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Coated Particle Fuel Control

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TECHNICAL NOTE

Coated Particle Fuel Control

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DEPARTEMENT D'ETUDES DES COMBUSTIBLES

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Abstract: The HTR-F1 Shared Cost Action sets out to regain past knowledge HTR- type fuel, by accumulating as much past data as possible, initiating new irradiation tests on existing fuel elements, developing new irradiation behaviour code, and re-establishing a fabrication capacity in Europe. Work package 4 (WP4) is concerned with this later aspect.
This document aims to provide a state-of-the-art review of the characterisation methods for quality control that can be applied to the fabrication of fuel particles. The review will particularly focus on quality control methods used by countries involved in R&D in the field of high temperature reactors (HTR).

The first part of the document reviews the main characteristics of HTR fuel, particularly the fuel specifications that clearly identify the fuel parameters to be characterised. The methodology used to carry out this state-of-the-art review is then described: the characterisation methods (classified by parameter) existing in Japan, the USA, China, Germany and France are mainly examined.

Mots Clés	HTR – fuel – characterization – quality control

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1 INTRODUCTION

Within the 5th Framework programme (FP5) of the European Commission, the retention, reestablishment and development of HTR related key technology and innovation potential has been supported in a number of Shared Cost Action programmes. The HTR-F&F1 programme concentrates on the fuel for this type of reactor, and includes workpackages (WP's) on recuperation of past experience, irradiation testing of fuels still available from previous programmes, modelling of fuel performance, and finally re-establishment of fuel manufacturing technology including bibliography work on quality control of coated particle fuel.

In this later scope, the present document aims to provide a state-of-the-art review of the characterisation methods for quality control that can be applied to the fabrication of fuel particles. The review will particularly focus on quality control methods used by countries involved in R&D in the field of high temperature reactors (HTR).

The first part of the document reviews the main characteristics of HTR fuel, particularly the fuel specifications that clearly identify the fuel parameters to be characterised. The methodology used to carry out this state-of-the-art review is then described: the characterisation methods (classified by parameter) existing in Japan, the USA, China, Germany and France are mainly examined.

2 HTR FUEL PARTICLE CHARACTERISTICS

The provisional specifications for the HTR fuel particle were defined by AREVA NP in a document [GUI2001] that was produced within the scope of the 5th EC Framework Programme for the HTR-F project. This document made it possible to identify the main parameters characterising HTR fuel and thus provide guidelines for laboratory-scale fabrication.

The HTR particle is defined according to Table 1[GUI2001].

Parameter	Preliminary specifications
UO2 Kernel	
Composition – O/U ratio	$1.99 \leq x \leq 2.02$
^{235}U Enrichment	$19.65 \leq$ Enr. $U^{235} \leq 19.75$
Mean diameter (µm)	500 ± 40
Density ($g.cm^{-3}$)	≥ 10.4
Sphericity (D_{max}/D_{min})	<1.1
Impurity content	In compliance with ASTM C776
Water content	In compliance with ASTM C776
Boron-equivalent content	In compliance with ASTM C776
Particle	
Buffer layer thickness (µm)	95 ± 20
IPyC and OPyC layer thickness (µm)	40 ± 10
SiC layer thickness (µm)	35 ± 7
Density of buffer layer ($g.cm^{-3}$)	≤ 1.05
Density of IPyC and OPyC layers ($g.cm^{-3}$)	$1.80 \leq x \leq 1.95$

Density of SiC layer ($g.cm^{-3}$)	≥ 3.18
Anisotropy of IPyC and OPyC layers (BAF)	≤ 1.06
Fraction of heavy metal contamination (U_{cont}/U_{total})	$\leq 10^{-7}$
Fraction of defective particles (U_{libre}/U_{total})	$\leq 5.10^{-6}$

Table 1: preliminary specifications as defined in [GUI2001]

In this review of methods, the literature search focuses on parameters that are typically related to the intrinsic characteristics of the HTR particle, which are:

- Kernel diameter and sphericity,
- Kernel density,
- Layer thickness,
- Layer density,
- Anisotropy of dense pyrocarbon layers,
- Heavy metal contamination fraction,
- Defective particle fraction.

3 LITERATURE SEARCH METHOD

The literature search was carried out according to four methods:

- Search using the SIMBAD tool developed by the *Direction de l'Information Scientifique et Technique* (DIST) at Cadarache,
- Computer search on the internet, using key-words on international databases (SINTER Base, OSTI, etc),
- Collection of information on the HTR-F&F1 project or on laboratory visits (particularly the ORNL visit in February 2005) within the scope of international conferences.

SIMBAD (*Système d'Interrogation MultiBase et d'Aide à la Découverte*) is a Multibase query system tool designed for bibliographic data processing and analysis support. It contains a specific HTR database that is regularly updated by the DIST/Cadarache.

SINTER (**S**ustainable & **I**nnovative **N**uclear **T**echnology **E**volution - **R**&D Network) is a European database that mainly serves as a communication system for European R&D programmes initiated as part of the 5th Framework Programme, such as the HTR-F&F1 project. In this way, it was possible to obtain German documents from the 70s and 80s.

OSTI (Office of Scientific and Technical Information) is an American body whose mission is to promote science and technological innovations in close collaboration with the Department of Energy (DOE). Through this organisation (www.osti.gov), a certain number of American documents at least 15 years old were thus accessible, as were very some very recent documents.

It must be pointed out that no South African documents (PBMR Project) could be collected.

Among all the documents collected, the following are worth highlighting:

- Document [DEL1977] describes the status of characterisation methods for the quality control of pyrocarbon, established on an international scale in 1977.
- Documents [BON1974, DEL1976] are state-of-the-art reviews of the different characterisation methods (not only for quality control but also from a thermomechanical or microstructural viewpoint) that were developed and mastered in Germany at the end of the 70s for pyrocarbon and silicon carbide.

- The [HUN2003] slides are a good illustration of the developments underway at ORNL, and are shown in Appendix 1.
- Document [KOB1992] describes the quality control methods used in Japan.

4 MEAN DIAMETER AND SPHERICITY OF KERNEL OR PARTICLE

Knowledge of the mean kernel diameter (and the distribution of the mean diameter per manufacturing batch) is needed to certify the "nuclear design" of HTR fuel [GON1990], [SAW2001], [PET2002]. The two main criteria associated with the mean diameter vis-à-vis the nuclear design are the fissile uranium load (taking into account density) and the internal pressure of the coated particle. The smaller mean diameter (defined in specifications) will condition the minimum acceptable uranium load, whereas the bigger mean diameter (also defined in specifications) will determine the maximum internal pressure of the particle (taking into account the physical characteristics of the deposited layers, especially the buffer).

The kernel sphericity is related to the performance of the HTR particle under irradiation [HEI1985], [GON1990], [SAW2001], [XFU2004]. It is an established fact that the fissile kernel's degree of sphericity directly conditions the state of the mechanical stresses in the subsequently deposited layers, and in particular that of silicon carbide [SAW1999]. In other words, the kernel sphericity is measured and its upper bound value is specified so that the probability of SiC failure (and therefore fracture of HTR particles) is reduced and that the performance of the HTR particle is guaranteed under irradiation.

The different methods used to measure the diameter and sphericity of the kernel (or particle in general) are summarised by country in Table 2. In most cases, the same method is used to determine both parameters.

Among all the means that have been studied and developed to measure the kernel (or particle) diameter and sphericity, two non-destructive characterisation methods particularly stand out and are common to each country. This can be explained by the resolution, precision and degree of automatism (and therefore the measuring time) that is possible in using these methods. These two methods are both based on image analysis but with different image sources:

- Analysis of optical images via the diascopic illumination (transmitted light) of kernels or particles using a microscope [HUN2003a, HUN2004, PRI2004] or macroscope possibly equipped with a PC-controled stage [CHA2002] for automatic imaging. This method consists in depositing kernels on a clear support under white light, acquiring optical images via a microscope and a digital camera, and then processing them using image analysis tools to obtain the kernel diameter size and sphericity. Appendix 1 shows the equipment and methods used.

- Image analysis by passing particles in front of a light beam (laser). A first device was developed in Austria at the Seibersdorf Research Centre [WAL1977] both for R&D and production purposes in Germany in the 70s and 80s. The particles were transferred to the light source-detector via a pneumatic conveyor. Approximately 50 diameters per particle were measured by a photosensitive diode. At the time [KOI1976, WAL1977], the Austrian device had a measurement error of ± 2 µm (on particle diameters between 100 and 1500 µm) at a rate of 100 particles per second. The signal processing was improved in this device, which is still marketed by Seibersdorf RC under the name PSaDA – Particle Size and Diameter Analyser – and was bought by the Japanese [KOB1992] and most likely by the South Africans (PBMR). A photo of this tool is provided in Appendix 2. The Japanese

specify that they have the same particles analysed several times in order to better estimate maximum and minimum diameters.

A recent article published in 2005 [KAR2005] mentions a device (see Figure 1) operating on the same principle as the previously-mentioned device. It is being developed at ORNL and is rivalling in some ways the device mentioned in [PRI2004, HUN2004]. The "TRISO Particle Counting And Sizing Tool" (TP-CAST) is composed of an optical system that projects a laser through a target cell and collects the laser transmitted onto a photoelectric cell. This cell converts the analog signal into digital data in real time. This device is currently being developed (see Appendix 3) and the first characterisations published by ORNL were carried out using the first technique [HUN2004], i.e.: image analysis using a microscope with diascopic illumination. In the long run and on the condition that TP-CAST meets accuracy and measuring time expectations (they hope to reach over 200 particles per second), this device may prove to be very useful for industrial control.

It must be noted that the laser beam particle-size analyser (dry mode) only allows the measurement of the mean particle diameter based on the hypothesis that these particles are spherical. According to Fraunhofer's theory valid for particles of over 2 µm, the laser beam particle-size analyser is based on the interpretation of diffraction-diffusion figures created by the objects to be measured in the presence of an incident laser beam. The diameter resulting from this interpretation is that of a sphere that has the same volume as the particle to be measured. Thus the laser beam particle-size analyser does not make it possible to reach a sphericity value. Moreover, it is impossible to obtain diameter unit values (per particle), and estimating uncertainty on the value of the measured diameter proves difficult, given the very principle of the measurement.

