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REPROCESSING OF NUCLEAR FUELS BY VOLATILITY SEPARATIONS IN FLUIDIZED BEDS

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BROOKHAVEN NATIONAL LABORATORY

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Introduction

The use of fluidized beds of inert granular material as an effective means of temperature control in the volatility reprocessing of nuclear reactor fuels has received increasing attention in recent years.¹ The unique advantage of the fluidized bed is that it is an efficient, non-corrosive, heat transfer medium for carrying out highly exothermic gas-solid reactions. Since 1958 a continuing experimental study has been conducted at Brookhaven National Laboratory (BNL) in which the basic steps of the fluidized bed volatility process have been developed for a number of unirradiated fuel types. Generally, two major operations are required:

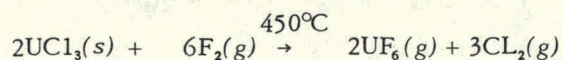
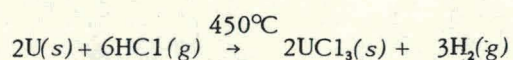
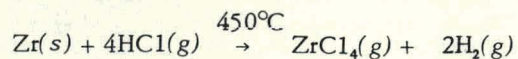
- 1) A head-end reaction at elevated temperatures in which the fuel cladding or matrix material is disintegrated or volatilized.
- 2) A reaction with fluorine at temperatures as high as 550°C in which the fissile material is recovered as volatile hexafluoride.

This report summarizes the results of the BNL fluidized bed volatility studies.

Uranium and Zirconium-Uranium Alloy Fuels

In these fluidized bed studies the first highly exothermic reaction carried out was that between fluorine and metallic uranium coupons.² These early experiments showed that relatively massive pieces of uranium could be rapidly reacted with fluorine gas to form volatile UF₆ with close temperature control, whereas without the fluidized bed such a reaction would produce large temperature excursions.

Further studies, also reported by Reilly *et al.*,² demonstrated that uranium could be recovered from Zircaloy clad Zr-U alloy fuel using a de-clad-fluorination sequence. The primary reactions are as follows:



The fuel was immersed in a fluidized bed of alumina and reacted with gaseous HCl to convert the zirconium constituent of the cladding and alloy to volatile ZrCl₄. The uranium, at the same time, was converted to nonvolatile UCl₃, which was then reacted with fluorine and recovered as UF₆. In other experiments it was demonstrated that ZrCl₄ could be converted to ZrO₂ with steam in a separate fluidized-bed reactor. Hydrochlorination experiments, carried out in a 6-in.-diameter fluidized-bed reactor with PWR-type multiplate fuel assemblies, proved that the alumina bed material could be fluidized between the plates (<0.1 in. spacing) of these assemblies to remove the heat of the de-cladding reaction, thereby preventing melt-down of the fuel. Results of these runs and of the uranium fluorination studies were reported by Regan *et al.*³ Based on these findings an extensive program was initiated at Argonne National Laboratory to carry out the process development through the integrated pilot plant stage.^{4,5}

Graphite Matrix Fuels

As in the case of Zr-U alloy fuels, graphite matrix fuels lend themselves readily to the fluidized-bed volatility method of reprocessing. While a number of other methods have been considered for reprocessing graphite matrix fuels (such as grinding the fuel followed by acid leaching of the uranium,⁶ or high pressure aqueous chemical combustion⁷), burning of the graphite material with oxygen remains the most straightforward and efficient method of exposing the contained uranium for subsequent recovery by fluorination or acid leaching.

In direct burning experiments, without the addition of any inert bed material,⁸ it was found that the high heat fluxes generated, coupled with poor thermal conductivity, resulted in high fuel centerline temperatures, sintering of the product, and excessive local temperatures at the vessel walls.

