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Document title

Review on technologies to recover TRISO fuel from graphite matrix

Executive summary

This report provides the results of some previous studies performed in AREVA in the frame of the ANTARES program to assess HTR compact/fuel particle separation methods for further reprocessing (see [9]). This document complements the work performed by FZJ in the frame of the CARBOWASTE WP2 (see [10]).

Methods consisting in successive grinding and burning are considered as the reference and technically most advanced head-end process for HTR fuel reprocessing. The major drawbacks are the off-gas treatment and the dust management. Some other innovative technologies for separating fuel particles from the graphite matrix of compact have been identified:

- disintegration by electrical pulse
- disintegration by ultrasounds
- treatment by hydrofluorination
- combustion in molten salts

Physical methods require further sorting out of fuel particles and eventual further combustion of residual carbon. Chemical methods require preliminary destructuration of compacts. Except disintegration by ultrasounds, these methods have been proved to allow the elimination of the particle SiC coating.

It is still difficult at this stage to privilege one method of compact /fuel separation since elements such as economical point of view have not been assessed and even technical feasibility is still not demonstrated on entire compact.

However the most promising innovative technologies to be tested and compared with the classical methods (grinding/burning) performance, appear to be the disintegration by electrical pulse and the treatment in molten salts. With regard to nuclear constraints, these methods carried out in liquid phase are of interest since they do they do not induce dust spreading. Nevertheless treatment in molten salt should be carried out at high temperature and generates gas to be treated.

Thus it is important to pursue and follow the electrical pulse tests performed by CEA.

Disintegration by ultrasound should be investigated through a bibliographical review to determine if further experiments of this method on compact would be of interest.

Treatment in molten salts should be investigated prior to hydrofluorination (HF/O_2) since this latter treatment in gas phase requires dust management. In addition hydrofluorination requires more preliminary treatment steps (crushing of fuel particles and burning off in oxygen).

Revisions						
Rev.	Date	Short description	Author	Internal Review	Task Leader	WP Leader
00	27/05/2010	1. Issue	F. Cellier, ANP 	V. Grabon, ANP 	v. Lensa, FZJ 	v. Lensa, FZJ 

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1 Introduction

This report provides the results of some previous studies performed in AREVA in the frame of the ANTARES program to assess HTR compact/fuel particle separation methods for further reprocessing (see [9]). This document complements the work performed by FZJ in the frame of the CARBOWASTE WP2 (see [10]).

The HTR spent fuel includes significant amounts of carbon and SiC waste, since the fuel kernels account for only few percent of the entire fuel element mass (see figure 1). This characteristic is specific of HTR design and is different from most other reactor types. With regard to direct disposal of spent fuel as High Level Waste (path A on figure 2), separating the fuel compacts from the fuel elements allows the disposal or recycling of moderator graphite - which represents the bulk of the waste volume - as Low Level Waste. Reprocessing fuel particles allows recycling of elements of interest (uranium, plutonium) that would reduce the radiotoxicity of the final waste. It allows the dissociation of problems linked with Carbon 14 release from those linked with fission products treatment. This way is identified as path C among the different options on figure 2.

The separation of compacts from the graphite fuel assembly is assumed to be achieved in a first step by physical processes (mechanical extraction, electrical pulse, ultrasounds). The scope of this work is to examine the technologies that are currently available for separating the fuel particles from the graphite matrix of the compact and to identify R&D needs in this area. The aim is to recover the bare fuel kernel without its coating layers – particularly the silicon carbide coating – to allow further dissolution of fuel kernel in nitric acid prior to chemical separation and recovery of the valuable fuel elements (U, Pu).

The difficulties to separate fuel kernels from the compact graphite are due to compact characteristics. Fuel kernels represent about 18% of the compact mass [1] but only 2.5% of the compact volume and is dispersed in numerous little particles of diameter 920 µm (500µm UO2 diameter, 95µm buffer, 40µm PyC internal layer, 35µm SiC layer, 40µm PyC external layer). The SiC coating is characterized by its high thermal and chemical stability.

Available technologies for separating fuel particles from graphite compacts have been identified through a preliminary bibliographical review.

2 Feedback of relevant experience

HTR fuel particles reprocessing experience is gained through former HTR development: Peach Bottom, Fort St.Vrain in USA, AVR in Germany. Some information about graphite treatment also issue from the feedback of Natural Uranium Graphite Gas reactors (UNGG).

2.1 Feedback from former HTR

HTR fuel reprocessing has already been studied and experienced in 1960-1980 [2], [3], [4] in the USA (Oak Ridge National Laboratory, General Atomic Corporation, Allied Chemical Corporation) and in Germany (Forschungszentrum Jülich).

All methods used to separate fuel particle from graphite (compact or pebble) consist in successive grinding and burning steps. Grinding gives access to the fuel particles and enhances the reactive surface for burning. Burning is used to remove the large amount of graphite compared with the relatively small amount of fuel material. This procedure is considered as the reference and technically most advanced head-end process for HTR fuel.

