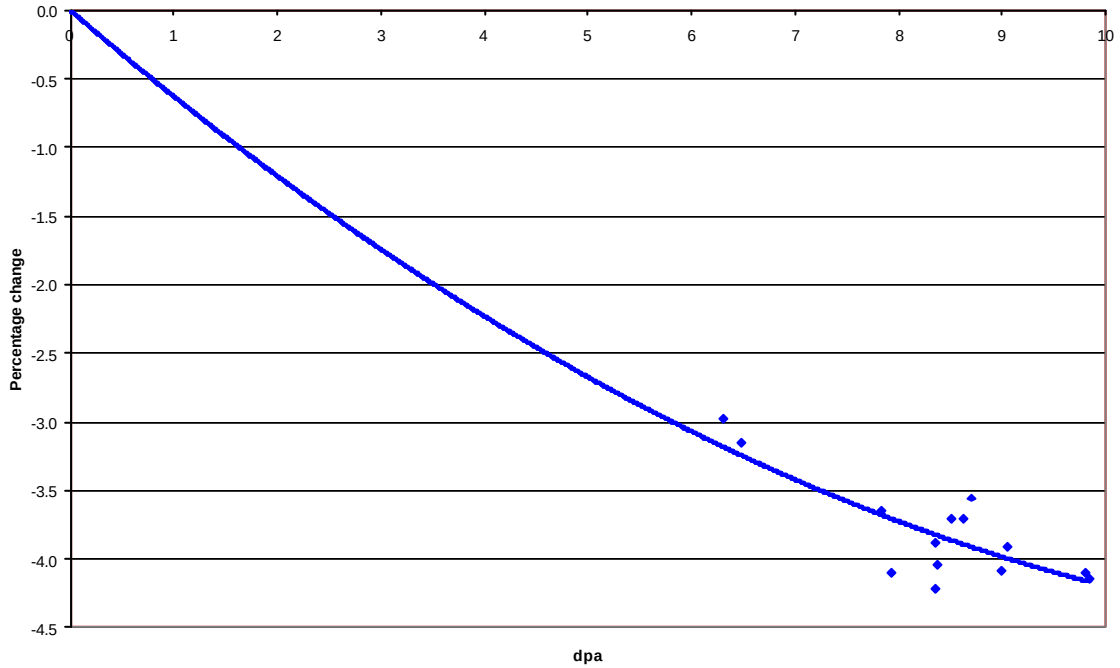


Figure 25 Dimensional change behaviour for PCEA (WG & AG)

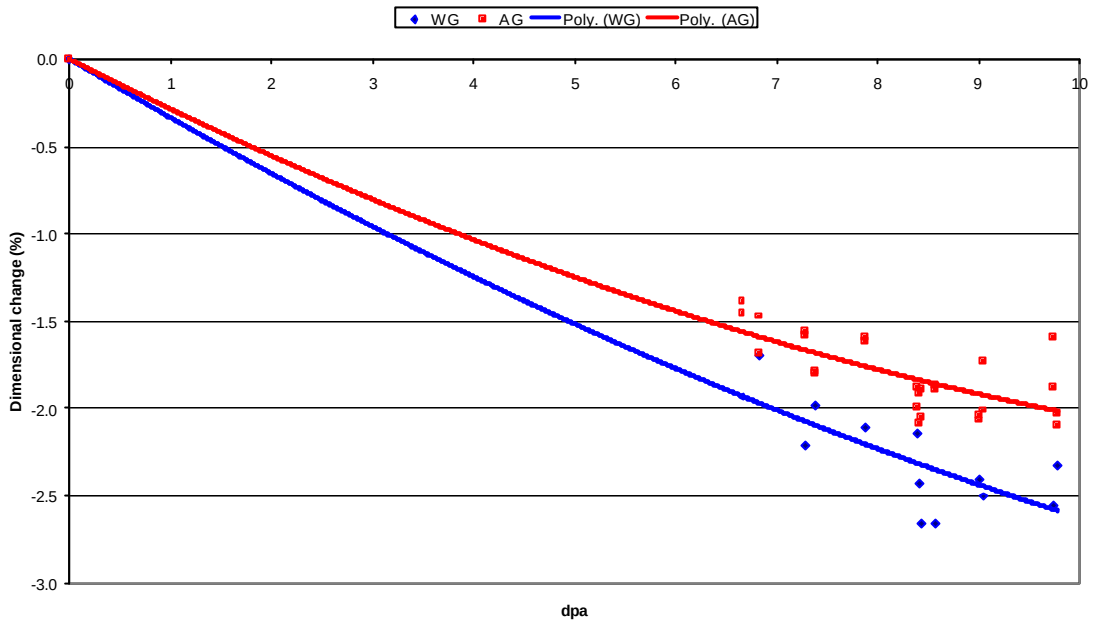


Figure 26 Volume change behaviour for PCEA (WG & AG)

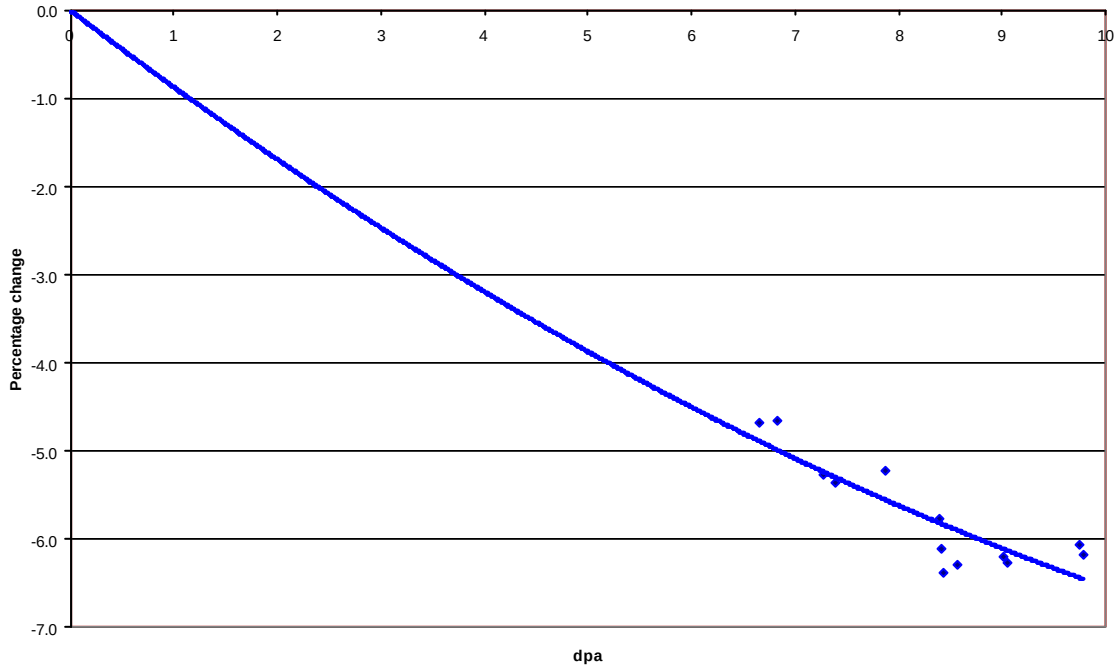


Figure 27 Dimensional change behaviour for PPEA (WG & AG)