	Japan [KOB1992, KIT1996, XFU2002]	**China** [TAN2000, XFU2002]	**USA**	**Germany**	**France**
Diameter	1. Automatic particle-size analyser 2. X-ray image analysis 3. Image analysis of metallographic cross sections	1. X-ray image analysis 2. Image analysis of metallographic cross sections	1. Image analysis by diascopic illumination (particles observed via transmitted light) (see Appendix 1) [HUN2003a&b, HUN2004, PRI2004, SAU2003, WIC1992] 2. Image analysis with pneumatic conveying of particles in front of a laser beam (see Appendix 1) [HUN2003a&b, KAR2005, MAC1977]	Automatic particle-size analyser developed in Austria at the Seibersdorf Research Centre [WAL1977]. This device was sold to the Japanese (mentioned in [KOB1992]) and most likely to the South African PBMR. This device is known as the PSaDA (Particle Size and Diameter Analyser).	1. X-ray image analysis 2. V-slot technique (around a hundred kernels are aligned in a V-shaped groove and the total length is measured and then divided by the number of kernels) **DRAGON era** --- Image analysis by diascopic illumination (particles observed through transmitted light) [CHA2002] **Currently being developed at the CEA**
Sphericity	As above and sphericity is defined by max diameter/min Diameter ratio	Image analysis (no additional information)	As above and sphericity is defined by max radius/min radius ratio min [PRI2004]	As above and sphericity is defined by max diameter/min diameter ratio	As above and sphericity is defined by max diameter/min diameter ratio

Table 2: Kernel diameter and sphericity measurement methods used in China, Germany, Japan, USA and France

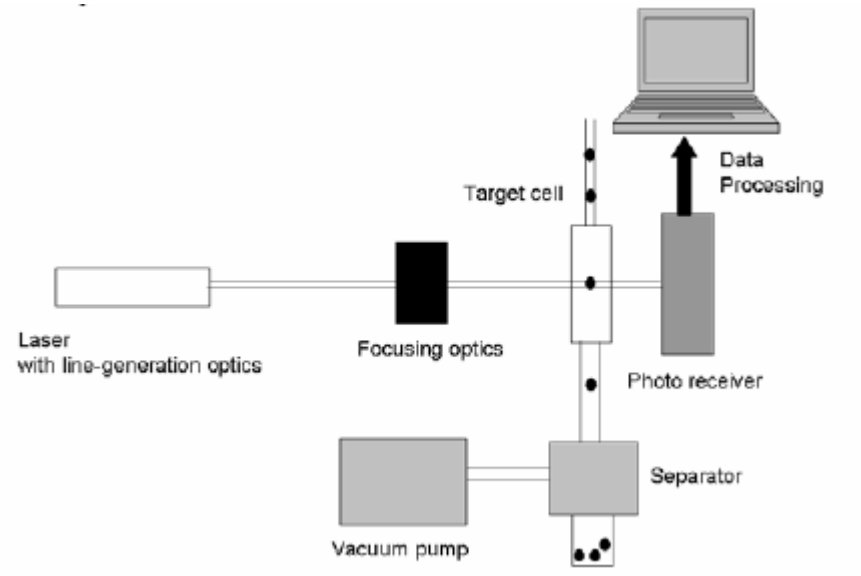

Figure 1: Schematic diagram of the TP-CAST principle under development at ORNL [KAR2005]

5 KERNEL DENSITY

The density (and diameter) of the fissile kernel of an HTR fuel particle is essential in determining the uranium load: this is a “nuclear design” criterion.

Table 3 reviews the kernel density measurement methods used in Germany, China, France, Japan and the USA.

The principle common to these countries for determining UO_2 kernel densities (or UCO in the USA) is based on measuring the mass by weighing a sample that is representative of a batch of kernels, before measuring the volume of this sample using one of the following three methods: Hg porosimetry [HUN2003a&b, KOB1992, XFU2002], He pycnometry [HUN2003, SAU2003] or image analysis [WAL1977]. Imagine analysis involves measuring the kernel diameter and then calculating the overall sample volume based on the assumption that the kernels are symmetric. The density measured by He pycnometry only takes into account the closed porosity (with gaseous helium taking into account open porosity). Hg porosimetry can be used to quantify the geometrical density at low mercury pressure (in this case, open and closed porosities are taken into account), whereas a density comparable to that measured by He pycnometry is obtained at maximum mercury pressure (200 to 400 MPa depending on the device) seeing that mercury intrusions occurs in the open pores. In the present case where the UO_2 kernels are manufactured according to the sol-gel routes and do not show any (or hardly any) open porosity, all three methods must be comparable in terms of measurement. However, He pycnometry would appear to be the most suitable method as it is non-destructive and does not generate waste, contrary to Hg porosimetry. This method is more sensitive than image analysis.

	Japan	China	USA	Germany	France
Kernel density	Volume measured by Hg porosimetry and mass measured by weighing [KOB1992]	Volume measured by Hg porosimetry and mass measured by weighing [TAN2000, XFU2002]	Volume measured by Hg porosimetry or He pycnometry and mass measured by weighing [HUN2003a&b, SAU2003, WIC1992]	1. Volume measured by image analysis or by the V-groove technique and mass measured by weighing [WAL1977]. 2. Volume measured by Hg porosimetry and mass measured by weighing. [KOI1976]	1. Volume measured by Hg porosimetry and mass measured by weighing. 2. Volume measured by image analysis or by the V-groove technique and mass measured by weighing **DRAGON era**

Table 3: Kernel density measuring methods used in China, Germany, France, Japan and the USA

6 LAYER DENSITY

The layer density is required for the following main reasons, all of them related to a performance objective under irradiation:

- The buffer density is related to its thickness and determines the "buffer" volume available to hold gas fission products and to handle the internal pressure generated in the particle,
- The IPyC and OPyC density – specified with a lower bound value – ensures low permeability, limiting the transport of chlorine to the kernel and capable of interacting during the deposit of SiC, whereas the upper bound value determines the adequate creep behaviour (direct influence on the stresses occurring in the layer under irradiation),
- The SiC density, specified with a lower bound value, ensures the minimum leaktightness in this layer.

Table 4 evaluates the methods for measuring the density of the different layers which are used in Germany, China, France, Japan and the USA.

Measuring buffer density

The buffer density is measured by determining the ratio between the buffer mass (estimated by calculating the difference between the "kernel + buffer" mass and the "kernel" mass) and the volume occupied by the buffer (estimated by calculating the difference between the "kernel + buffer" mass and the "kernel" mass).

ρ (buffer) = (mass (particle)-mass (kernel))/(volume (particle)-volume (kernel))

The mass is weighed before and after the buffer deposit. The volume is measured either indirectly by image analysis based on the diameter [WAL1977], or by Hg porosimetry based on the first stage of mercury intrusion at low pressure when the mercury has filled the inter-particle space [KOB1992, HUN2003a, HUN2004].

On the one hand, this density is geometric (both open and closed porosities are taken into account) and on the other hand, it is easier to measure the density of all layers composing the HTR particle using this method than it is using the flotation method, which is certainly more sensitive but much more difficult to use (long sample preparation).

For example, the Japanese use the geometrical method (Hg porosimetry) to obtain the OPyC density by measuring the volume before and after combustion of this layer. For layers with a geometrical density greater than 1.5, both methods (geometric and flotation) produce an equivalent result, as shown in Figure 2 from [DEL1977]. This figure compares the densities of more or less dense buffer layers and PyC ex-propylene layers, measured both by flotation (sink-float) and by geometrical means (PSaDA) (see § 4). Concerning the flotation method, impregnation of the flotation liquid in the considerable open porosities existing in the buffer fragments causes the density to increase, thereby creating a non-systematic difference between the flotation and geometrical density values.

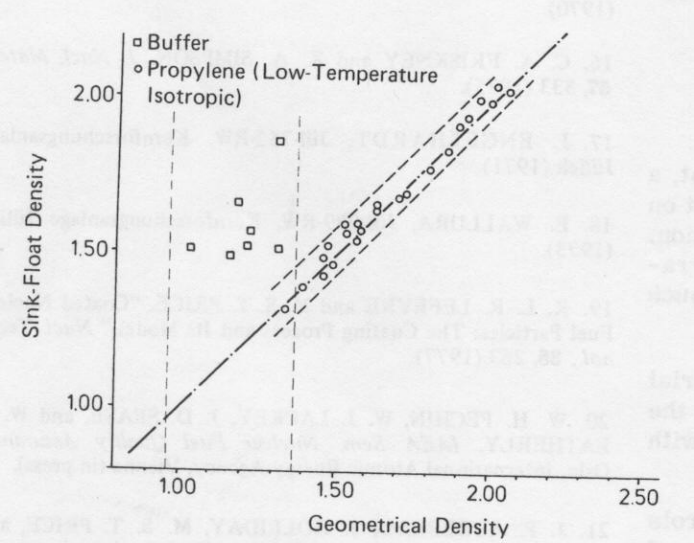

Fig. 2. Correlation between geometrical and sink-float density.

Figure 2: Correlation between geometrical densities and flotation densities [DEL1977]

The reference method for OPyC, SiC and IPyC layers is the so-called flotation method, which implements a density gradient column. This method uses a thermostated glass column containing a mixture of two liquids whose respective densities bound the density of the sample to be measured. The column must have a density gradient that is uniform down the entire length, with density decreasing from the bottom to the top. Glass beads of precisely known densities (standard beads) are used to map the column density (calibration curve) and to check the gradient linearity. Appendix 4 provides the most frequently used binary mixtures and a description of the standard floats.

Measurements are carried out by introducing layer fragments into the flotation column. These will sink to a point of equilibrium corresponding to the level where the mixture of liquids and the sample are identical in density.

This measurement method is among the most accurate, but it is also very costly in preparation time and thus in manpower.

Preparing the samples for measuring density by flotation measurement [KOB1992]:

For IPyC or even OPyC layers, micro-fragment samples are obtained by polishing in the following manner: the particles are glued to a grooved sample-holder. The depth of the groove corresponds to the thickness of the layers whose densities are to be determined (for IPyC, this refers to particles prior to SiC deposition). Large-grain polishing is then carried out until the particles no longer protrude from the groove. The sample-holder is placed in a beaker of hot ethyl alcohol to dissolve the glue and retrieve the layer fragments. This methodology cannot be used with buffers due to excessive porosity rendering the layer too fragile (see results in Figure 2). The fragments of pyrocarbon layers then undergo vacuum degassing before being immersed in the liquid used for flotation measurements.