In contrast, rapid combustion of the fuel in a fluidized bed of alumina with pure oxygen used as the fluidizing gas was carried out with good

temperature control at approximately 750°C.⁹ For example, a ΔT , centerline of the bed to the reactor wall, of only 30°C was experienced in a 4-in.-diameter vessel in which graphite matrix fuel was being burned at a rate of 15 kg/hr-ft² reactor cross section. The heat transfer coefficient, bed to wall, was calculated to be ≈ 70 Btu/hr-ft²-°F.

Bench-scale experiments were carried out to determine uranium recovery from unirradiated graphite matrix fuels using the fluidized-bed oxidation-fluorination steps. On the basis of

the favorable results obtained in these tests,¹⁰ a pilot plant was constructed to carry out uranium recovery experiments with unirradiated graphite matrix fuels on a more comprehensive scale.⁹ Fig. 1 shows the pilot plant flow sheet for the reprocessing studies on the graphite matrix fuel typical of that being tested in the Rover nuclear rocket program.¹¹ The Rover fuel used contains pyrographite-coated uranium dicarbide particles dispersed in a graphite matrix. Propellant passages through the fuel element are lined with niobium carbide to protect the graphite against the corrosiveness of high temperature hydrogen.

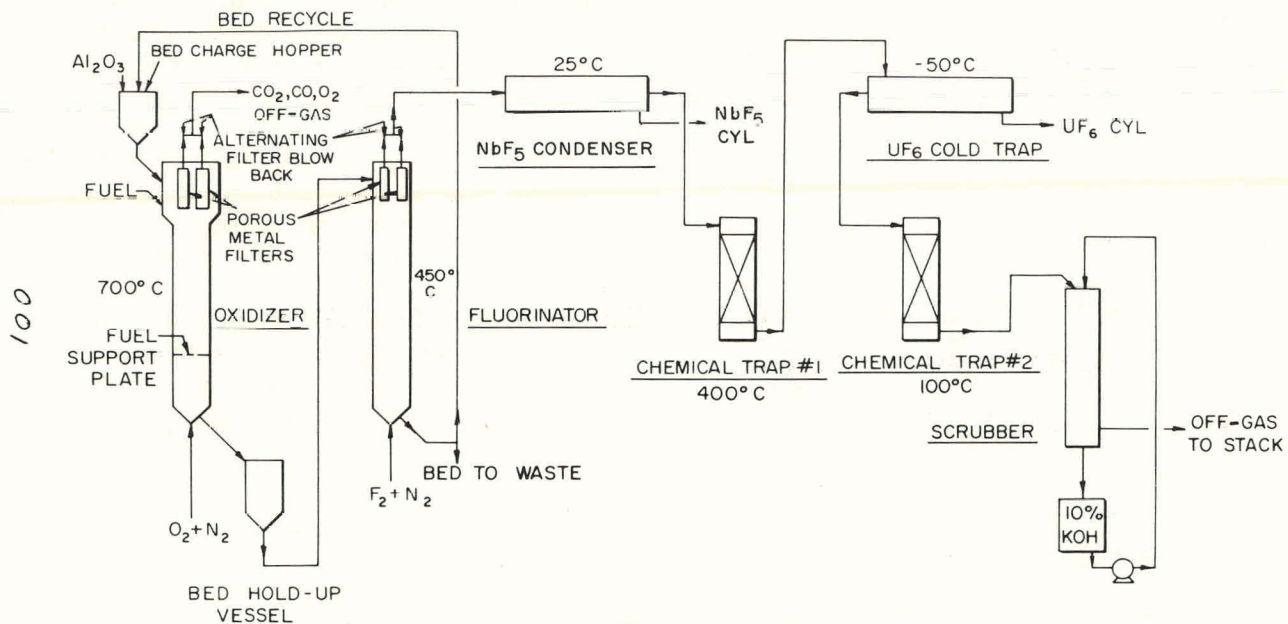
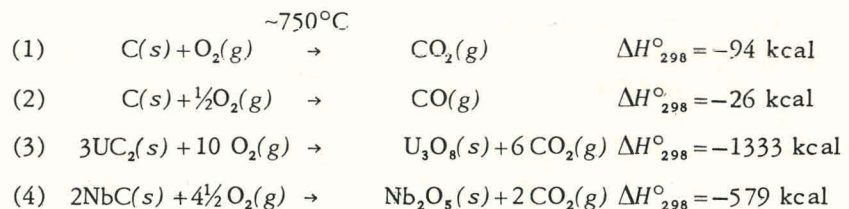
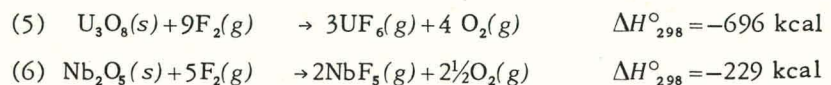