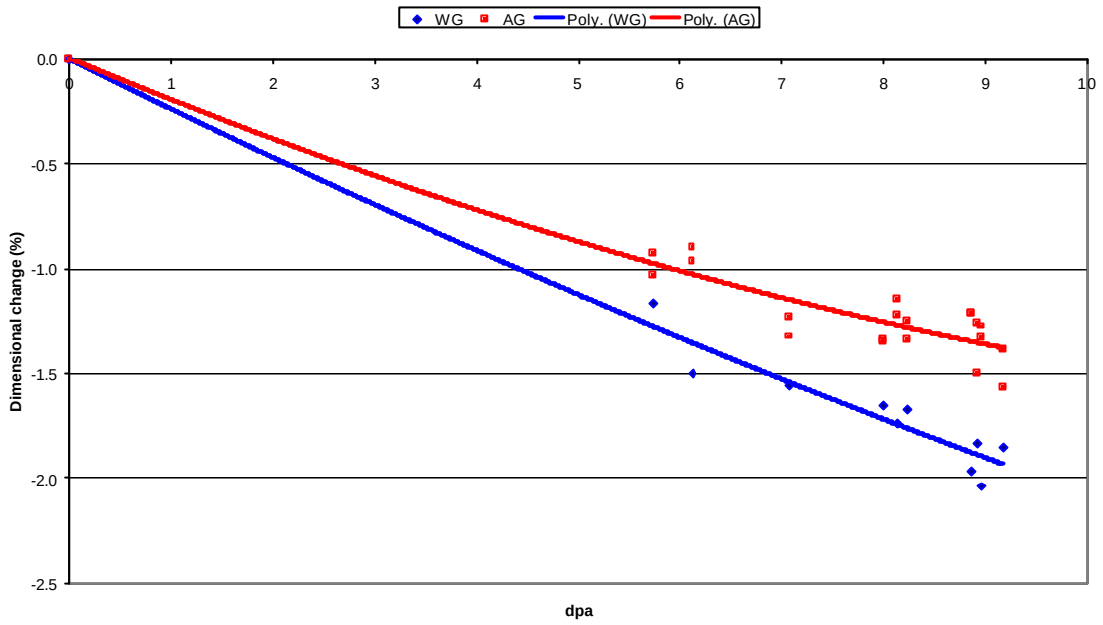


Figure 28 Volume change behaviour for PPEA (WG & AG)

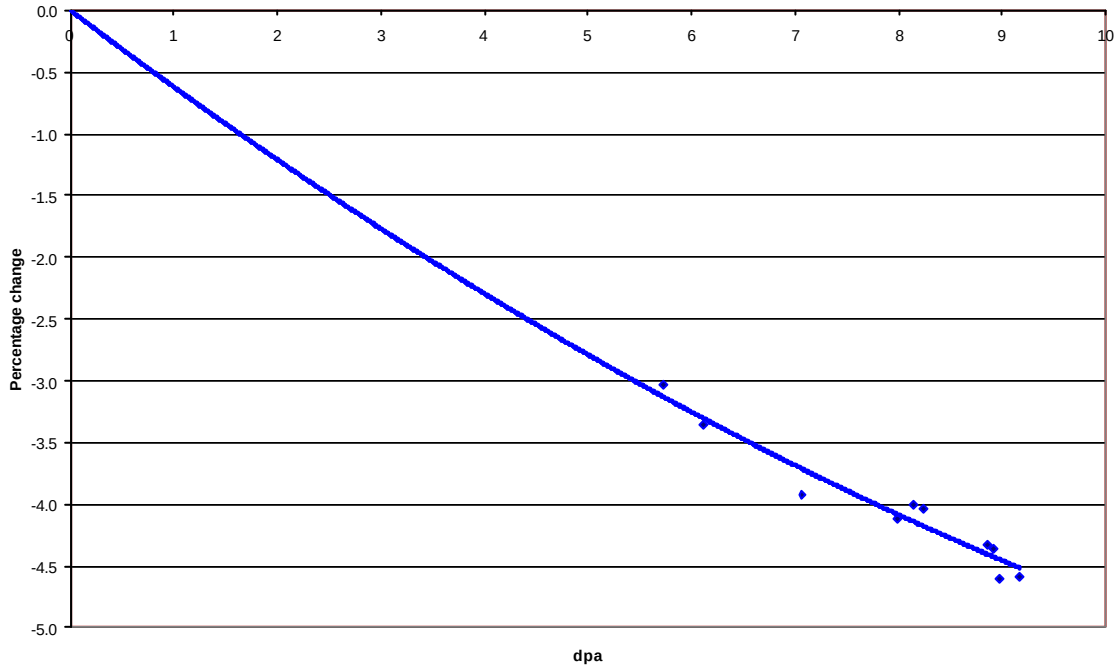


Figure 29 Dimensional change behaviour for PCIB (WG & AG)