Concerning the SiC layer, the fragments are isolated by oxidising the layers that have been crushed and detached from the fissile kernel (combustion of the pyrocarbon). Oxidation occurs at 850°C in air or pure oxygen.

The only innovative method remarked during this review, and which differs radically from the present reference method (sink-float), is the measurement of x-ray attenuation by 3D X-ray computed tomography, currently being developed in the USA (ORNL).

	Japan [KOB1992]	**China** [TAN2000, XFU2002]	**USA** [HUN2003], [SAU2003] et annexe 1	**Germany**	**France**
Buffer density Estimated by differential measurement of the volume and mass.	Volume measured by Hg porosimetry (kernel only and then kernel + buffer) mass measured by weighing (kernel only and then kernel + buffer) NB Measurements are performed on a supposedly representative number of particles.		Volume measured by Hg porosimetry (kernel only and then kernel + buffer) and mass measured by weighing (kernel only and then kernel + buffer) NB Measurements are performed on a supposedly representative number of particles.	Volume measured by Hg porosimetry (kernel only and then kernel + buffer) or by the PDaSA device Mass measured by weighing (kernel only and then kernel + buffer) [WAL1977] NB Measurements are performed on a supposedly representative number of particles.	Volume and mass measured by weighing ((kernel only and then kernel + buffer) **DRAGON era** Volume measured by laser beam particle-size analyser (kernel only and then kernel + buffer) or by image analysis Mass measured by weighing (kernel only and then kernel + buffer)
IPyC density	Use of a flotation column operating with a density gradient liquid. Liquid used: tetrabromoethylene	Use of a flotation column	Use of a flotation column operating with a density gradient liquid. Liquid used: mixture of tetrachloroethylene and dibromoethylène	Use of a flotation column operating with a density gradient liquid. Liquid used: mixture of isobutanol and bromoform [BON1974]	Use of a flotation column operating with a density gradient liquid. Liquid used: mixture of ethyl iodide and toluene or xylene
SiC density	Use of a flotation column operating with a density gradient liquid. Liquid used: methylene iodide - benzene	Use of a flotation column	Use of a flotation column operating with a density gradient liquid. Liquid used: bromoform and methylene iodide	Use of a flotation column operating with a density gradient liquid. Liquid used: bromoform and methylene iodide	Use of a flotation column operating with a controlled-density liquid. Liquid used: mixture of methylene iodide and toluene or xylene
OPyC density	Volume measured by Hg	Use of a flotation column	Use of a flotation column	Use of a flotation column	Use of a flotation column

	porosimetry (full particle then particle after combustion (thus up to the SiC)) Mass measured by weighing (full particle then particle after combustion)		operating with a density gradient liquid. Liquid used: tetrachloroethylene and dibromoethylène	operating with a density gradient liquid. Liquid used: mixture of isobutanol and bromoform [BON1974]	operating with a controlled-density liquid. Liquid used: mixture of ethyl iodide and toluene or xylene
Density of all layers			Upstream R&D on measuring x-ray attenuation by 3D X-ray tomography		

Table 4: Layer density measurement methods used in China, Germany, France, Japan and the USA

7 LAYER THICKNESS

A considerable number of more or less developed methods for measuring layer thicknesses have been recorded in the literature. A distinction will be made between the 1) methods that are currently (or in the short-term) operational and therefore used in each country concerned by the HTR fuel topic, and 2) methods that are more innovative and require further development.

The technique based on the image analysis of metallographic cross sections using an optical microscope (opaque projection) belongs to the first category. This requires having metallographically prepared the sample beforehand. After coating the HTR particles in a resin, this preparation consists in polishing them to obtain the most equatorial plane possible whilst respecting a given methodology. The relevance of this method is to obtain the highest level of automatic image processing in order to decrease the cost of manpower and minimise operator effects. Such processing techniques are developed by ORNL [PRI2004] and the CEA [CHA2002].

X-ray image analysis is a variation of the previous method, which is briefly mentioned by each country in question without it being possible to obtain more specific information apart from the work carried out within the scope of the 5th FP in Petten [ACO2002].

Among the innovative techniques under development, there is the use of eddy currents [HOC2004] and 3D X-ray tomography illustrated in Appendix 1 from [HUN2003], which is under development at ORNL. These two methods represent a real technological leap in quality control on an industrial scale, on condition that they are associated with an ad hoc particle conveyor device for real automatic measurement.

Table 5 summarises the methods for measuring layer thicknesses that are used in Germany, China, France, Japan and the USA.

	Japan [KOB1992], [KIT1996], [XFU2002]	**China**	**USA**	**Germany**	**France**
Layer thickness	1. Automatic measuring device developed in Austria at the Seibersdorf RC. The measurements are to be carried out on particles between each deposit. 2. X-ray image analysis 3. Image analysis of metallographic cross sections	Measurement based on X-ray images [XFU2002]	1. Image analysis (opaque projection) of metallographic cross sections (particles observed in reflected light) (see Appendix 1) [HUN2003a&b, HUN2004, PRI2004] 2. X-ray or 3D X-ray tomographic image analysis under development (see Appendix 1) [HOC2004, HUN2003b, RON2003, SAU2003] 3. Eddy currents under development [HOC2004]	1. Microscopic measurement after metallographic polishing or on radiographs 2. Automatic particle measurement device developed in Austria at the Seibersdorf RC. The measurements are to be carried out on particles between each deposit. [DEL1977]	Manual measurement on x-rays or micrographs (polished cross sections). Measurements at the time were not automatic and therefore were very expensive in terms of manpower. **DRAGON era** Image analysis (opaque projection) of metallographic cross sections (particles observed in reflected light). **Under development at the CEA**

Table 5: Layer thickness measurement methods used in China, Germany, France, Japan and the USA

8 FREE-URANIUM CONTAMINATION AND FRACTION OF DEFECTIVE PARTICLES (in defective SiC layers)

The release of fission products (FP) during irradiation may have several causes vis-à-vis the fabrication of coated particles and fuel elements (compacts in our case). FP releases may be due to:

- Particles with defective SiC coatings,
- Free-uranium contamination in the OPyC layer,
- Uranium contamination in the graphite of the compact matrix.

Among these potential sources of FP releases, the number of particles with non-leaktight SiC layers (broken, porous or cracked) must be estimated as accurately as possible before irradiation as it was shown in the cases of completed HTR fuel fabrications [GON1990, IAE1997] that uranium was the predominant source term for FP releases. This is why it is crucial to know the fraction of defective particles[1] directly resulting from fabrication before irradiation, in order to assess the quality of these particles (direct effect on fuel acceptance for irradiation in a reactor) and to subsequently quantify fuel behaviour under irradiation by a post-irradiation measurement of the fraction of broken particles.

The Germans [HEI1985, HUS1977] showed that particles with defective SiC layers were mainly generated during the cold semi-isostatic pressing of pebbles, during direct particle-particle contacts or with particles that are not very spherical. This is why a selective sorting phase using a vibration machine and a fuel particle coating phase were developed before final compacting.

Uranium contamination in the OPyC layer is a result of the chemical vapour deposition (CVD) process, when particles come into contact with the inner walls of the fluidised bed furnace, which are subject to pollution by the UO_2 kernels. Uranium contamination of the graphite composing the matrix mainly comes from the residual impurities in the graphite itself. The total fraction of U contamination (OPyC and compact graphite) is usually referred to as “exposed uranium fraction” or “tramp uranium” by the Americans and “free uranium fraction” by the Japanese, Chinese and Germans.

The countries involved in a programme designed to qualify HTR fuel are implementing a reference method relying on the same measurement principle (see Table 6) that is based on the (leach)-burn-leach test [IAE1997].

This test is based on the accessibility of gases or liquids to reach uranium[2], existing as contamination in the graphite (or even OPyC) of the compact or the intentionally “exposed” kernels. This is done by oxidising the carbonated materials (matrix, porous and dense PyC) of particles with defective SiC layers. The fraction of defective particles is then quantified by the amount of uranium (or U-Th) leached by a gas or a reactive liquid.

Another method, which is non-destructive, consists in subjecting particles or compacts to short-term irradiation – referred to as “weak irradiation” – and analysing the short-lived fission gases released as a result of fission recoil. This method was used in the past in the USSR [IAE1997] (this reference only mentions the use of weak irradiation in the USSR without giving any more details) and in France by the Grenoble CEA, leading to the registration of a patent. This method is briefly described in paragraph 8.2.

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[1] In certain documents of American origin, particularly [MYE1989], a semantic distinction makes it possible to differentiate between post-fabrication broken particles and post-irradiation broken particles:

- Defective particles resulting from particle and/or compact fabrication processes (cracks, porosities, missing buffer, etc.) are referred to as “defective coated fuel particles”;
- Particles damaged during irradiation, which therefore release an abnormally high quantity of FP are referred to as “failed coated fuel particles”.

[2] Or uranium-thorium in the case of $(U,Th)O_2$ fuels

8.1 Leach-Burn-Leach Test Method

The general procedure for this leach-burn-leach test method consists in leaching the compact (or coated fuel particle) in an acid solution, before subjecting it to thermal oxidation and final leaching. During the first leaching, the “exposed” kernels and the heavy metal contamination (uranium and possibly thorium) are extracted. The oxidising combustion phase makes it possible to burn the carbonated materials (compact matrix, OPyC and IPyC-buffer for the defective SiC layer particles). During the second leaching, the fuel kernels of the defective particles in the SiC layer are dissolved by the acid.

The relative assay of the uranium extracted (or U-Th) at the end of the first leaching makes it possible to quantify the heavy metal contamination (assuming that no HTR particles are broken), whereas the relative assay of the uranium (or U-Th) extracted at the end of the second leaching quantifies the fraction of defective particles (damaged or porous SiC).