Figure 1. Continuous oxidation-batch fluorination pilot plant flow sheet.

The major reactions taking place in the oxidation step are:



Uranium was recovered from the bed in a fluoride volatility step in which the following major reactions occur:



The oxidation was carried out in a continuous manner in a 4-in.-diameter, 12-ft.-long reactor vessel. The granular alumina bed was fluidized to a 6-ft length with oxygen which was fed through a ball check in the conical bottom of the reactor. Chopped fuel was added intermittently to the fluidized bed as required to maintain a high burning rate. Combustion gases, mainly CO_2 , with small quantities of CO , N_2 and O_2 , were passed through porous metal filters and then to the stack. Plugging of filters was prevented by means of a pulsed blowback with nitrogen. The reactor was air cooled to remove the heat of reaction, and the temperature was maintained between 700° and 750°C . During the course of the reaction, bed material, rich in U_3O_8 and Nb_2O_5 oxidation products and essentially free of carbon, was withdrawn periodically from the bottom of the reactor while recycled bed material from the fluorinator, or makeup alumina, was added at the top.

Bed material discharged from the oxidizer was pneumatically conveyed to a 4-in.-diameter vessel where it was reacted with fluorine gas at 450°C in a batch operation. The off-gases from the fluorinator, which contained UF_6 , NbF_5 , F_2 , N_2 , and O_2 , were passed through porous metal filters and conducted to the uranium recovery system which consisted of four units. These were: 1) an NbF_5 condenser, 2) a high temperature NaF column which served as an NbF_5 cleanup trap, 3) a UF_6 desublimator, and 4) a low temperature NaF column for final UF_6 recovery. The off-gases were finally passed through a spray tower where they were contacted with a 10% KOH solution to remove unreacted fluorine from the stream.

Results of the continuous oxidation of the Rover fuel are shown in Fig. 2. High burning rates, averaging 1.2 to 1.3 kg of carbon/hr [14 to 15(kg C)/hr-ft² reactor cross section], were maintained during periods of extended operation. Oxygen utilization consistently was greater than 95% and the off-gas contained, generally, 80 to 90 vol % CO_2 and 5 to 10 vol % CO .

Five fluidized-bed fluorination experiments were carried out. The bed material contained up to 30 wt % combined U_3O_8 and Nb_2O_5 and, with one exception, <0.3 wt % carbon. Over-all uranium recovery for the five experiments was 99.2%. Temperatures during fluorination ranged from 450° to 510°C . Separation of UF_6 from the NbF_5 was satisfactory.

The pilot plant was also operated with the

graphite matrix fuel used in the high temperature gas-cooled reactor (HTGR).⁹ This fuel is composed of pyrocarbon-coated uranium-thorium dicarbide particles dispersed in a graphite matrix. Oxidation of this fuel proceeded smoothly as with the Rover fuel but at a somewhat lower rate. The fluorination step, however, was more complex. With Rover fuel all of the uranium becomes exposed as the carbon is removed and can be volatilized as UF_6 . However, with HTGR fuel, a significant portion of the uranium is trapped as U_3O_8 within the solid thorium fluoride (ThF_4) formed by fluorination of the ThO_2 oxidation product. In order to recover this uranium by fluorination, the ThF_4 solid must be broken up. The action of high temperature steam, which serves to reconvert the ThF_4 to ThO_2 , has been found effective in this regard.