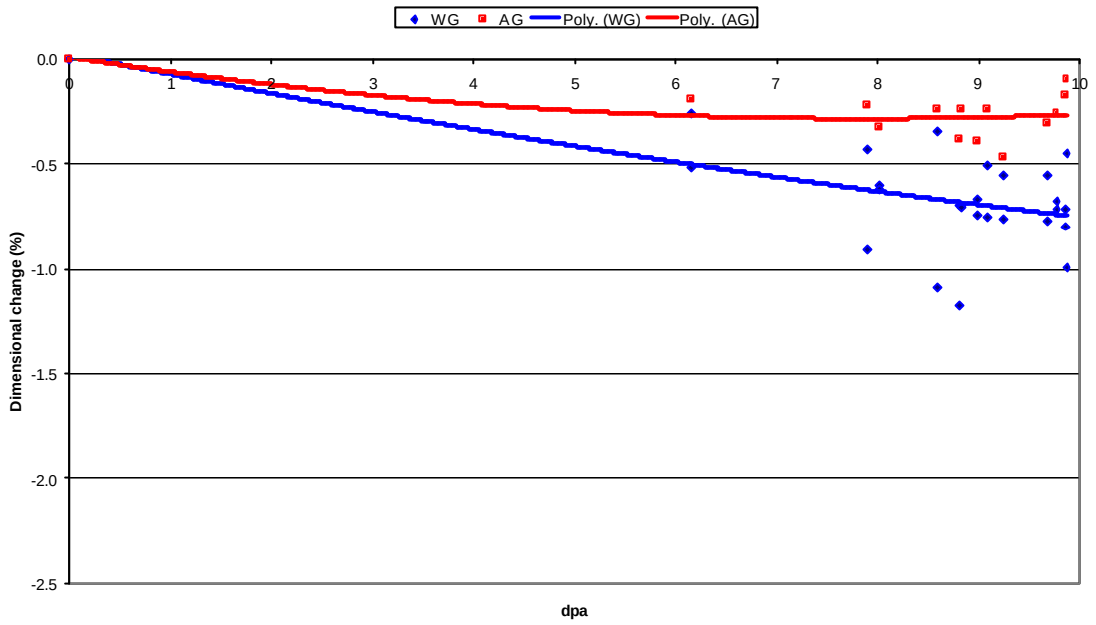


Figure 30 Volume change behaviour for PCIB (WG & AG)

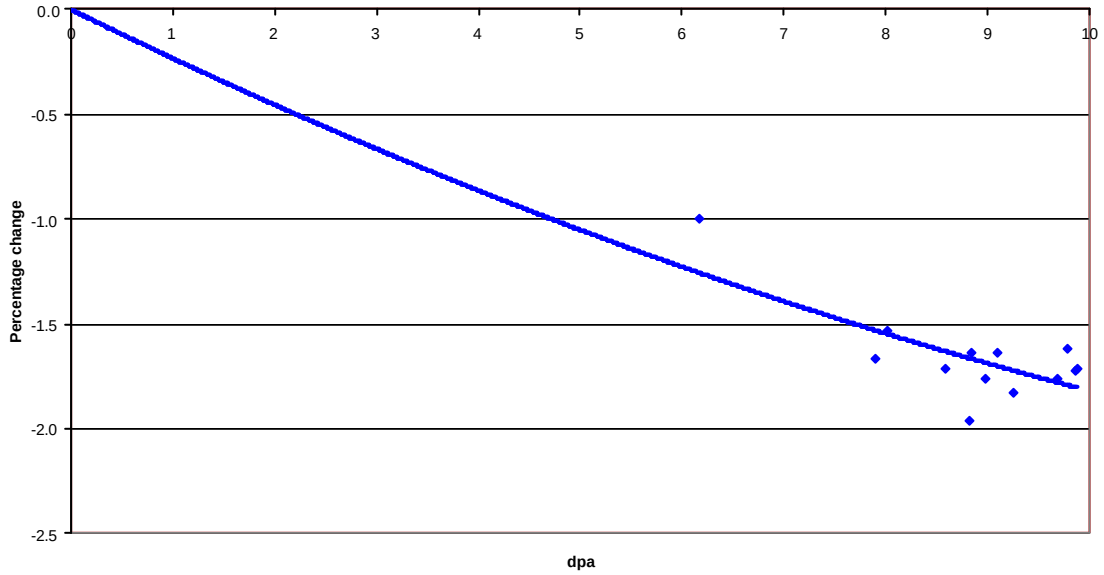


Figure 31 Dimensional change behaviour for IG-110 (WG & AG)

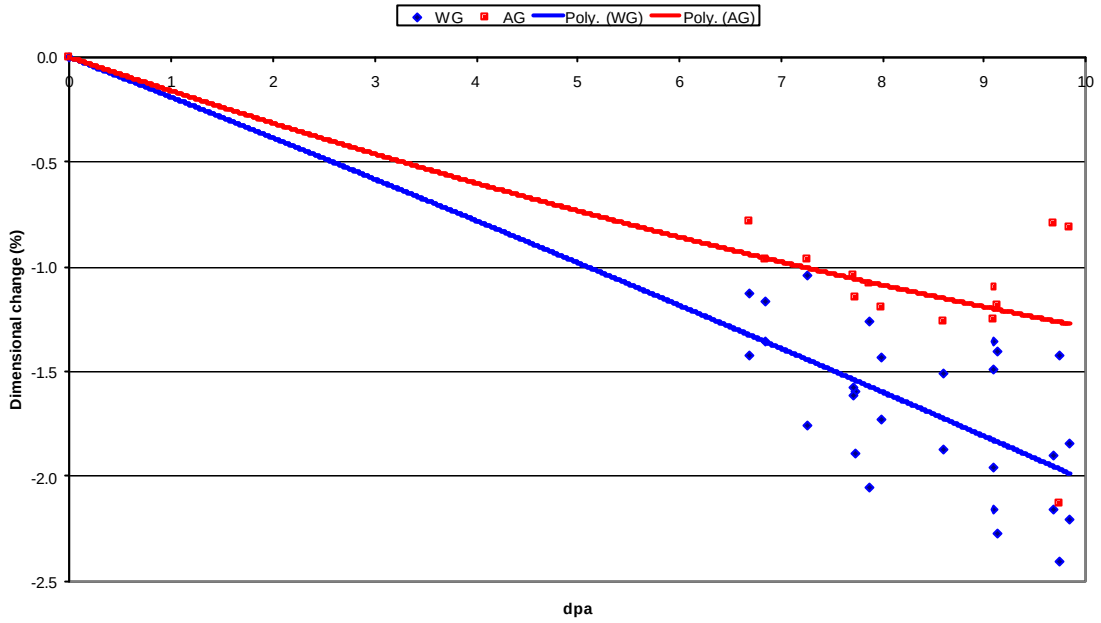


Figure 32 Volume change behaviour for IG-110 (WG & AG)