	Japan	Chine	USA	Germany	France
Contamination	Electrochemical disintegration of the fuel compact in an HNO3 solution brought to boiling point, then filtered. The filtered uranium is then assayed by either the "arsenazo III" method or by fluorometric analysis Contamination is defined according to the ratio of the quantity of uranium on the filter to the quantity of uranium in the compact [KOB1992, SAW2001, XFU2002]	Nitric acid leaching (acid leaching). The Chinese are following the same approach as that developed by the Germans. [XFU2002] No details on their procedure.	Leach test [MYE1989, SAU2003, WIC1992]: procedure detailed in the document	HNO3 acid leach test for particle contamination [IAE1997, MYE1989]]	HNO3 acid leach test for particle contamination
Failed SiC particles: the contamination and fraction of failed/macroporous SiC layer particles are accumulated	Combustion in air at 850°C for about 20 hours (to enable oxidation of UO2 in U3O8). The combustion residue is then dissolved in boiling HNO3. The uranium present is then assayed by the same methods as those used for contamination. The fraction of failed SiC particles is defined by the ratio of the quantity of uranium in the failed particles in the SiC layer to the total quantity of uranium in the compact.	Combustion (burn) then nitric acid leaching (leach). [XFU2002] No further details (as above)	Burn-leach test [MYE1989, SAU2003, WIC1992]: procedure detailed in the document Possible use of Hg porosimetry after combustion of the OPyC layers (burn) at 750-850°C, followed by radiographic measurement to visualise the possible presence of Hg in the SiC layer [HUN2003a]	Burn-leach test [IAE1997, MYE1989] procedure detailed in the document	Combustion (burn) then nitric acid leaching (leach). --- Weak irradiation test [CHE1974]

	[KOB1992, SAW2001, XFU2002]				

Table 6: Methods for the measurement of contamination and the fraction of defective particles used in China, Germany, France, Japan and the USA

Detailed procedures of leach-burn-leach tests on compacts were only found in reference [MYE1989]. This document specifies the operating procedures implemented by the GA laboratories specialised in quality control and HTR fuel production (San Diego, USA), by US national laboratories, and by the HOBEG production laboratory (German HTR fuel fabrication plant, Hanau).

GA procedure:

The two leach phases are identical, apart from the fact that the compacts are dried overnight at 80°C (353K) after the first leaching and before combustion.

The burn-leach test is carried out according to the nine following steps:

1. 15 to 20 compacts are positioned vertically at the bottom of a 600 ml quartz beaker.
2. The beaker and compacts are placed in a muffle furnace of an internal volume of about 280 dm^3.
3. An air atmosphere at 0.17 MPa and 27°C (300K) flows through the furnace at a rate of 2.7 dm^3/min maintained at a temperature of 750°C (1023K). The furnace atmosphere is thus completely renewed every 1h and 45 minutes.
4. After 50 to 60 hours, the particles are x-rayed to detect any unburned OPyC layers. If this is the case, combustion is continued.
5. The particles after combustion (free of OPyC and matrix graphite) are placed in an Erlenmeyer with 100 ml taken from a stock solution of leaching acid (leach solution) made from 87.7%vol of concentrated HNO_3, 0.1%vol of HF and 12.2%vol of H_2O.
6. The Erlenmeyer and its contents are heated to 109°C (382K), and the liquid is maintained in a reflux for 24 h.
7. The liquid is then decanted (and kept); 100 ml of the leach solution is added and the liquid is maintained in a reflux for a further 24 h.
8. The decanted liquids are evaporated until solid residues are obtained, which are then dissolved in 250 ml of 1N solution of HNO_3.
9. This last solution is measured by spectrophotometry at 578 nm for uranium assaying and at 550 nm for thorium assaying, using bromo-PADAP as a complexing agent for uranium and thorine as a complexing agent for thorium.

NUKEM-HOBEG procedure:

The burn-leach test is carried out according to the following three steps in an ultra-clean laboratory. The procedure is less detailed:

1. The spherical elements (pebbles) are oxidised at 750°C for about 100 h. It is indicated that a period of about 65 hours is needed to burn the carbonated materials outside the SiC layer; the burning time is increased to ensure complete combustion of the carbon. The temperature of 750°C was chosen to avoid oxidisation of SiC.
2. The particles after combustion are placed in a vessel containing HNO_3 6N nitric acid, in a reflux at a temperature of 95°C for 16 h.
3. The uranium in the leach solution is then detected spectrophotometrically using Arzenazo III as an indicator.

Moreover, a recent publication by ORNL [KER2005] refers to the combustion of compacts in a nitric medium inside a microwave oven, known as “microwave digestion”, which leads to an improvement in the leach-burn-leach test process.

By way of conclusion on the burn-leach test, the limits of this method as reported in the summary document [MYE1989] are briefly reviewed:

- The leach rate depends directly on the size of the hole or defect in the SiC layer and thus on the leaching time. For example, for UCO and ThO2 particles, 82% of the uranium and only 9% of the thorium initially present was extracted and assayed after 48 hours of leaching of laser-drilled particles (hole size: 22 µm). Document [MYE1989] gives no information on the leach rate of UO2 kernel particles. However, the leach rate of UCO and ThO2 kernel particles does not appear to follow a linear rule vis-à-vis the defect size. Below a lower-bound critical size (to be determined!), no heavy metal can be attacked by acid in a reasonable time lapse. The limit defect size capable of influencing the gaseous FP release rate remains to be determined.
- The combustion temperature in air must be as low as possible to avoid filling the narrowest defects in the SiC layer with a layer of silica (SiO2). [MYE1989] shows that by dropping from 950°C to 750°C, the defect volume that will be filled in for silicon carbide (see Appendix 5) is divided by at least a factor of 3. For example, at 950°C for 10h in air, a defect of less than 35 nm in SiC will be filled in, whereas at 750°C, all things equal, the defect had to be less than 7 nm to be filled in. Here too, it remains to be defined what minimum defect size in the SiC is to be considered as a failure in terms of gaseous FP releases. The use of Hg porosimetry is also said to help characterise the defect size in SiC.

8.2 Weak irradiation test

The weak irradiation test consists in subjecting particles or compacts to short-term irradiation so that the tested fuel can be manipulated just like a virgin fuel, before analysing the fission gases to estimate the fraction of defective particles.

The only feedback available in the literature is French [CHE1974]. A Russian reference to this process is mentioned in a summary report by IAEA [IAE1997] without any details.

This method is relevant as it tests the SiC layer function, i.e.: the leaktightness to gaseous and metallic fission products. This method is also non-destructive and direct in comparison with the burn-leach test where the failure rate is estimated indirectly by “exposed” uranium assays.

In patent [CHE1974], it is specified that this “weak irradiation” can be carried out in a reactor with a thermal neutron flux (ex: Mélusine or Siloé, former Grenoble test reactors) of 10^{10} to 10^{12} $n_{th}.cm^{-2}.s^{-1}$, the samples (compacts or particles) having being placed in a furnace at 600-800°C. An irradiation time between 15 min and 2 hours is said to be sufficient to obtain a reliable response, with fission gas analysis being carried out by on-line gamma ray spectrometry in a flow of He or in a vacuum for ^{138}Xe or ^{89}Kr, and by accumulation of ^{133}Xe or ^{85}Kr on cold traps. At the time, feasibility studies were carried out on DRAGON compacts.

9 ANISOTROPY OF OPYC AND IPYC LAYERS

The isotropic nature of thick PyC layers is essential for guaranteeing the role of confinement layer against fission products and, above all, for guaranteeing for as long as possible the compression of the SiC

layer sandwiched between the two IPyC and OPyC layers in reactor operations. It should be noted in particular that during irradiation, the layers of thick pyrocarbon are subjected to four major phenomena (densification or anisotropic withdrawal of the material, creep during irradiation, elastic behaviour and variation in the constraints generated during irradiation), which play a decisive role in the thermomechanical behaviour of the particle. These four phenomena are all related to the pyrocarbon's degree of isotropy[3] (or anisotropy) and to its variation during fuel irradiation.

9.1 Pyrocarbon structure

Pyrolytic carbon can have one of three main microstructures (column structure, lamellar structure or isotropic structure), depending on the method of production used. Here, only the last type of pyrocarbon is discussed, which is obtained under HTR particle deposition conditions (deposition temperature around 1300-1400°C, atmospheric pressure).

The basic element is the crystalline lattice of the graphitic carbon (see Figure 3a), where the carbon atoms are distributed over a hexagonal network. These atoms form layers of graphene in a 2D plane. These arrangements are limited to a few hundred atoms. The crystalline pyrocarbon structure can then be considered at first sight to be a packing of a dozen layers of graphene, parallel, but randomly oriented, resulting in the formation of spheroids (see Figure 3b), which are deposited on the substrate to form the PyC layer itself (see Figure 3c).

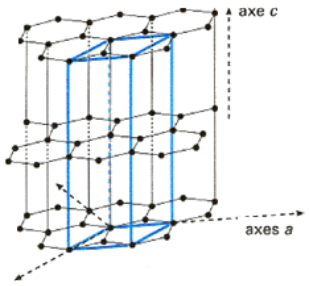

Fig 3a: Hexagonal graphitic network

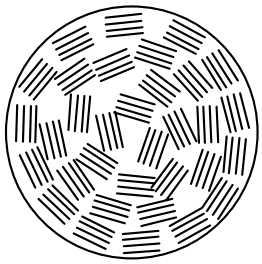

Fig. 3b: Spheroid

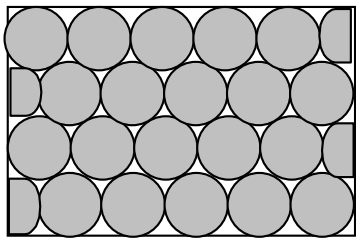

Fig. 3c: Packing of spheroids

9.2 Measuring anisotropy by X-ray diffraction

The first technique used to evaluate isotropy was derived from an X-ray diffraction (XRD) technique developed by Bacon [BAC1956] in the 1950s for blocks of nuclear graphite. This technique makes it possible to define the anisotropic factor known as the BAF (Bacon Anisotropy Factor). To introduce his factor, Bacon hypothesized that, for deposits on planes, the behaviour under irradiation is equivalent to the thermal expansions at 400°C following the two main axes of graphite. The BAF (obtained by XRD measurement) thus means expressing the carbon anisotropy in the form of a relationship, with the heat expansion coefficients parallel and perpendicular to the deposit at 400°C.

Bokros [BOK1965] then suggested that this measurement designed for graphites is adapted to the pyrocarbons deposited on substrate planes.

Measurement of the BAF is based on the technique for measuring textures (known as the pole figures technique). The principle consists in setting the position of the detector in relation to the incidental direction at an angle of 2θ of the 002 carbon planes. The sample is then oriented in every position in space so as to

[3] Anisotropy can be defined as the mean fraction of crystallites meeting certain orientation conditions, and weighted by volume of these crystallites. A material is considered to be anisotropic if all the crystallites composing it show a preferred orientation. On the contrary, a material is qualified as isotropic if all the crystallites show a null average orientation. A material can appear to be globally isotropic yet locally anisotropic, depending on the scale of analysis. This is particularly true of pyrocarbon, showing a tendency for global isotropy on the scale of spheroid packing, and local anisotropy on a smaller scale of analysis.

record the diffracted intensity in accordance with angles ϕ and χ of inclination. In the certified protocols, only direction ϕ is swept for reasons of symmetry.