Grinding

The fuel must be crushed to suitable particle size for maintaining fluidization quality during further combustion in the fluidized-bed burner. Different types of grinding machines have been used: jaw crushers, hammer crushers, pebble crushers, crushing rollers, etc. For example the process developed by ORNL for primary crushing of compacts is described on figure 3 [2]. Generally the size of the obtained particles - in the order of few millimetres – was still high with regard to the fuel particles diameter (250 µm fuel oxide diameter) to minimize fuel particle breakage and to prevent undesirable cross-over of fuel. Only few percent of the particle SiC coatings are broken. Thus, after burning, the particles are sent through a small roll crusher or jet grinder for a second crushing in order to break the SiC shell. About 5% of the fuel particles stayed intact and inaccessible for further dissolution. During grinding sorting of

the particles is necessary with recycling of larger particles. Pneumatic or cyclone separators are often used.

Burning

After grinding, combustion is carried out in fluidized bed burners operating at temperature up to 950°C. Processes are different depending on the temperature of combustion. At low temperature the SiC coating remains intact because of its high thermal stability. To destroy it, the burning temperature should be higher than 1300°C, but it would involve the volatilization of fission products that would lead to difficulties to treat exhaust gases.

US process [2] consists in primary burning at 850-875°C (so-called "exothermic") to remove the graphite and the outer pyrolytic carbon coating from the TRISO particles. Then, after second crushing to break the SiC coating, particles are burned in a secondary fluidized bed burner (so-called "endothermic") to remove the inner carbon coatings. Generally about 95% of fissile elements were recovered. Carbon residues due to non complete combustion are reported to form carboxylic acids during further dissolution in nitric acid, that lead to difficulties during separation processes.

The primary burner is the main source of off-gas: carbon dioxide with associated relatively very small volumes of radioactive gases 85Kr, 129I, 220Rn, 3H, and also entrained fines. Fines are removed through cyclone and/or sintered metal filter to be recycled while exhaust gases pass through off-gas treatment. ORNL off-gas treatment [2] consists in removal of krypton by absorption by liquid carbon dioxide (KALC process), removal of iodine by lead and silver zeolites, hold up of radon on molecular sieve to permit decay to solid products and tritium removal in the form of tritiated water on molecular sieve. Off-gas treatment was not optimized with regard to carbone 14 release.

As said previously, the crushed compacts introduced in the primary burner may contain a small but still significant fraction of particles with broken SiC coating. According to experiments carried out between 700-900°C [5], the kernel of damaged particles is oxidized to U3O8 [4] associated with exothermic conversion, considerable swelling and disintegration of kernels into dust. Thus a non-negligible fraction of dusty heavy metal is to be expected in the exhaust gas.

2.2 Feedback from UNGG

A prototype incinerator was built in the 90s in Le Creusot in France in order to treat the graphite issued from French graphite gas-cooled reactors (UNGG commissioned in 1950-1970s) [6]. Graphite waste is mainly in the form of spent fuel sleeves or graphite blocks.

Graphite blocks are first crushed by shredder, hammer-type and cylindrical crushers in order to obtain a final average particle size of one millimeter. The particle size is expected to be larger than 100 µm to limit dust dissemination and dust explosion risk. The crushing station is located inside an enclosure kept under negative pressure, swept by high air flow rate and filtered. Thus inerting of the crushing room did not seem to be necessary.

Then the crushed graphite is burned in a circulating fluidized bed incinerator. The fluidized bed consists of powdered refractory material under high air flow rate and high turbulence. Solid particles are separated from the combustion gases through a cyclone separator and recycled by a recirculating loop with no moving mechanical parts. Incineration of the unburned graphite contained in the combustion gases leaving the cyclone separator is completed in a post-combustion chamber. The tightness of the combustor allows it to work under relative negative pressure. Emitters 3H and 14C are released in the atmosphere with gradual and controlled dilution. An impact assessment of graphite incineration on the environment was performed.

A validation program was carried out on the prototype incinerator. Combustion was complete and well-controlled.

3 Review of available technologies

Different technologies using physical or chemical methods have been identified for compact/fuel separation.

Physical methods:

- ✓ Grinding
- ✓ Electrical pulses

- ✓ Ultrasound

Chemical methods:

- ✓ Combustion
- ✓ Treatment by halogen gas
- ✓ Treatment in molten salts

3.1 Grinding

With regard to the past experience and to the performance of existing fine/ultra fine grinding machines (see table 1), it could be expected to reach graphite particles granulometry close to the fuel particle diameter or lower. Separation method of graphite from fuel particle based on particle size difference could only be used if SiC coatings remain intact during grinding. However fuel particles would be broken during fine grinding. A separation method based on particle density difference has been performed in Germany for HTR fuel sphere (pebble design) and allowed pre-separation of 90% of the main matrix graphite prior to a further treatment [7]. The process principle is shown on figure 4. First the graphite sphere is introduced in a peeling and brush-off unit to remove the outer heavy metal-free zone. Then the remaining sphere is disintegrated by rotating metal brushes. Graphite fines are removed at each step. The residual fragments are crushed by jaw crusher. It is noteworthy that the coated particles remain intact at this stage. In the further crushing step SiC shells are broken. Kernel particles (density about 10) are separated from graphite fines (density about 1.7) and SiC shells by gas elutriation through a pneumatic classifier. However the remaining carbon residue of about 10% has to be burned.