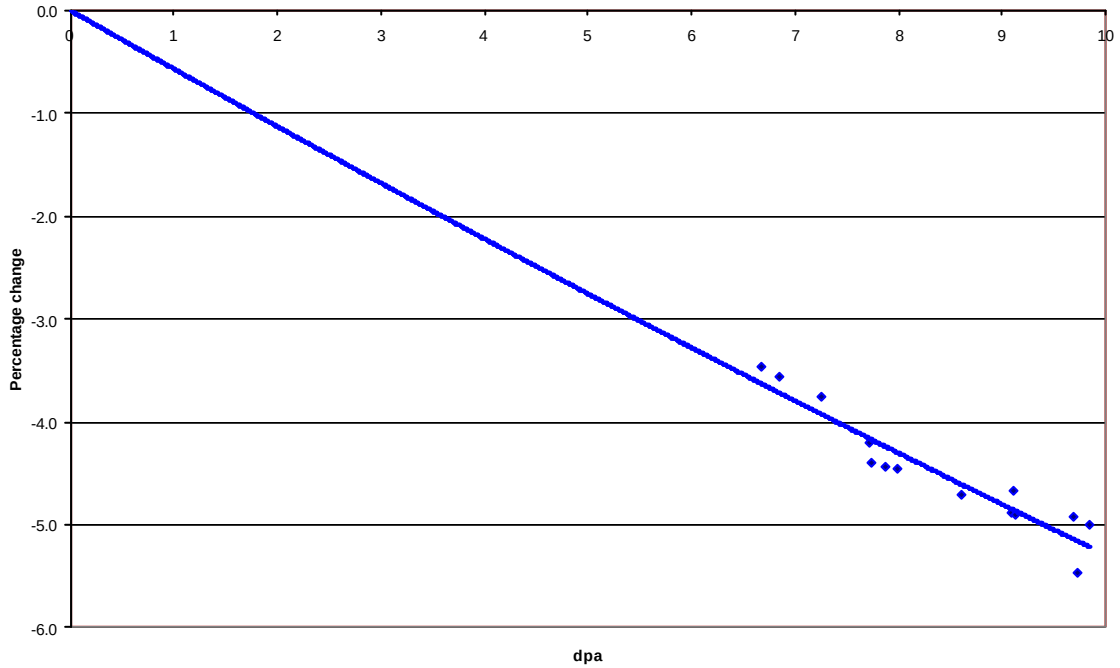


Figure 33 Dimensional change behaviour for IG-430 (WG & AG)

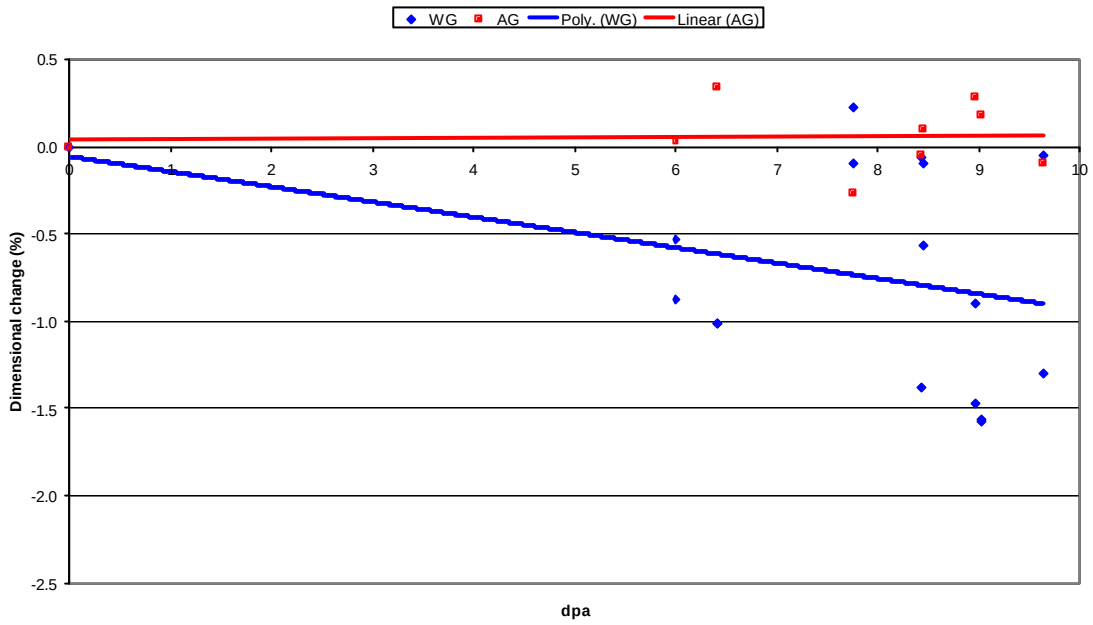


Figure 34 Volume change behaviour for IG-430 (WG & AG)