An experimental curve $I(\phi)$ is thus obtained, making it possible to determine the distribution function $f(\alpha)$ which represents the density of the 002 layers depending on their orientation in relation to the deposition plane. When the distribution remains constant regardless of the value of α, the deposit is then isotropic. The BAF is finally deduced from experimental recording $I(\phi)$ according to the following equation:

$$BAF = 2 \cdot \frac{\int_0^{\pi/2} I(\phi) \cdot \cos^2\phi \cdot \sin\phi \cdot d\phi}{\int_0^{\pi/2} I(\phi) \cdot \sin^3\phi \cdot d\phi}$$

9.3 Measuring anisotropy by optical measurements

In the history of pyrocarbon for HTR particles, the first characterisations were made by XRD with measurement of the BAF on substrate planes before the optical techniques from metallography were developed to measure the layers directly on spherical nuclei. These techniques have gradually taken over in the field of quality control.

These measurements have been developed in a number of different laboratories all over the world (the OAF and SMP methods in GA, and OPTAF in ORNL in the USA [BOM1986, STE1975], the DAR or RAPAX method at the CEA in Grenoble [HOL1972] in France and the OAF method in the Seibersdorf Research Centre [KOS, KOI1974] for the Germans). The common practice has always been to convert the optically-obtained measurements into BAF values. It should be noted that the German equipment developed in Seibersdorf has been purchased by South Africa, China and Japan for current HTR projects as a means of controlling the quality of the PyC anisotropy. The Seibersdorf Centre is still marketing this product.

In short, all the countries involved in an HTR program use a technique based on optical polarimetry for quality control, except the United States, which has developed a method based on the ellipsometry technique described below.

9.4 Measurement of anisotropy by ellipsometry

This method is currently being developed at ORNL. A light beam (from an Hg lamp) is aimed and then polarized by passing through a polarizer before passing through a Quartz crystal that vibrates at 50 Hz (photo-elastic modulator). This then creates an elliptically-polarized light wave that “probes” the polished sample. The reflected light beam then passes through a second Quartz crystal (which vibrates at 60 Hz this time), then a second polarizer before being sent to the detector. Signal processing is then performed and makes it possible to trace back to a certain number of values, such as the diattenuation coefficient which is directly correlated with the degree of anisotropy of PyC.

10 OTHER PARAMETERS AND SAMPLING

The other parameters defined in the preliminary specifications are typically intrinsic to UO_2-type nuclear fuel and are dealt with in this paragraph. The methods for characterising these parameters are

summarised in Table 7, and are common to all the countries involved in the HTR program (China [XFU2002]; Japan [KOB1992]; USA [WIC1992, SAU2003]).

Parameters	Methods
Composition (or the O/U ratio) of the nucleus	Thermogravimetric analysis (TGA)
Enriching in U-235	Mass spectrometry coupled with inductive plasma (ICP-MS)
Impurity content	Atomic Emission Spectrometry coupled with inductive plasma (ICP-AES)
Water content	Cf. ASTM C696 - Coulometry
Equivalent Boron content	Cf. ASTM C696 - Conversion based on impurity analysis

Table 7: Methods for characterising the intrinsic parameters of UO_2 fuel

The sampling aspect is generally not developed in the documents collected. The only interesting document on this subject is reference [XFU2002]. This document compares the design, manufacture and quality control methods between the Japanese HTTR program and the Chinese HTR10 program. The (short) description of the sampling done in both countries is given in the tables in Appendix 6.

11 CONCLUSION

This overview of quality control for manufacturing HTR particles has made it possible to:

- Review the different techniques implemented in the past document archives which are in general more than 20 years old),
- Compare them with those currently in use and operational in China and Japan, where strong, HTR-specific R&D has been underway for more than 20 years (both countries have produced and characterised loads for irradiations in their respective reactors: HTTR in Japan and HTR10 in China),
- Collect American and French documents describing their methods that are under development in most cases. We were not able to obtain any documents related to the subject of this review from South Africa.

This state-of-the-art review shows that the currently operational quality control methods are the same in each countries surveyed, and that there has not yet been any technological breakthrough in the physical principles of these methods in relation to those developed in the 70s and 80s, except for automation of the methods to which this can be applied (image analysis, for example). On the other hand, a strong R&D culture has been identified, essentially in the USA, which precisely aims to propose innovative, non-destructive methods, particularly where layer density and/or layer thickness would make it possible to reduce the cost of such controls.

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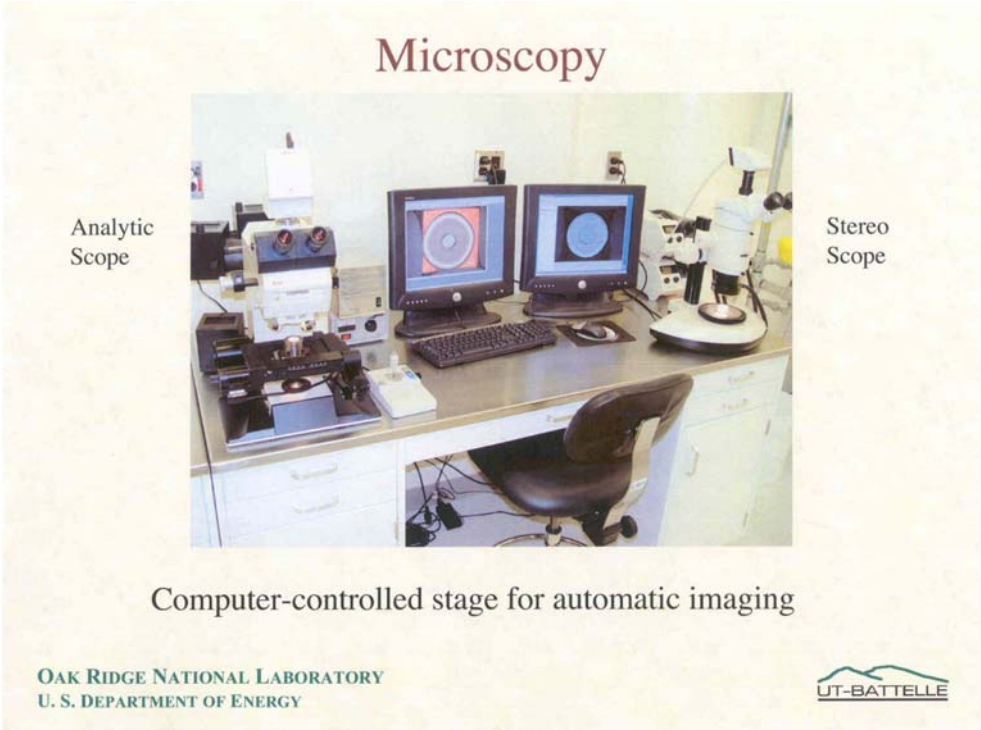
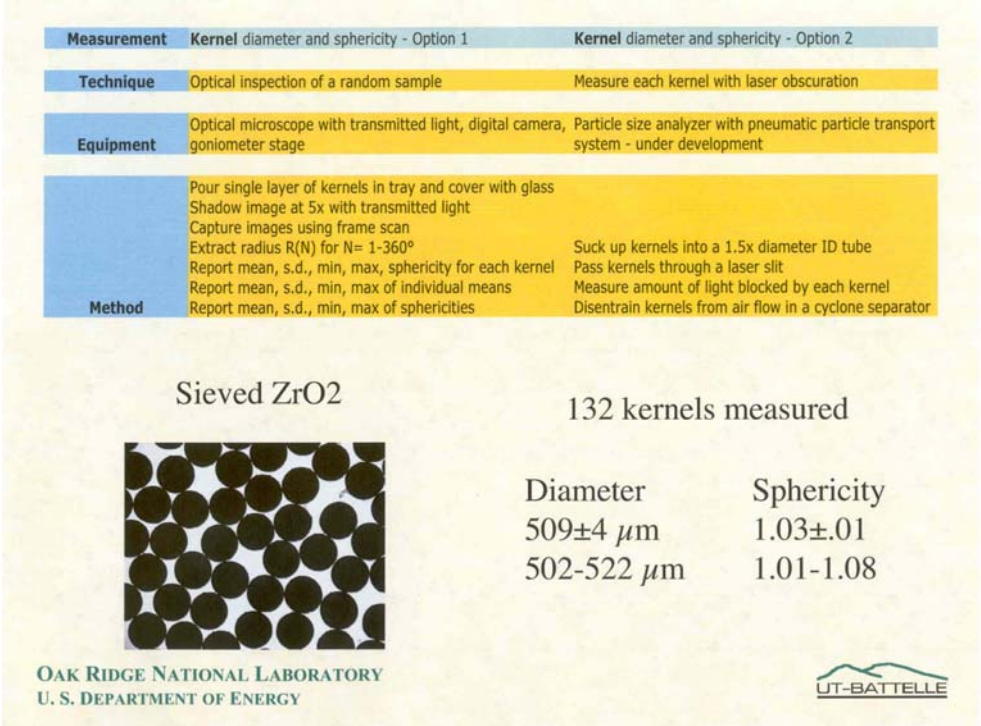
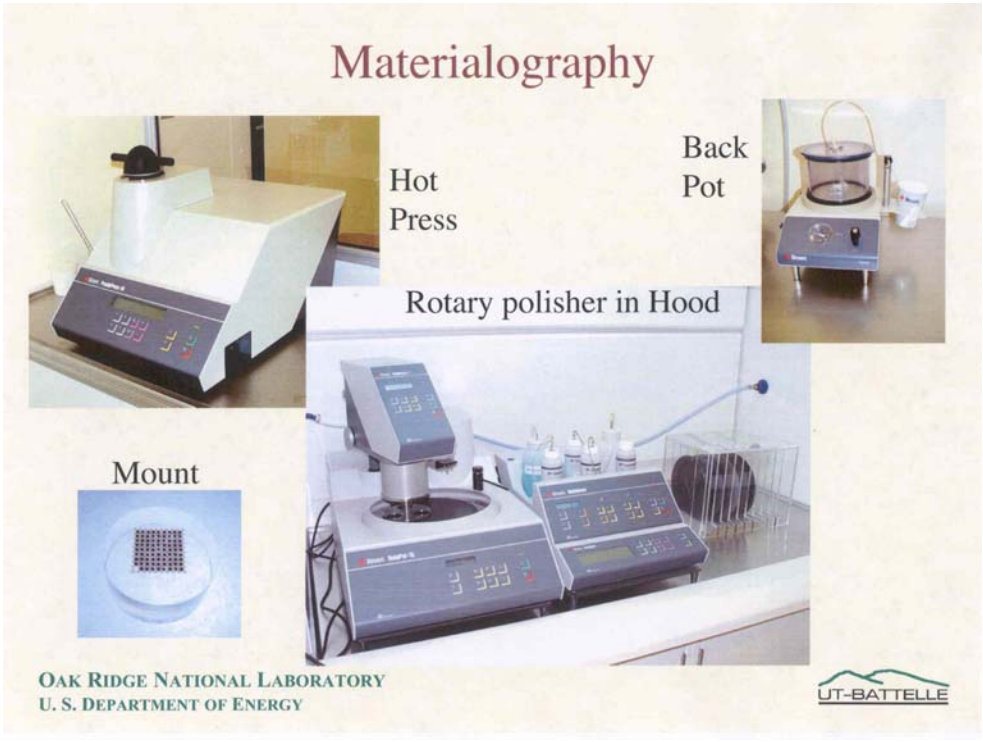
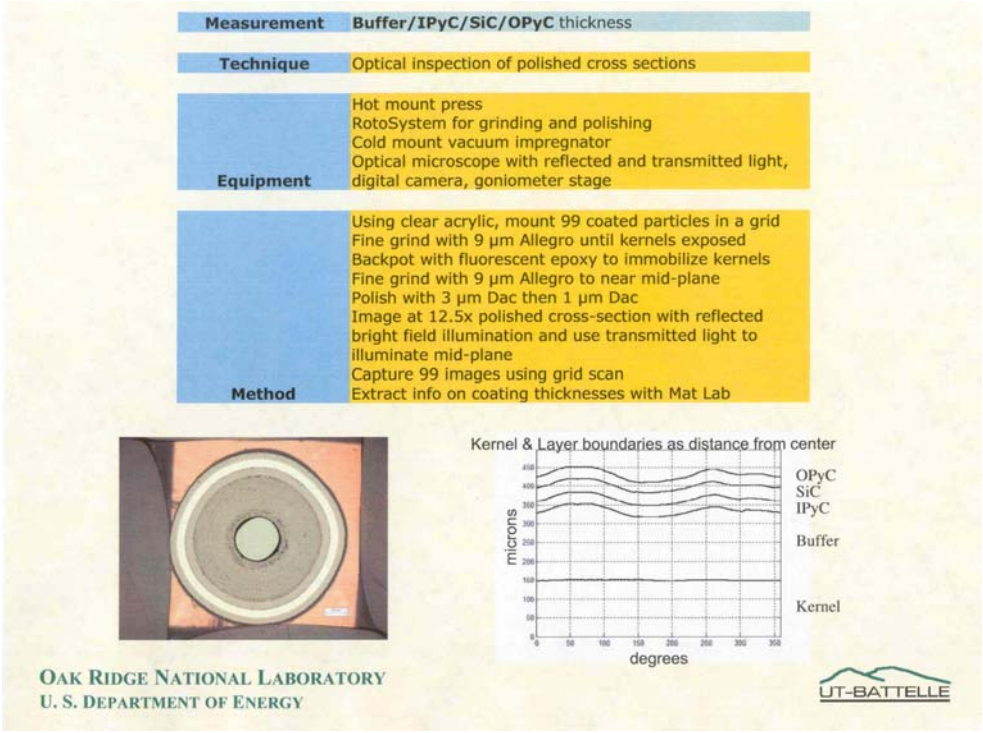
Appendix 1: Slides presented at Global2003 illustrating characterisation by ORNL [HUN2003]