Constraints associated to grinding are dust dissemination, abrasion of moving pieces, further treatment required to eliminate graphite. Advantage is generally high capacity.

Grinding is an available technology only to be used as a pre-treatment or a pre-separation method of fuel particles from the main part of graphite prior to further treatment.

3.2 Electrical pulses

Fragmentation of solids is induced by high voltage electrical pulses. Electric energy is stored in capacitors and discharged in very short time through the solid between two electrodes (see figure 5). The solid is immersed in water. In heterogeneous materials, fragmentation occurs at the interface between the different constituents. Fine granulometry may be obtained on graphite (see figure 6).

This technology is currently being investigated by CEA. Feasibility tests were carried out in June 2004 in which graphite and SiC samples and TRISO particles were submitted to the direct process (also called localized process : electrical arc is directly applied to the sample) using MAX pulse generator. Applied conditions were:

- Energy level: 500 J
- Voltage: 250 kV
- Impulsion duration: 200 ns
- Peak intensity: 6 kA

Adjustable parameters were impulsion number and time between impulsions.

Interesting results have been obtained on TRISO particles (see figure 7): Nearly total destructuration of particle coatings after 40 impulsions whereas the fuel kernels remain intact. This process would allow further dissolution of bare fuel kernels after sorting out of cracked SiC shells from the fuel kernels (see figure 5).

It is expected that destructuration by electrical pulse technology applied to the entire compact could allow the recovering of fuel particles from the bulk compact graphite assuming that sorting out of fuel particles is possible as shown on figure 5.

Interests of this method are short time treatment and no dust management as the solid destructuration is carried out under water.

Nevertheless further studies and tests are needed to demonstrate the feasibility of this technology according to following requirements:

- performance on representative samples (compact with TRISO particles)

- nuclear application of this technology
- application to high capacity (feasibility tests were limited to little samples : 3 g TRISO particles)
- development of particles sorting out
- energy consumption

3.3 Ultrasound

Ultrasound waves may produce heavy effect on material depending on the transmitted power. For disintegration applications ultrasound power ranging from some watts to several kilowatts is applied with an ultrasound frequency of about 20 kHz. Ultrasounds are transmitted through a liquid medium; generally the piece is immersed in water containing abrasive particles. Ultrasounds induce particles vibrations. The disintegration of the solid results from the mechanical action of the particles - shocks and friction due to particles vibrations - and from the cavitation effect due to pressure variations inside water. This technology is used to machine hard and fragile or porous materials. Theoretically graphite could be easily disintegrated by this process whereas disintegration of harder and less fragile material as SiC would be more difficult and would lead to heavy wear of the sonotrode. Generally silicon carbide is used as abrasive particles for graphite. It has been shown that the optimum size of abrasive particles is in the order of 100 µm to obtained maximal disintegration rate.

If the disintegration of compact could reduce graphite to fine particles - compared with the fuel particles size – and without damage to particle SiC coating, sorting out of intact fuel coated particle could be possible. Then fuel coated particles could be treated separately from the relatively large quantity of graphite. This method would not require dust management since it is carried out in liquid phase.

Preliminary study and tests would be necessary to assess the feasibility of this method applied to compact disintegration. Application to high treatment capacity should be demonstrated.

3.4 Combustion

Burning of compact issued from former HTR in fluidized bed has been largely developed in the past. This technology requires preliminary grinding of compacts. By burning, the main graphite is eliminated. As said previously, the elimination of SiC particle coating can be expected only at very high temperature combustion leading to undesirable volatilization of fission products. Thus after burning at usual temperature (700-900°C) further treatments will be required to break the SiC coating (fine grinding) and to eliminate inner carbon layers (secondary burning or other chemical method) to recover the fuel kernel.

The main difficulties consist in the management of contaminated dust and in the treatment of combustion gases.

3.5 Treatment by halogen gas

Treatment by halogen gas allows the elimination of the SiC particle coating. After grinding of compacts, SiC can be removed by elementary fluorine or chlorine at very high temperature (>1500°C) as proposed by Argon National Laboratory (USA).

The reaction (given for fluorine),

$SiC + 4\ F2 \rightarrow SiF4 + CF4$ (fluorination)

is strongly exothermic and may result in a sintering of the particles. It leads to the volatilization of fission products and of uranium in the form of UF6 and to the formation of the following volatile halogen species [7][8]:

CFx, SiF4, UF6, PuF6.

The presence of SiF4 in the fluorination exhaust gas causes difficulties in the separation and purification of UF6.

According to German authors [8], this problem could be avoided by another method allowing the particles treatment at lower temperature. This method consists in hydrofluorination. SiC does not react with HF directly, but in a HF/O2 mixture. The entire PyC/SiC/PyC coating of particles could be totally removed at low temperature (about 500°C):

$$SiC + 4\,HF + 2\,O_2 \rightarrow SiF_4 + 2\,H_2O + CO_2$$

This method also requires preliminary grinding of the particles (to about 200 µm) and burning off the PyC layer with oxygen at about 800°C. In these conditions SiC can be removed by hydrofluorination at a temperature lower than 700°C at which severe corrosion problems may occur. However corrosion resistant material (Nickel, Monel) must be preferred for the hydrofluorination reaction vessel.