A series of bench-scale experiments⁹ was carried out in which HTGR fuel was first oxidized in a fluidized bed of alumina and was then fluorinated. Fig. 3 shows the results of the fluorination experiments. The bed initially contained 20,000 ppm of uranium and 80,000 ppm of thorium. In a 150-minute fluorination the uranium concentration was reduced to approximately 1000 ppm. An additional 130 minutes of fluorination with 100% F_2 at 550°C resulted in essentially no further reduction in uranium concentration. At this point the bed was reacted with a mixture of 80%

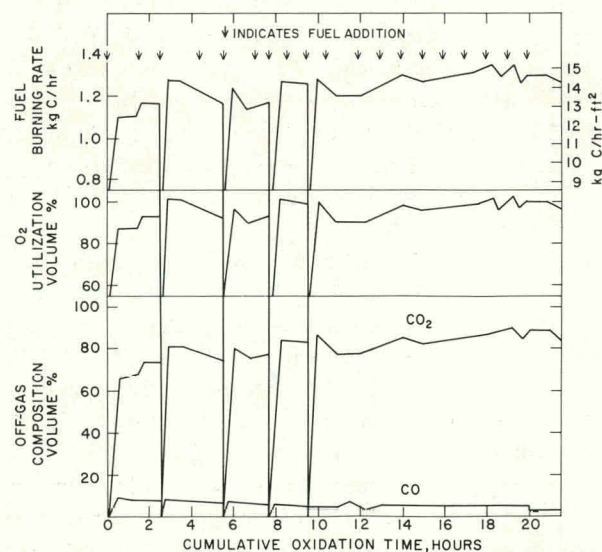


Figure 2. Continuous fluid-bed oxidation of Rover fuel.

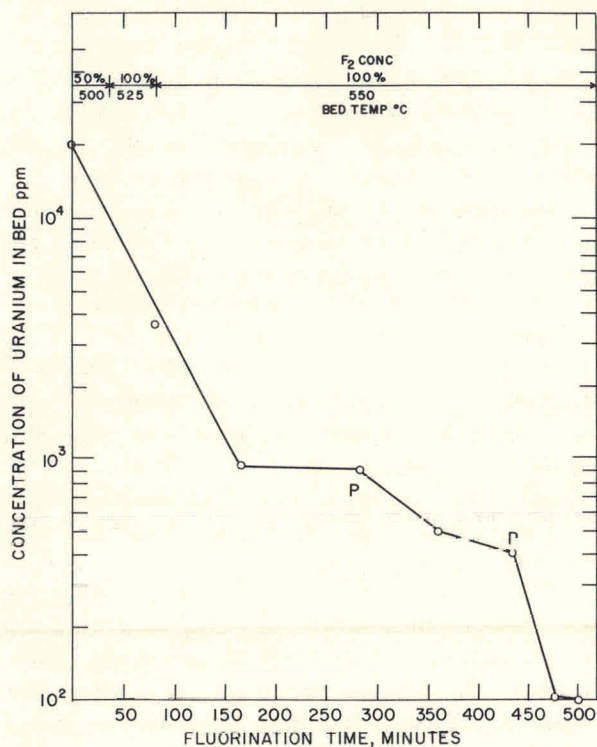


Figure 3. Recovery of uranium from high temperature gas-cooled reactor fuel oxidation products. The points labeled P indicate interruption for pyrohydrolysis step.

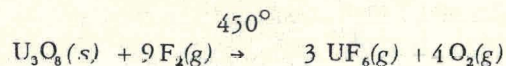
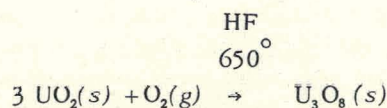
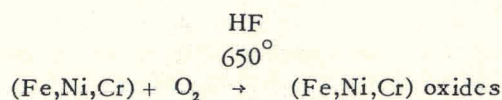
steam-20% nitrogen at a temperature of 500°C for 2 hours. After this pyrohydrolysis step, fluorination was resumed. The cycle was repeated once more and, after a total of 500 minutes of fluorination, the residual concentration of uranium in the bed was found to be approximately 100 ppm. This is equivalent to 99.5% uranium volatilization from the bed.