Appendix 1 (continued): Slides presented at Global2003 illustrating characterisation by ORNL [HUN2003]





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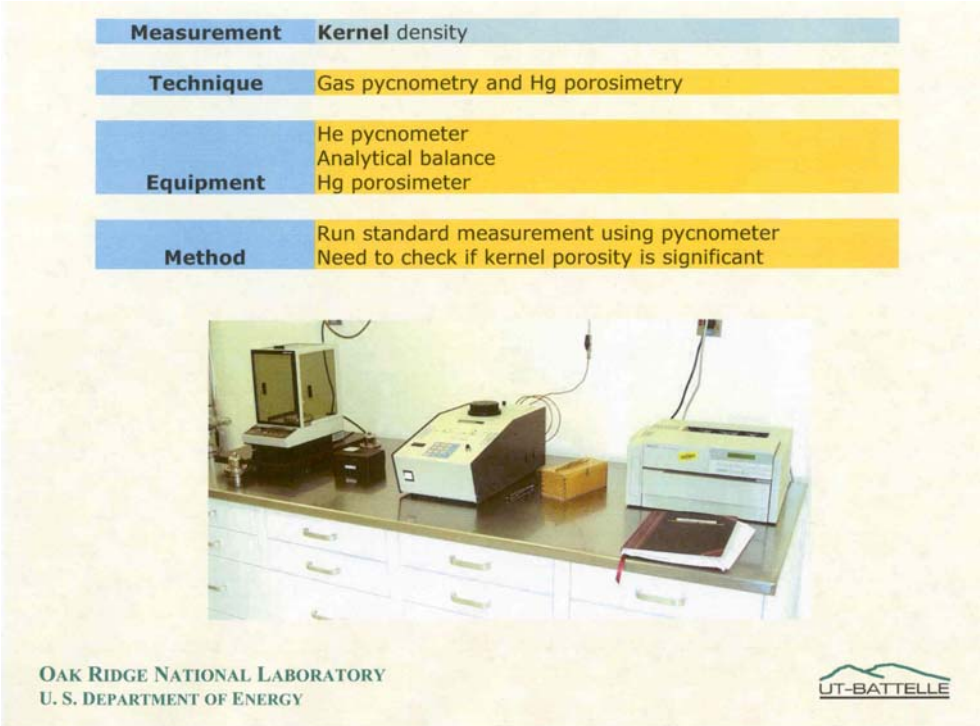
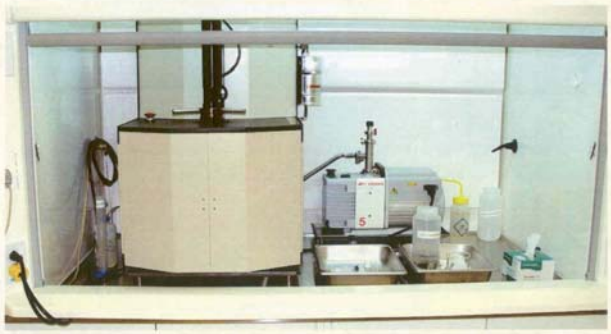


Appendix 1 (continued): Slides presented at Global2003 illustrating characterisation by ORNL [HUN2003]

Measurement	Buffer density
Technique	Measure buffer coated kernel and subtract out contribution of kernel
Equipment	Hg porosimeter Particle counter
Method	Count representative sample of kernels Measure weight and volume of kernels Count representative sample of buffer coated kernels Measure weight and volume of buffer coated kernels Density of buffer = Diff. in av. weight / Diff. in av. Volume



Mercury Porosimeter In Hood

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

UT-BATTELLE

Measurement	SiC density	OPyC density
Technique	Measure float density and check porosity	Same
Equipment	Liquid gradient density column Temperature controlled water bath Peristaltic pump with fill system Calibrated reference floats Hg porosimeter	Same
Method	Build 3.1- 3.3 g/cc column with Bromoform and Methylene iodide	Build 1.7-2.1 g/cc column with Tetrachloroethylene and Dibromoethylene

SiC

PyC

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

UT-BATTELLE

Appendix 1 (continued): Slides presented at Global2003 illustrating characterisation by ORNL [HUN2003]

Improved Ellipsometry Method for Measuring Pyrocarbon Anisotropy

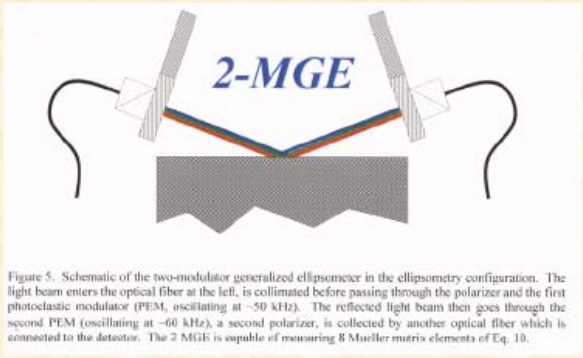


Figure 5. Schematic of the two-modulator generalized ellipsometer in the ellipsometry configuration. The light beam enters the optical fiber at the left, is collimated before passing through the polarizer and the first photoelastic modulator (PEM, oscillating at ~50 kHz). The reflected light beam then goes through the second PEM (oscillating at ~60 kHz), a second polarizer, is collected by another optical fiber which is connected to the detector. The 2-MGE is capable of measuring 8 Mueller matrix elements of Eq. 10.