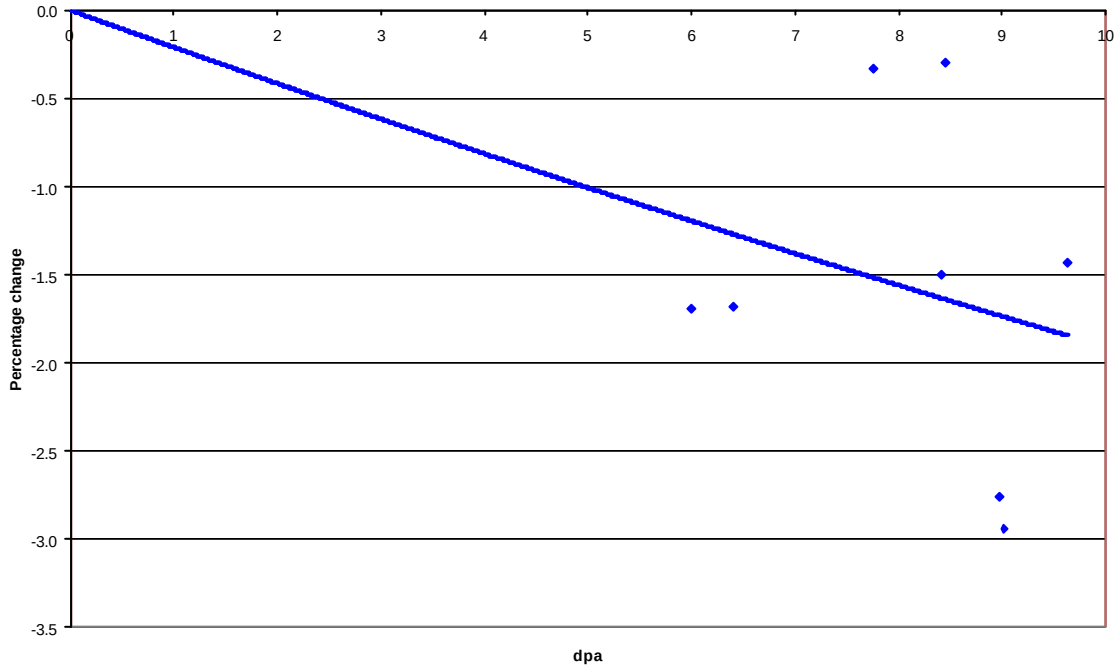


Figure 35 Change in DYM for NBG-10

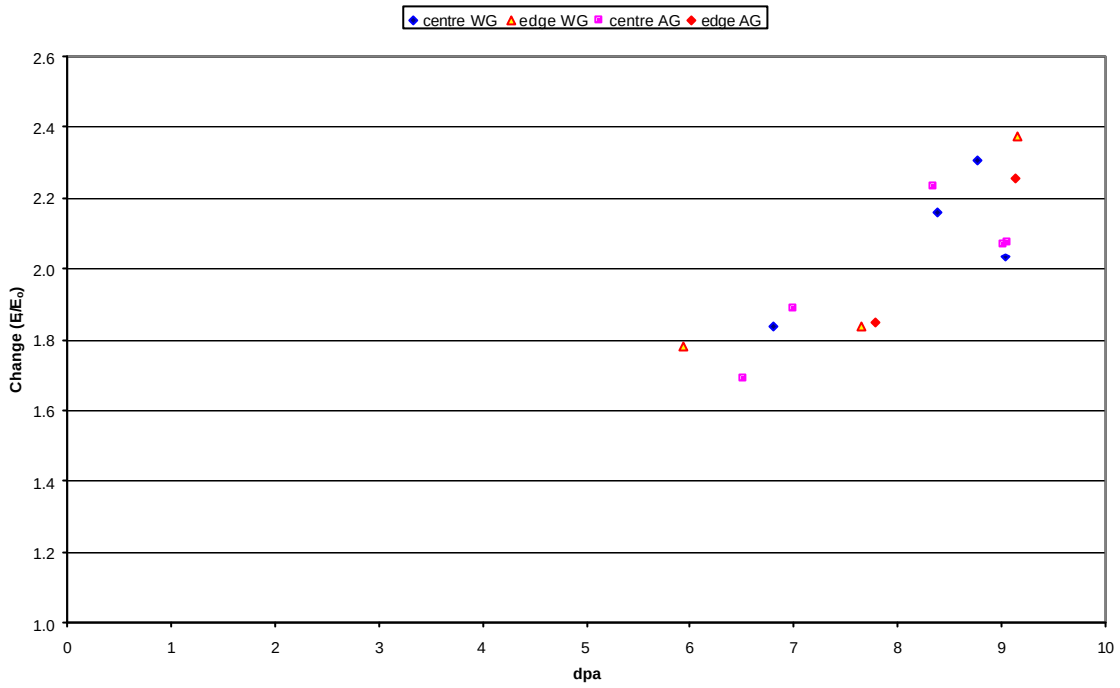


Figure 36 Change in DYM for NBG-20

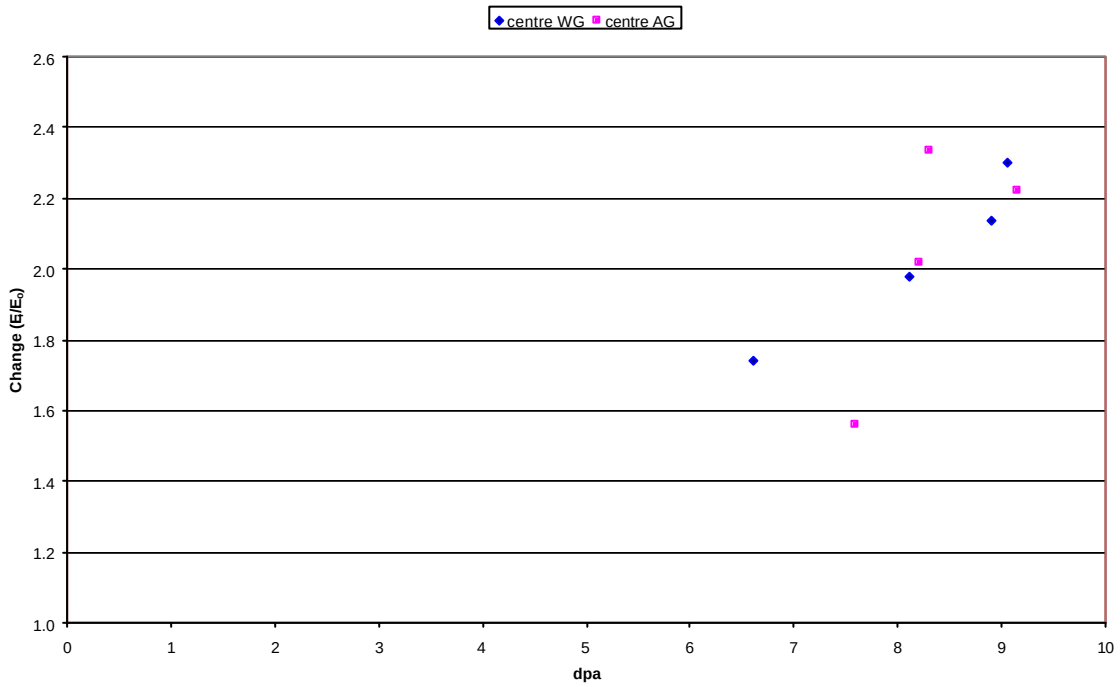


Figure 37 Change in DYM for NBG-25

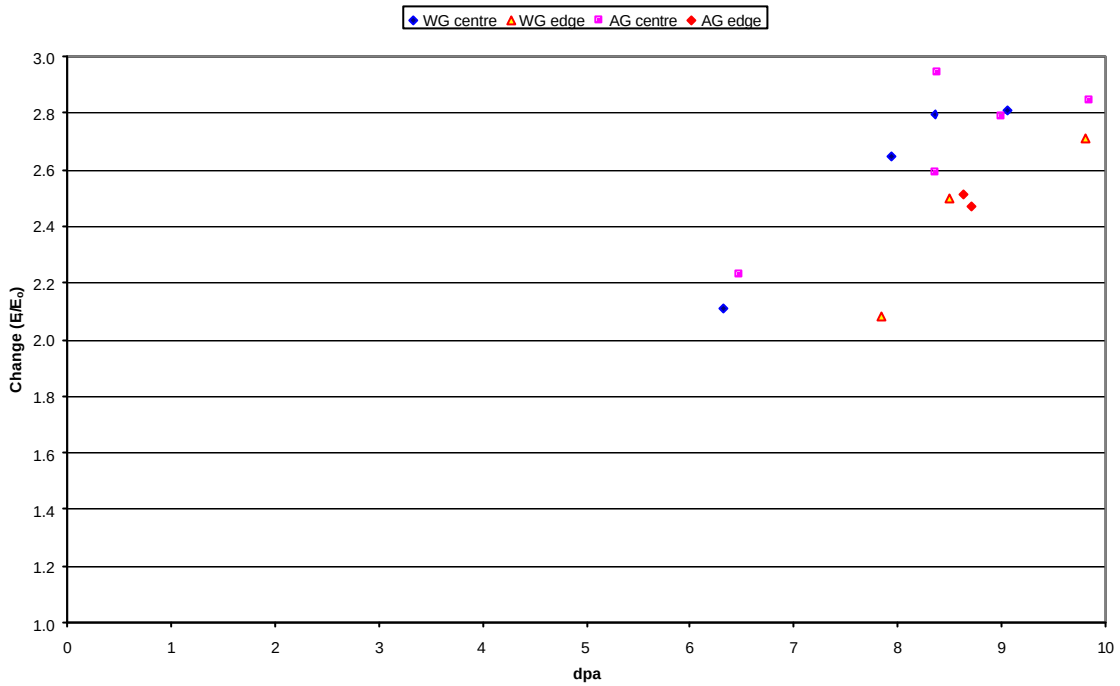