The feasibility of hydrofluorination in HF/O2 mixture has been demonstrated with good efficiency for limited batches. Total elimination of SiC has been obtained after some hours in HF/O2 in a fluidized bed reactor at 500°C on 150 g batches of oxygen treated particles.

3.6 Treatment in molten salt

Treatments by molten salts are also to be investigated.

This method consists of carrying out the combustion by oxygen or nitrogen dioxide in molten salt medium. Preliminary studies showed that the combustion kinetic close to dry combustion kinetic may be expected at high temperature (> 900°C).

The main interest of molten salt is that on one hand SiC may be eliminated and on the other hand the carbon may be partially used to dissolve fuel oxides according to the chemical reactions (for molten chloride application):

$$2\,Cl_2 + C \rightarrow CCl_4\uparrow$$

$$2\,Cl_2 + SiC \rightarrow C + SiCl_4\uparrow$$

$$MO_2 + 4\,Cl_2 + 2\,C \rightarrow MCl_4 + 2\,COCl_2\uparrow$$

Carbon and SiC are eliminated in the form of volatile halogen products whereas actinides and fission products may be recovered in the molten chloride solution by electrolyse or selective oxide precipitation.

The other main advantage of this method (as with any method carried out in the liquid phase) is that it does not require dust management. Nevertheless corrosion problems must be taken into account.

Similar treatments in molten sodium hydroxide or in molten carbonates are also reported. These treatments would generate carbon dioxide and solid silicate. Actinides could be recovered by classical process (PUREX for Uranium and Plutonium) from nitric medium.

4 Identification of R&D needs

It is obvious that several criteria must be taken into account for methods assessment:

- Performance in terms of: kinetic, % of obtained bare fuel particles or % of recovered fuel, treatment capacity
- Implementation constraints (associated problems: dust dissemination, abrasion, corrosion…)
- Effluent/waste management
- Nuclear constraints (treatment of nuclear material with respect to criticality, shielding, dose, proliferation, etc)
- Economical point of view

Constraints and requirements associated to the identified methods are summarized in table 2.

All physical methods are only expected to give access to the fuel particles but require further sorting out to recover fuel particles.

Electrical pulse appears to be one of the most attractive technologies which allow the elimination of the SiC coating to recover the intact fuel kernels assuming that sorting out is possible. Application to the disintegration of entire compact and to high capacity treatment is still to be demonstrated.

Grinding could be considered as pre-treatment/pre-separation for other methods. Grinding by steps would allow pre-separation of the main graphite followed by SiC breakage and recovering of fuel kernel by gas elutriation. Nevertheless this method requires further treatment (burning) to eliminate residual carbon.

All methods would require preliminary grinding of compacts except if destructuration by electrical pulses or by ultrasound technologies are proved to be directly applicable to compacts (electrical pulse tests to be performed by CEA).

Classical chemical treatments (burning) allow the recovering of fuel particles by elimination of the graphite and PyC coatings. But additional intermediate grinding step is required for breakage of the particle SiC coating. Classical burning methods require off-gas treatment and dust management.

Compared with classical combustion, chemical processes such as hydrofluorination (HF/O2) or treatment in molten salts would allow the elimination of SiC coating without formation of volatile compounds of actinides and fission products.

However, whereas classical methods - fluidized bed burning of crushed graphite material - have been experienced and developed to an advanced stage, the feasibility of these innovative methods - particularly the feasibility of treatment of compacts by molten salts - remains to be demonstrated and to be assessed in terms of performance on high capacity treatment and taking into account other associated constraints (corrosion).

The advantage of methods carried out in liquid phase (electrical pulses, ultrasounds, molten salts) is that dust management is not required.

5 Conclusion/recommendation

It is still difficult at this stage to privilege one method of compact /fuel separation since elements such as economical point of view have not been assessed and sometimes even technical feasibility is still not demonstrated.

However the most promising innovative technologies to be tested and compared with the classical methods (grinding/burning) performance, appear to be the disintegration by electrical pulse and the treatment in molten salts. With regard to nuclear constraints, these methods carried out in liquid phase are of interest since they do not induce dust spreading. Nevertheless treatment in molten salt should be carried out at high temperature and generates gas to be treated.

Thus it is important to pursue and follow the electrical pulse tests performed by CEA. Disintegration by ultrasound should be investigated through a bibliographical review to determine if further experiments of this method on compact would be of interest.

Treatment in molten salts should be investigated prior to hydrofluorination (HF/O2) since this latter treatment in gas phase requires dust management. In addition hydrofluorination requires more preliminary treatment steps (crushing of fuel particles and burning off in oxygen).