An alternative method for reprocessing graphite matrix fuels, i.e., by means of burning in a fluidized bed and leaching the oxidation product with acid, was studied with both Rover and HTGR fuel. The head-end fluidized-bed oxidation step was identical with that used in the burn-fluorination studies reported above. With Rover fuel, bench-scale and pilot plant scale leaching experiments, carried out after fluidized-bed oxidation, indicated that an oxidation-leach process for recovering the uranium is feasible.^{12,13} With HTGR fuel, the ThO_2 formed by oxidation of the ThC_2 must be dissolved since a substantial por-

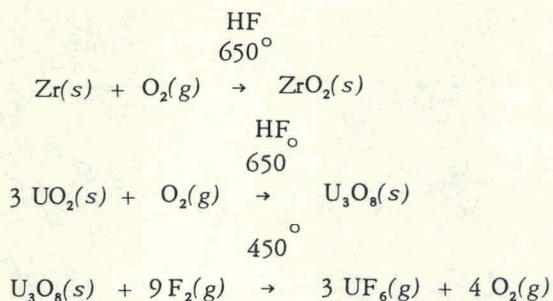
tion of the uranium is trapped in the solid ThO_2 . Conventional Thorex reagent, 13 M HNO_3 -0.04 M HF, may be used to dissolve this material. Results of leaching tests carried out with oxidation product obtained in the burning of HTGR fuel in the fluidized bed of alumina^{14,15} showed that >99% of the uranium may be recovered by these means.

Clad UO_2 Power Reactor Fuels

Other fuel types studied were the stainless steel clad and Zircaloy clad UO_2 power reactor fuels. With stainless steel clad fuels it does not appear practical to consider removal of the cladding by volatilization. However, the stainless steel cladding has been found to be subject to rapid attack and disintegration by means of a fluoride-catalyzed reaction with oxygen. Originally, metal fluorides¹⁶ such as FeF_3 and AlF_3 were used as the catalyst; however, gaseous HF was found to be more effective.¹⁷ The end products of the reaction of either FeF_3 - O_2 or HF - O_2 with the stainless steel in an alumina fluidized bed were found to be oxides of the metals. In the HF-catalyzed oxidation, the UO_2 oxidizes to form U_3O_8 which then may be reacted with fluorine to form volatile uranium hexafluoride. The iron, nickel, and chromium oxides react with the fluorine to form products which, with the exception of some chromium fluorides and oxyfluorides, are non-volatile. The major reactions occurring are shown below:



Zircaloy clad UO_2 fuels may be reacted in the same manner as the stainless steel clad fuels.¹⁴ ZrO_2 is formed during the HF-catalyzed oxidation. The major reactions occurring are as follows:



The effect of variation in feed gas composition on reaction rate (measured by weight loss of the metal coupons, and expressed as penetration) was determined for the HF-catalyzed oxidation of type 348 stainless steel (Fig. 4), and Zircaloy-2 (Fig. 5). The curves are similar for both cladding materials. Reaction rate rose rapidly as the HF fraction in the HF-O₂ feed gas was increased, and reached a maximum at 40% HF. With still higher HF fractions, the rate decreased. Experiments were carried out to determine the effect of temperature on reaction rate for both stainless steel and Zircaloy-2 using the optimum de-cladding mixtures of 40% HF-60% oxygen, and the results are presented in Fig. 6. At temperatures below 500°C essentially no reaction took place. Above 500°C the penetration rate rose rapidly with increasing temperature. With stainless steel, a maximum rate of ~60 mils/hr was reached at a temperature of 650°C above which a gradual decrease in rate occurred. The curve for Zircaloy follows that of stainless steel very closely to a temperature level of 630°C, above which it shows