Figure 38 Change in DYM for PCEA

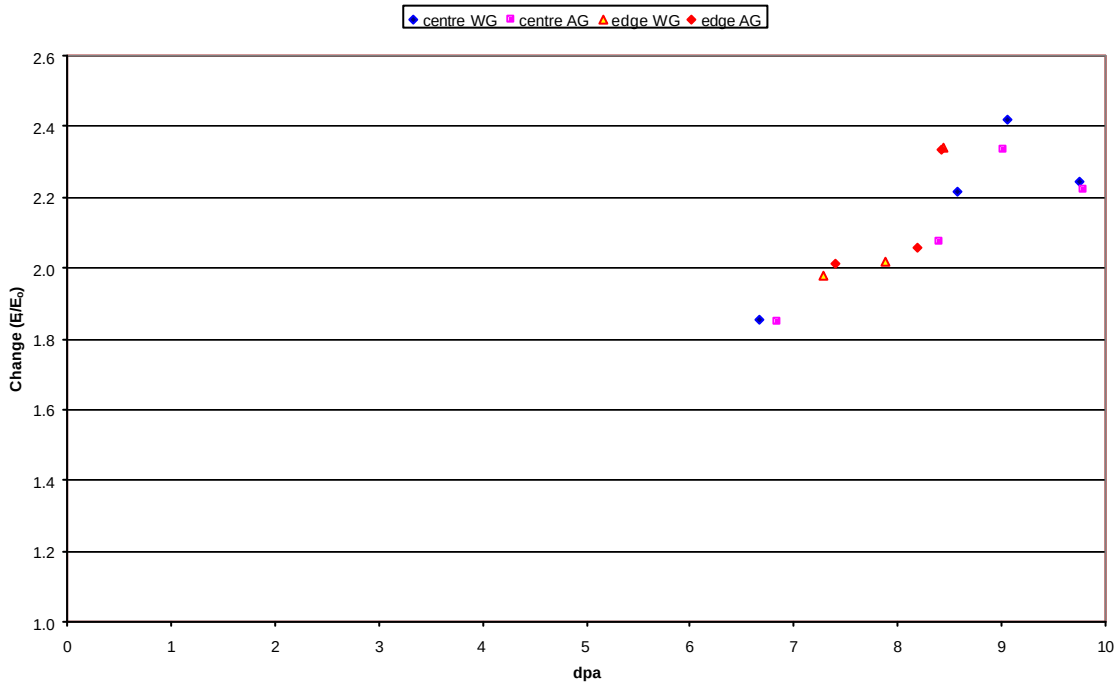


Figure 39 Change in DYM for PPEA

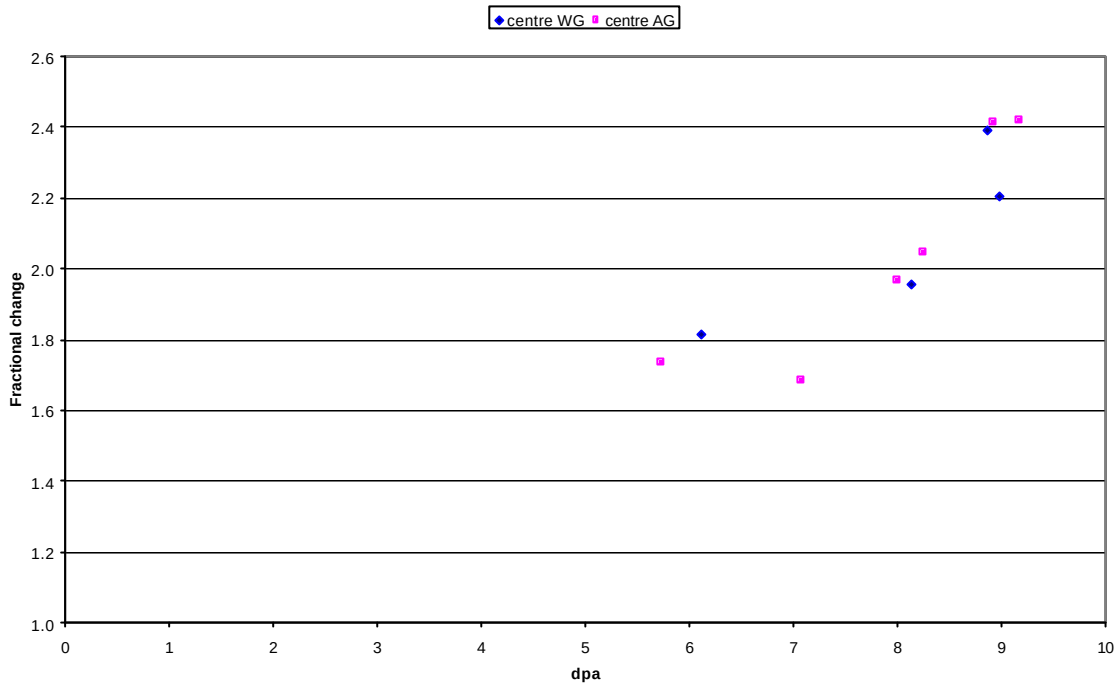


Figure 40 Change in DYM for PCIB

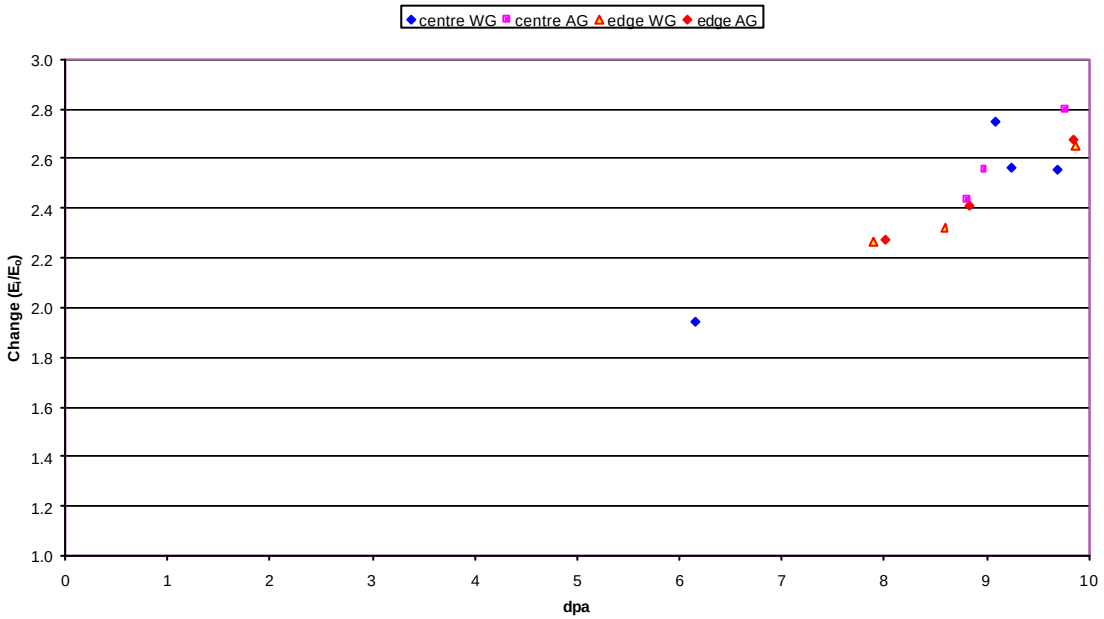


Figure 41 Change in DYM for IG-110