6 Figures & Tables

UO_2 mass: 5%
Buffer mass: 1%
Inner PyC mass: 1%
SiC mass: 2%
Outer PyC mass: 1%
Graphite mass: 90%

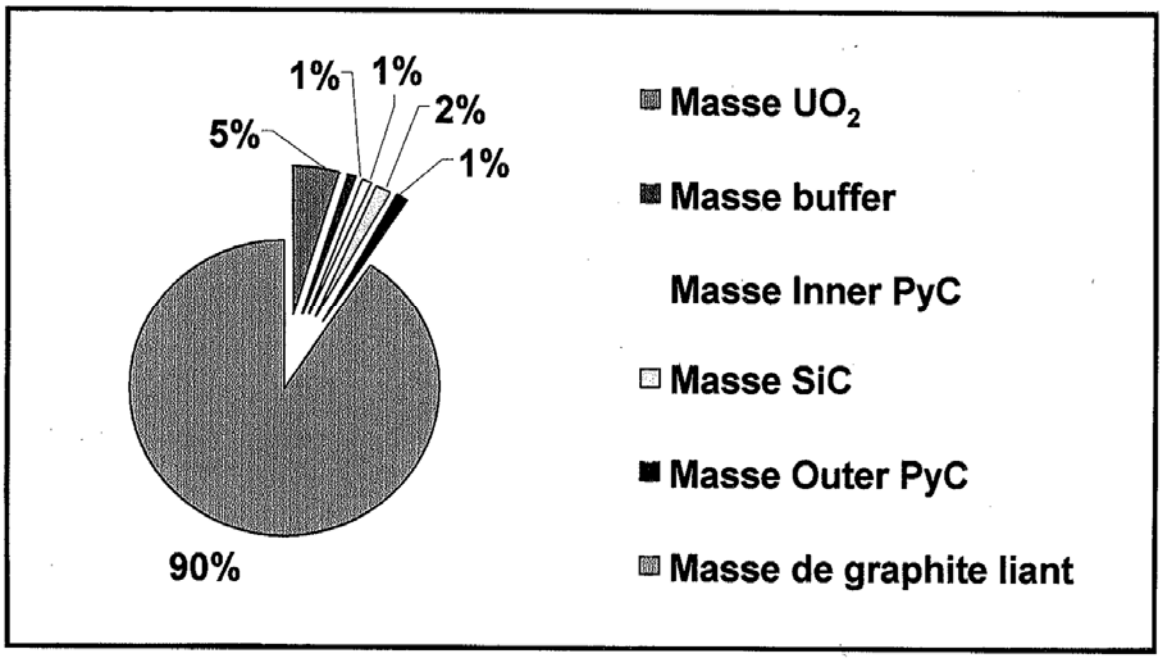

Figure 1: Typical weight composition of HTR fuel element

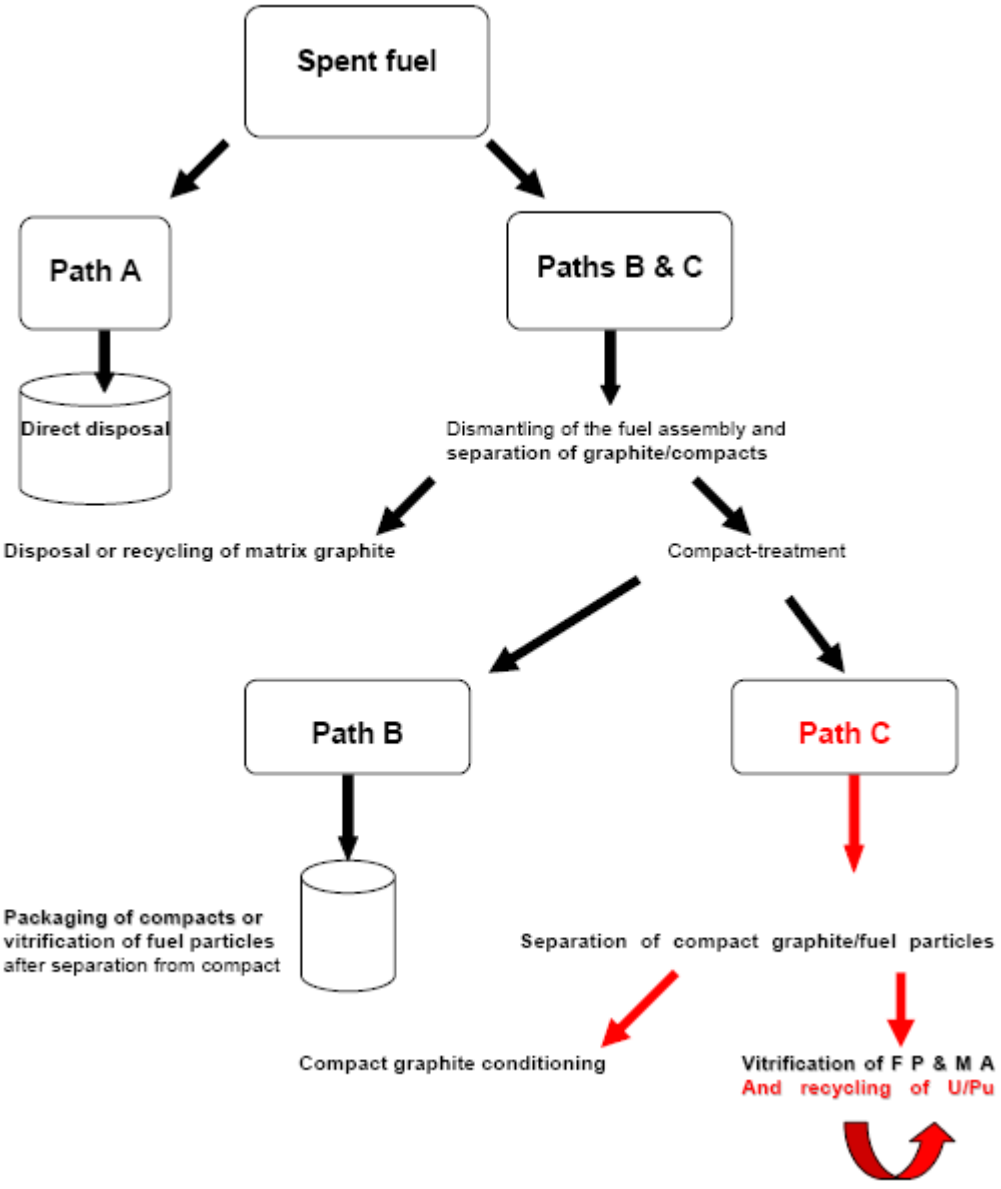

Figure 2: HTR spent fuel treatment options

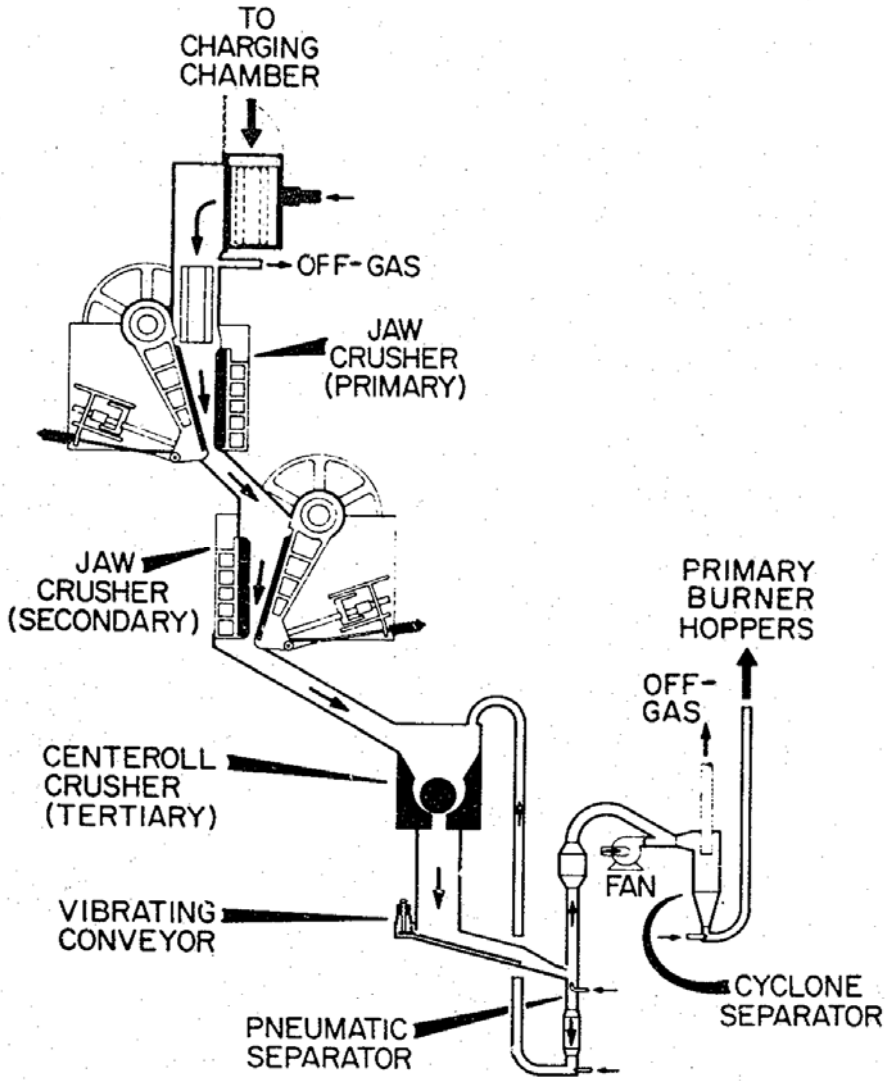

Figure 3: Schematic diagram of ORNL crushing system

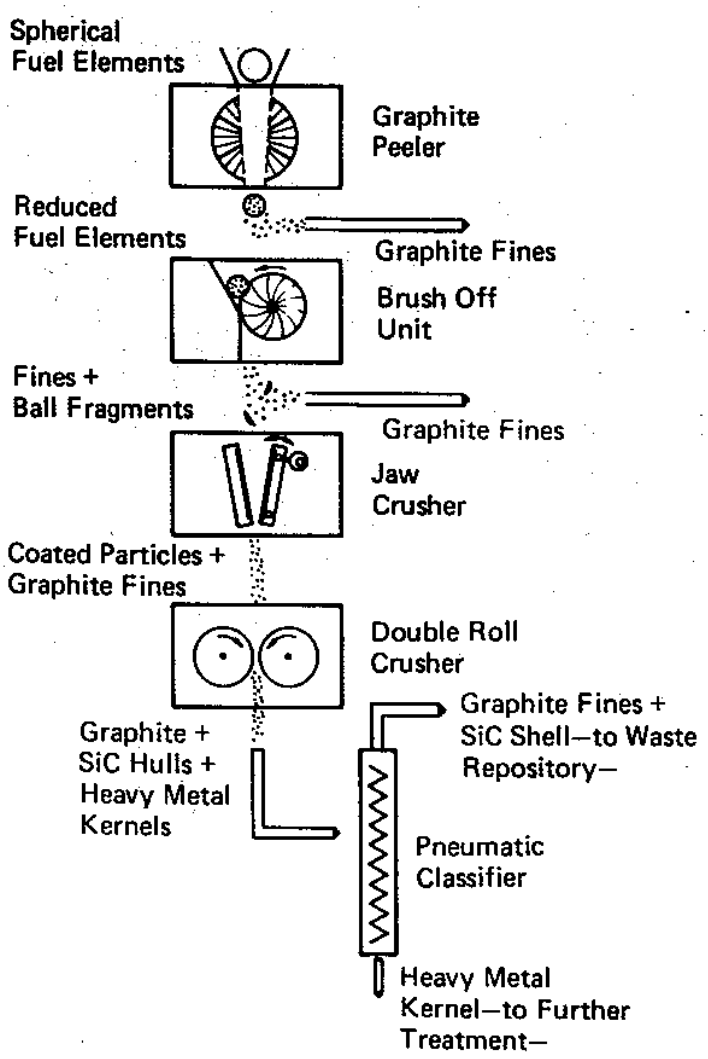

Figure 4: Graphite pre-separation by dry mechanical means

Separation method proposed by CEA

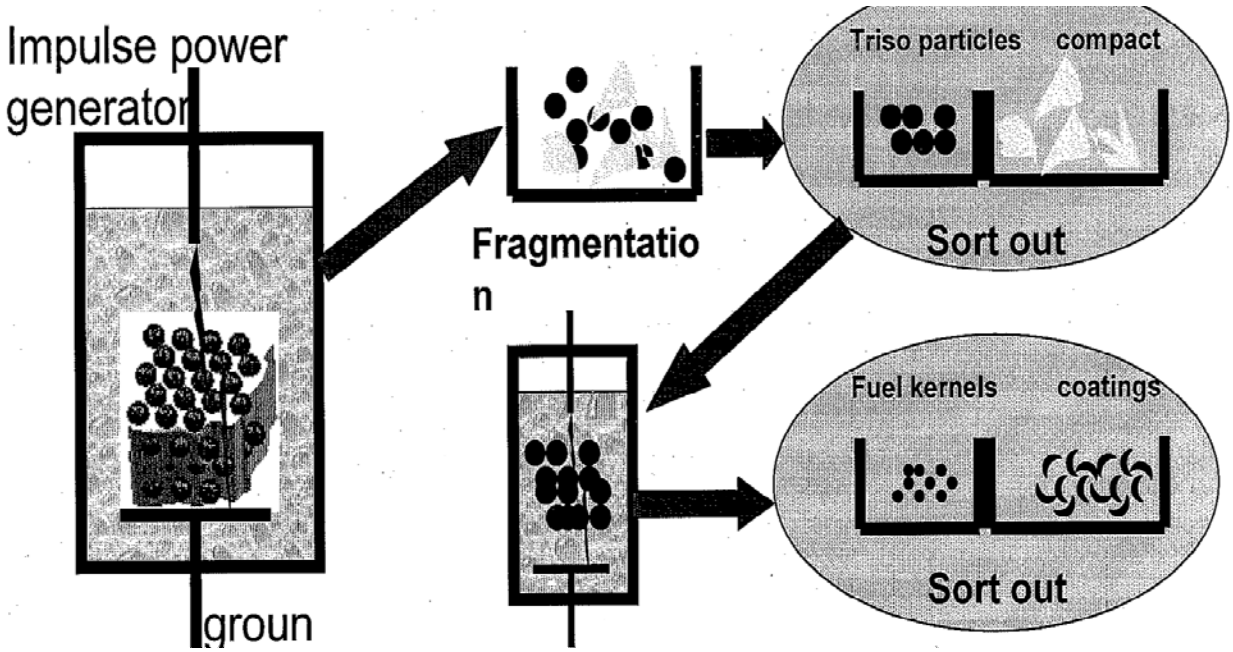