A key TRISO specification

Existing Method Drawbacks (OPTAF):

- reproducibility
- correlation with performance

New Technique:

- gives complete info.
- less sensitive to artifacts
  - stray reflections
  - polishing artifacts
  - alignment error
  - porosity
- should correlate better
- higher resolution – profiling is possible

Method has been calibrated using samples of highly oriented pyrolytic graphite

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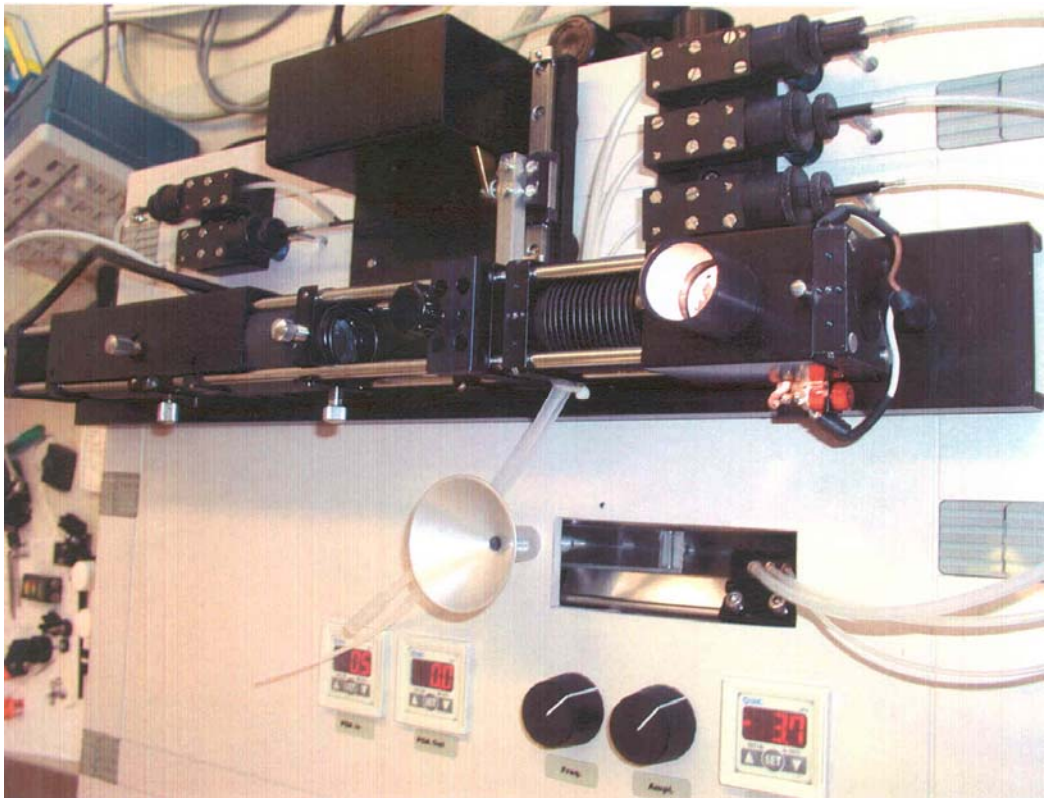
Appendix 2: Particle Size and Diameter Analyser (marketed by the Seibersdorf Research Centre in Austria).



Appendix 3: Images of the TP-CAST system under development at ORNL [KAR2005]

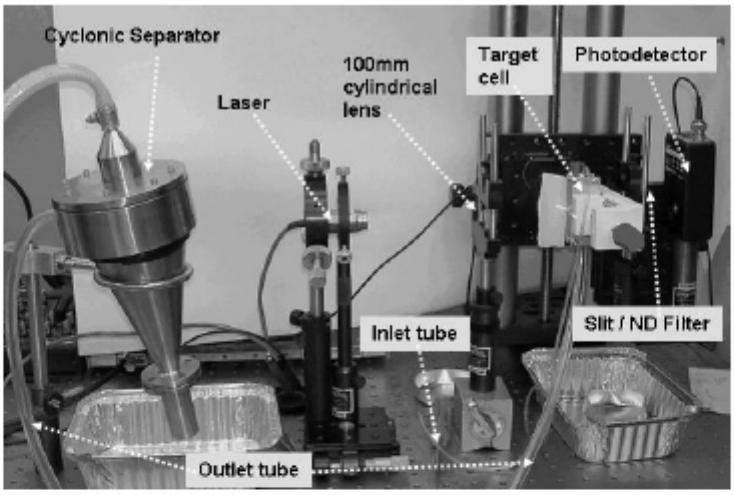


Figure 4. Experimental configuration of the TP-CAST

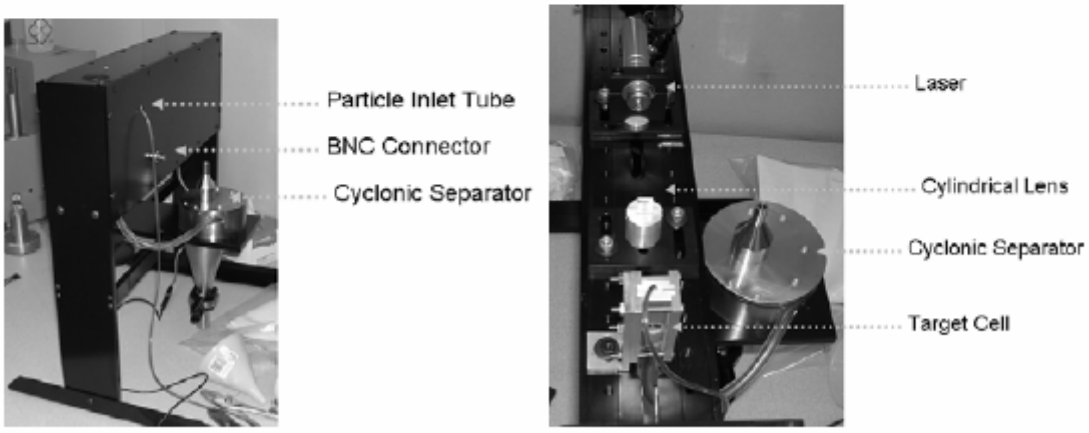
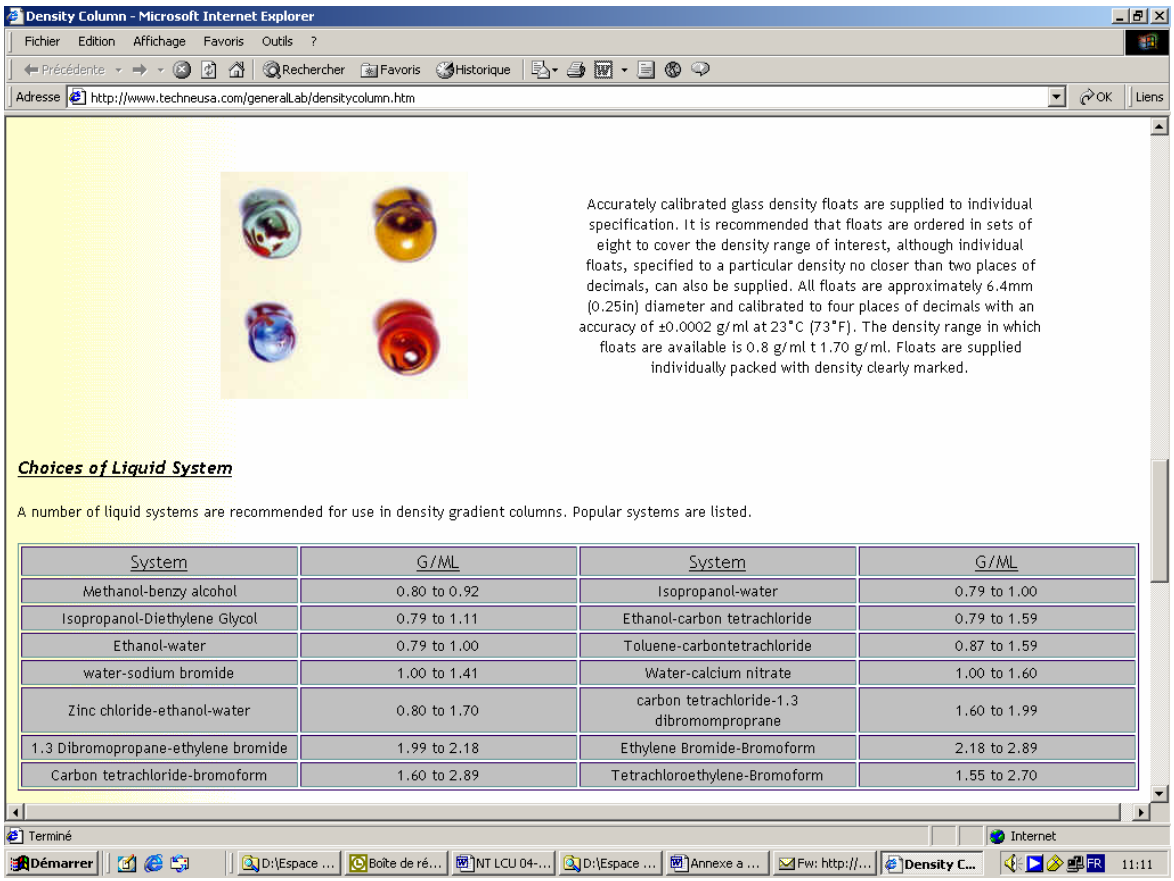


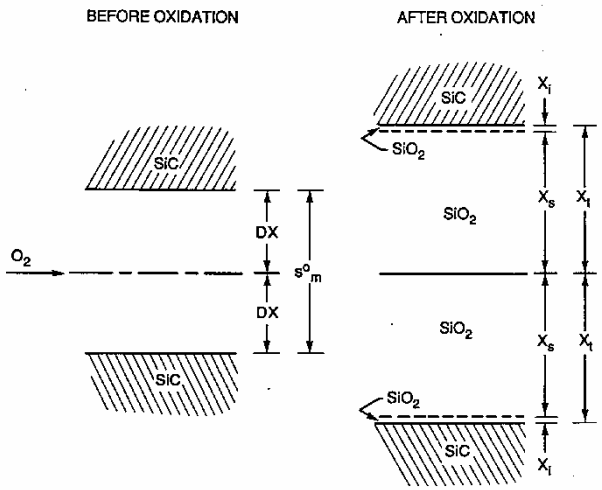
Figure 5. Completed TP-CAST instrument in light-shielding enclosure.

Appendix 4: Choice of liquid mixtures for a given density, description of calibrated floaters and database of the liquids used for floatation



Name	Formula	Mole mass	Boiling point (°C)	Density (at 20°C)
Isobutyl alcohol	$(CH_3)CHCH_2OH$	74.12	108.1	0.8018
Bromoform or tribromomethane	$HCBr_3$	252.73	150	2.89
Methylene iodide	ICH_2I	267.84	182	3.335
Toluene (methyl benzene)	$C_6H_5CH_3$	92.14	110.6	0.8669
Xylene	1,2- $(CH_3)_2C_6H_4$	106.17	144	0.8802
Ethyl iodide	CH_3CH_2I	155.97	72	1.936
Tetrachloroethylene	$Cl_2C=CCl_2$	165.83	121.14	1.6227
Dibromoethylene or ethylene,1-1-dibromo	$CH_2=CBr_2$	185.85	92	2.178
Tetrabromoethane or tetrabromoethylene	$Br_2CHCHBr_2$	345.65	244	2.966

Appendix 5: Behaviour of a fissure in a SiC layer during air combustion [MYE1989]



DX = THE TRANSLATION OF THE SURFACE ALONG THE NORMAL
s^o_m = THE INITIAL SEPARATION OF THE SiC SURFACES
X_l = THE THICKNESS OF THE REACTION-CONTROLLED OXIDE LAYER
X_s = THE THICKNESS OF THE TRANSPORT -CONTROLLED OXIDE LAYER
$X_t = X_l + X_s$

Fig. 3. Geometric changes upon oxidation of two, plane, parallel and facing surfaces of SiC.