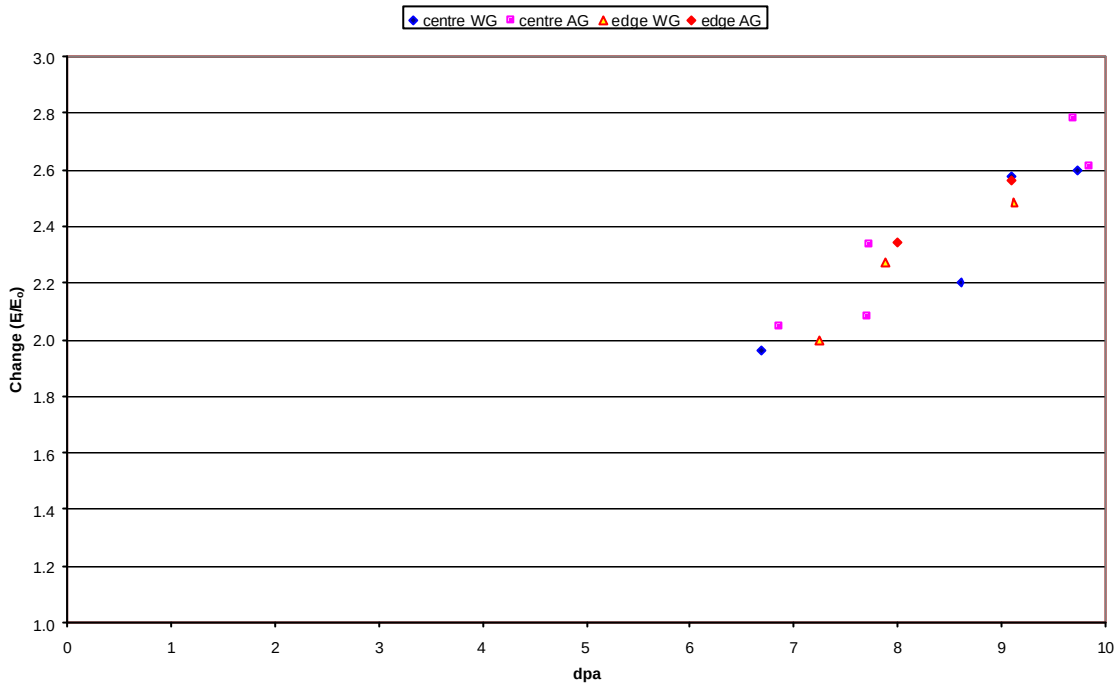


Figure 42 Change in DYM for IG-430

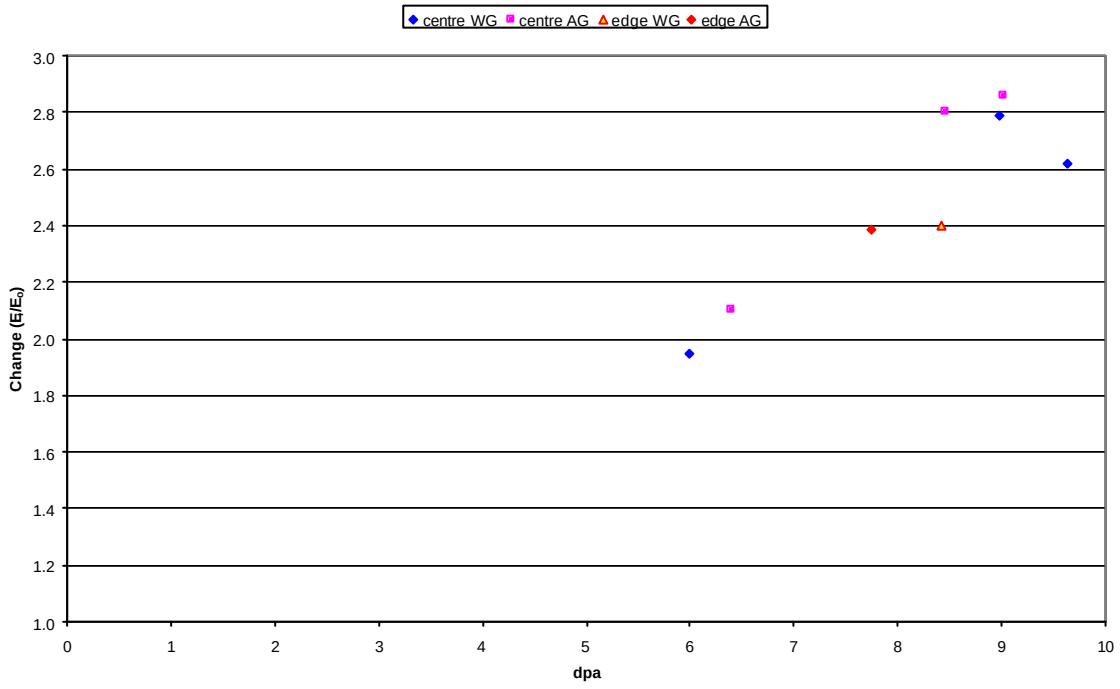


Figure 43 Comparison of dimensional change behaviour of NBG-10 with ATR-2E

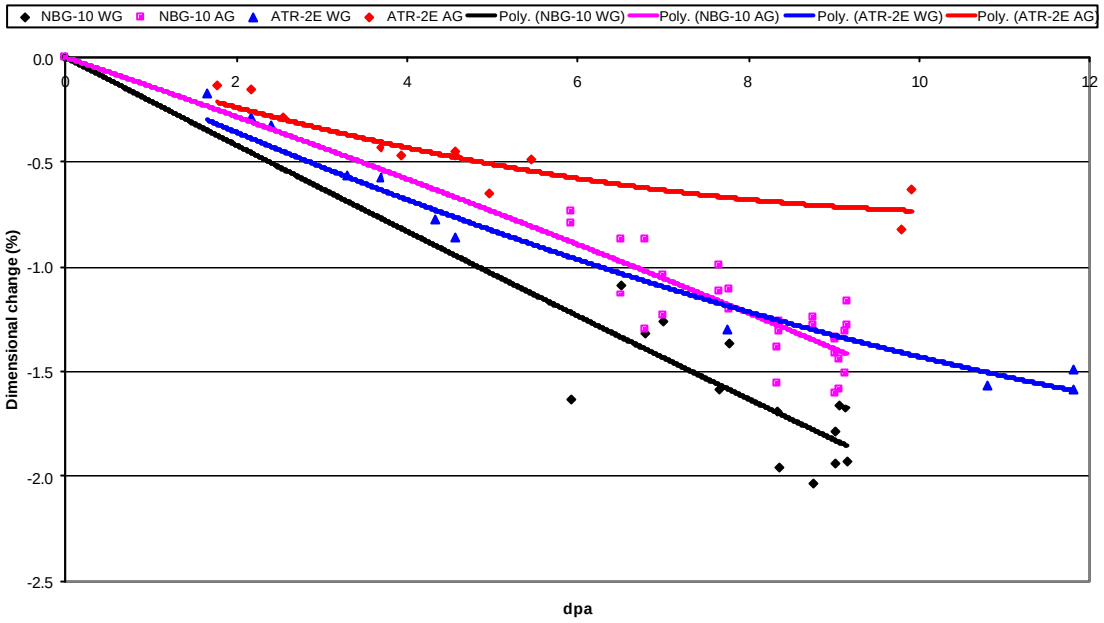