Figure 5: Electrical pulses separation method

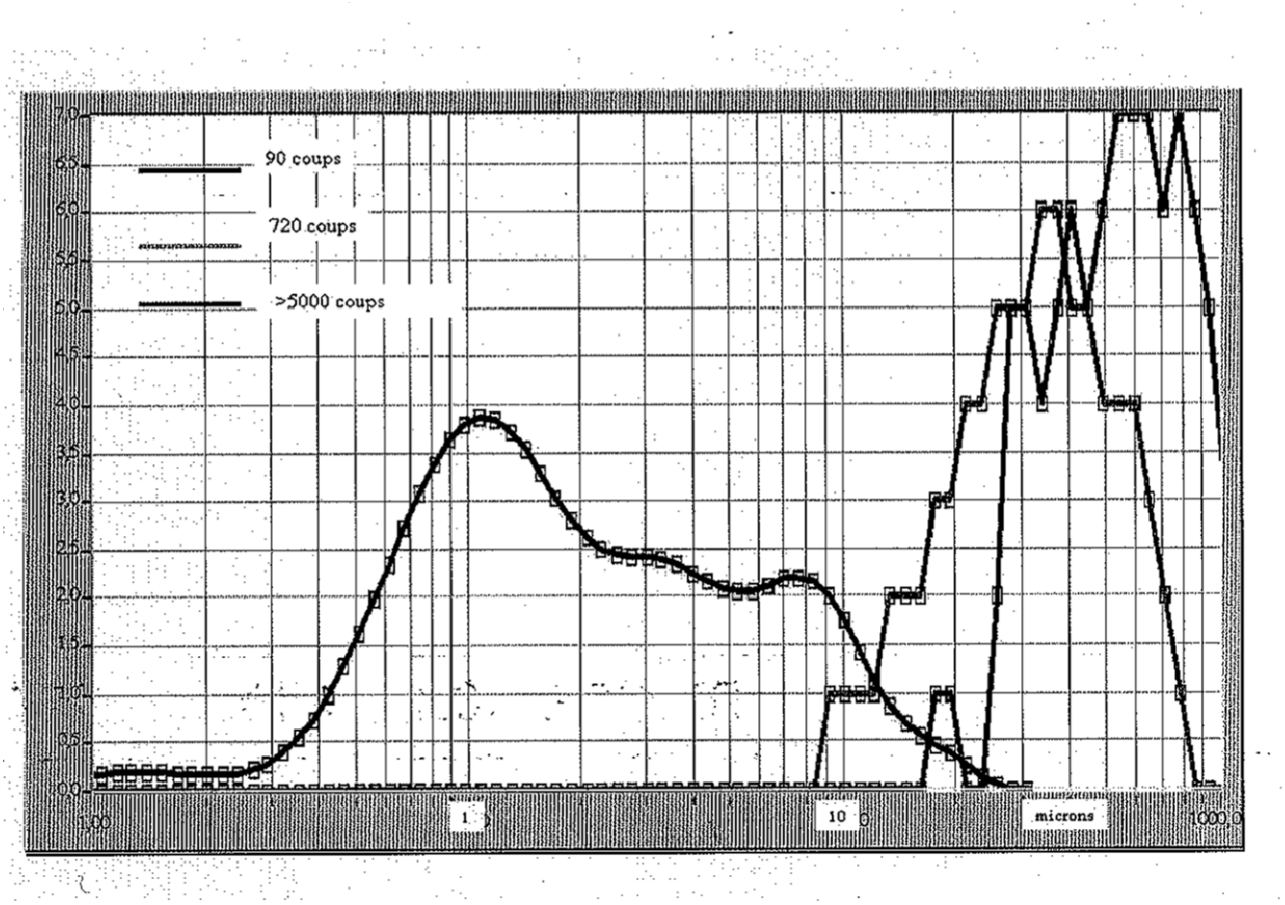

Figure 6: Graphite granulometry resulted from electrical pulse destructuration

After 40 impulsions

Figure 7: Triso-particle destructuration by electrical pulses (CEA tests)

Table 1: Fine/ultra fine grinding machine performances

Grinding machines	Granulometry (*) Inlet (mm)	outlet (µM)	Maximal capacity (t/h)	Power (kW)
Crushing rods	40	300	300	5 to 1500
Crushing roller	25	100	300	5 to 3000
Crushing tubes	25	40	400	5 to 8200
Pebbles crusher	10	100	350	5 to 1800
Semi-autogene crusher	150	100	350	15 to 6000