Table 1. Calculated thicknesses of oxide layers formed at selected temperatures and times on plane, parallel, and facing SiC surfaces and maximum separation of surfaces bridged by the oxide layers

Temperature (°C)	Time (h)	X_s [a] (nm)	X_t [b] (nm)	s^o_m [c] (nm)
750	10	5.4	6.6	7
750	20	8.1	9.3	10
750	40	15	16	17
900	10	22	24	24
900	20	32	34	35
900	40	69	71	74
950	10	32	34	35
950	20	48	50	52
950	40	103	105	109

[a] X_s = the thickness of that portion of the oxide layer formed after the initial oxidation of the pristine SiC surface.

[b] $X_t = X_s + X_i$ where X_i is the thickness of that portion of the oxide layer formed on the initial contact of O_2 with the pristine SiC surface that has been determined by a linear interpolation between the values 3 nm at 1072°C and 1 nm at 772°C.

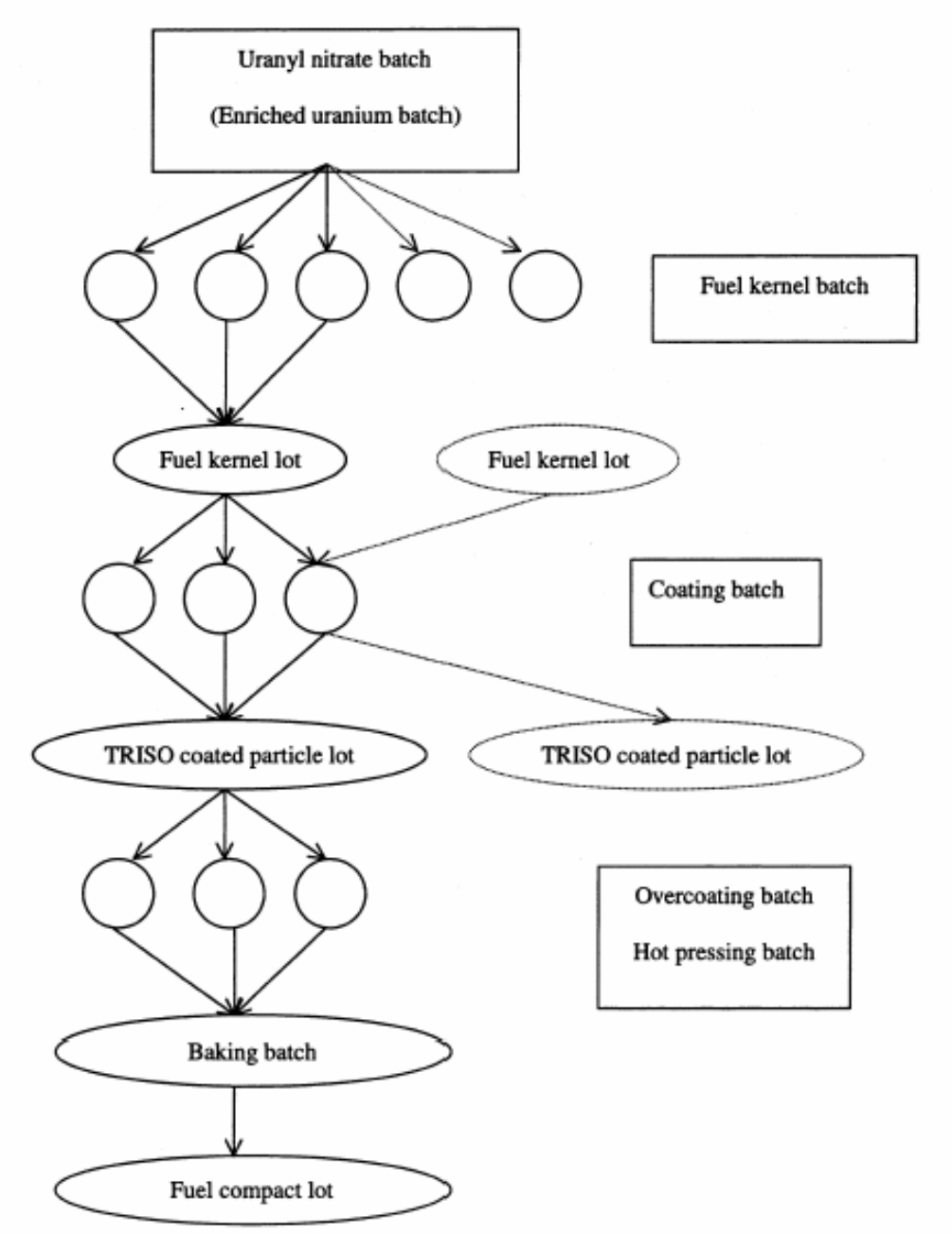
[c] s^o_m = the initial separation of the plane, parallel, facing SiC surfaces.

Appendix 6: Sampling carried out in Japan (HTTR) and China (HTR-10) [XFU2002]

Inspection items	Inspection method	Sampling rate
Fuel kernel		
^{235}U enrichment	Mass spectro analysis & γ-ray spectro analysis	1 sample / enrichment
Diameter	Optical particle size analysis	1 sample (100 particles) / Fuel kernel lot
Sphericity	Optical particle size analysis	3 samples (100 particles / sample) / Fuel kernel lot
Density	Mercury substitution	3 samples / Fuel kernel lot
O/U ratio	Oxidation & weighing	1 sample / Fuel kernel lot
Impurities	Emission spectro analysis	1 sample / enrichment
Coated fuel particle		
Layer thickness	X-ray radiograph	1 sample (50 particles) / coating batch
Layer density	Solvent substitution or sink-float	3 samples / coated fuel particle lot
Optical Anisotropy Factor	Polarization photometer	1 sample (5 particles / sample) / enrichment
Diameter	Optical particle size analysis	1 sample (100 particles) / Coated fuel particle lot
Appearance	Visual observation	1 sample (2000 particles) / Coated fuel particle lot
Cross-section	Ceramography	1 sample (20 particles) / Coated fuel particle lot
Sphericity	Selection by vibration table	All coated fuel particles.
Strength	Point crushing	30 particles / enrichment
Fuel compact		
^{235}U enrichment	Mass spectro analysis & γ-ray spectro analysis	1 sample / enrichment
U content	γ-ray spectro analysis	All fuel compacts
O/U ratio	Oxidation & weighing	1 sample / Fuel compact lot
Graphite powder	Density, impurities, grain size, water content	1 sample / graphite powder lot
Binder	Contents, ash, melting point, impurities	1 sample / binder lot
Exposed uranium fraction	Deconsolidation & acid leaching	2 samples / Fuel compact lot
SiC-failure fraction	Burn & acid leaching	3 samples / Fuel compact lot
Packing fraction	Weighing & calculation	3 samples / Fuel compact lot
Matrix density	Weighing & calculation	3 samples / Fuel compact lot
Dimensions	Micrometer	All fuel compacts
Appearance	Visual observation	All fuel compacts
Marking	Visual observation	All fuel compacts
Strength	Compression	3 samples / enrichment
Cross section	Ceramography	1 sample / Fuel compact lot
Impurities	Emission spectro analysis	1 sample / enrichment

Table summarising the quality control methods and sampling associated with HTR production for HTTR in Japan. [XFU2002]

Appendix 6 (continued): Sampling carried out in Japan (HTTR) and China (HTR-10) [XFU2002]



Composition of batches in accordance with the Japanese HTTR program [SAW2001],

According to [YOS1991], Batch size = 3-4 kg U; Lot size = 9-10 kg U - 1 lot = 3 batches; 1 Fuel Compact Lot = 700 Compacts

Appendix 6 (continued): Sampling carried out in Japan (HTTR) and China (HTR-10) [XFU2002]

Inspection items	Inspection method	Sampling rate
Fuel kernel		
^{235}U enrichment	Mass spectrometer	3 sample / batch
Diameter	Radiograph and project	1 sample (200 particles) / batch
Sphericity	Image analyzer	1 samples (200 particles / sample) / batch
Density	Pycnometer	1 samples / batch
Fraction of odd shape particle	Vibrating on plate	1 sample(30,000 particles)/batch
O/U ratio	Thermogravimetric method	1 sample (0.2g)/ batch
Impurities	Chemical analysis	1 sample (0.5g)/ batch
Coated fuel particle		
Layer thickness	Radiograph and project	1 sample (200 particles) / batch
Layer density	Sink-float method	1 samples(200 particles) / batch
Optical Anisotropy Factor	Optical microscopy method	1 samples(10 particles) / batch
Diameter	Radiograph and project	1 sample (200 particles)/batch
Appearance	Visual observation	
Microstructure	Ceramography	1 sample (200 particles) / batch
Failure fraction	Burning/ acid leaching	1 sample (25g particles)/batch
Graphite matrix ball		
Density	Weighing and calculation	5 sample / batch
Total ash	Burning and chemical analysis	1 sample / batch
Li content	Burning and chemical analysis	1 sample / batch
Impurity	Burning and chemical analysis	1 sample / batch
Thermal conductivity	Laser pulse method	3 sample / batch
Corrosion rate	Corrosion furnace test/ weighing	3 samples / batch
Erosion rate	Erosion device/ weighing	20 samples / batch
Number of drops	Free dropping device	5 samples / batch
Breaking loading	Pressing device	10 samples / batch
CTE anisotropy,$\alpha_{\perp}/\alpha_{//}$	Thermal expansion analysis	6 sample / batch
Spherical fuel element		
Diameter	Visual observation	All fuel elements
Thickness of fuel-free shell	X-ray project	All fuel elements
U-loading	Calculation	5 sample / batch
U contamination	Acid leaching	2 samples/ batch
Free Uranium fraction	Burning/ acid leaching	2 sample / batch

Table summarising the quality control methods and sampling associated with HTR production for HTR10 in China [XFU2002]