Figure 44 Comparison of dimensional change behaviour of PPEA with ATR-2E

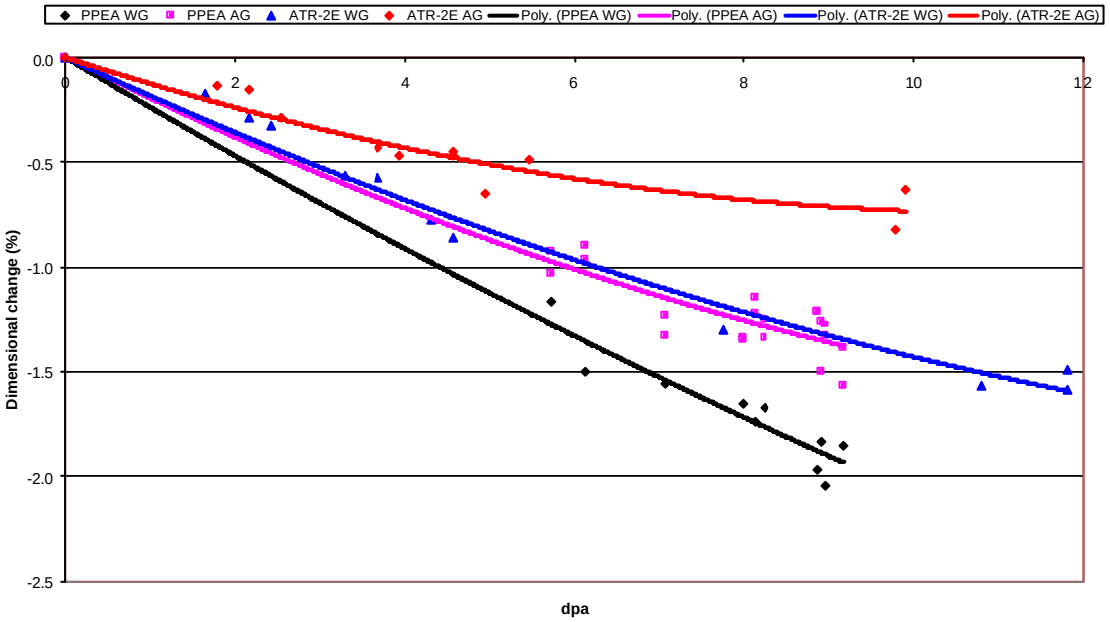


Figure 45 Comparison of changes in DYM for NBG-10 with ATR-2E

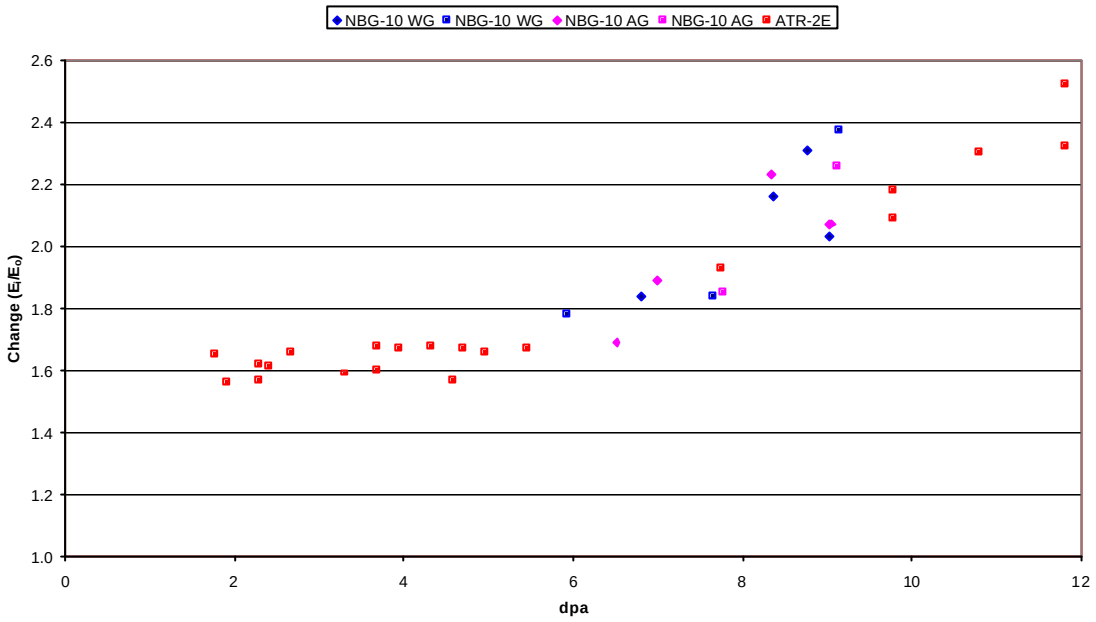


Figure 46 Comparison of changes in DYM for PPEA with ATR-2E