Autogene crusher	300	200	350	15 to 9600
Vibrating tubes	5	74	4	3,5 to 70
Crushing cylinders	5	500	250	2 to 300
Roller press	5	40	700	220 to 1800
Percussion crusher	20	200	800	6 to 600
Millstone or ball vertical crusher	25	100	60	90 to 900
Pendulous crusher	20	60	120	7,5 to 700
Corundum millstone crusher	0,5	20	0,45	0,75 to 75
Stirring crusher	0,3	1	2	2,5 to 200
Impacting crusher	0,1	20	2	5,5 to 315
Vibrating crusher	0,25	3	-	0,2 to 35
Ball crusher with forced circulation	0,1	5	4	1 to 700
Ball crusher with conical rotor and tank	0,1	10	2	2,2 to 225
Air jet crusher	1	10	3	1 to 500

(*) Approximate values that may vary depending on the material nature, grinding duration

Table 2: Compact/fuel separation methods constraints

Separation Method	Required Pre-treatment	Sorting out of particles	Dust management	Gas treatment	Nuclear constraint	Other constraints
Grinding	→	X	X		dust spreading	abrasion
Electrical pulses	Grinding?	X				
Ultra sound	Grinding?	X				
Burning	Grinding		X	X	gas + dust spreading	C and SiC residues
Halogen gas	Grinding and burning		X	X	gas + dust spreading	corrosion
Molten salts	Grinding			X	gas emission	corrosion

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