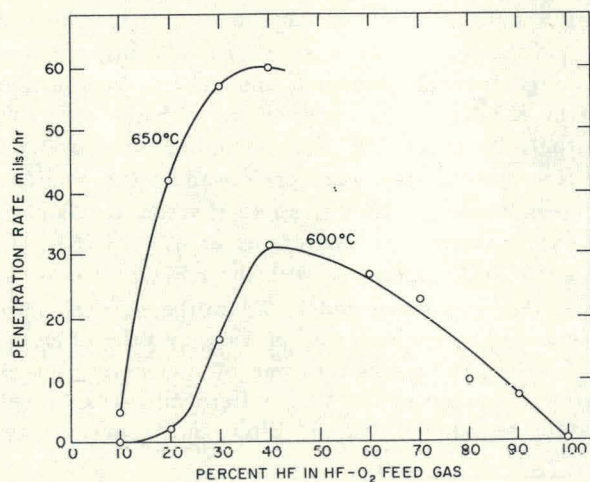


Figure 4. Penetration rate vs feed gas composition. Type 348 stainless steel. Reaction in fluid bed of 60 to 100 mesh Alundum.

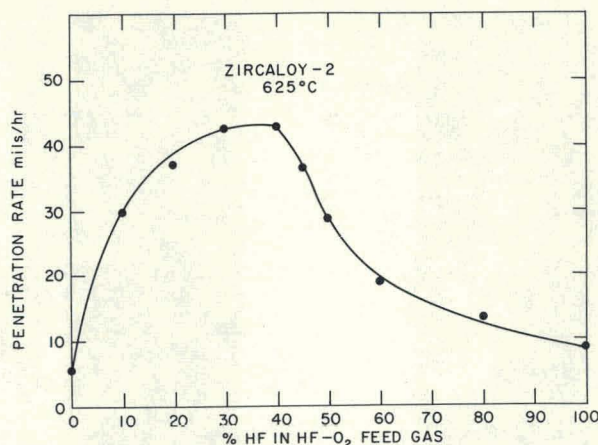


Figure 5. Penetration rate vs feed gas composition. Reaction in fluid bed of 60 to 100 mesh Alundum.

that the reaction rate decreases rapidly. The inverse temperature effect displayed with both these cladding materials in the high temperature region may be considered to be of advantage in the reprocessing of large multirod fuel assemblies in that excessive temperature excursions, which lead to bed caking, may be prevented.

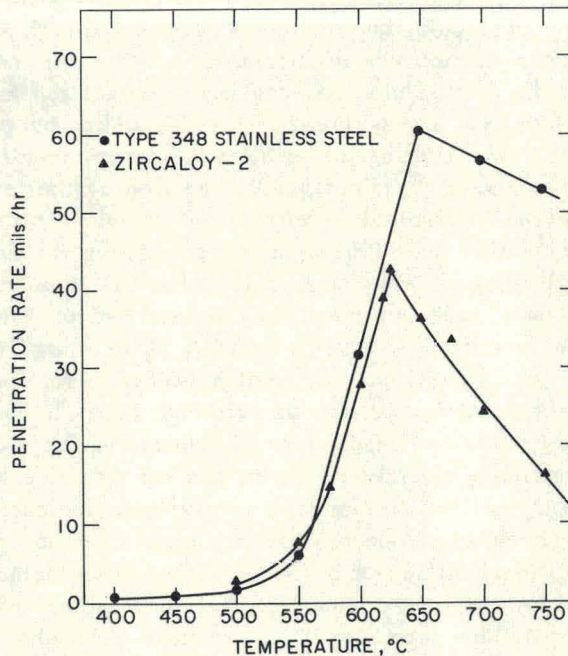


Figure 6. Penetration rate vs temperature. 40% HF-60% O₂. Reaction in fluid bed of 60 to 100 mesh Alundum.

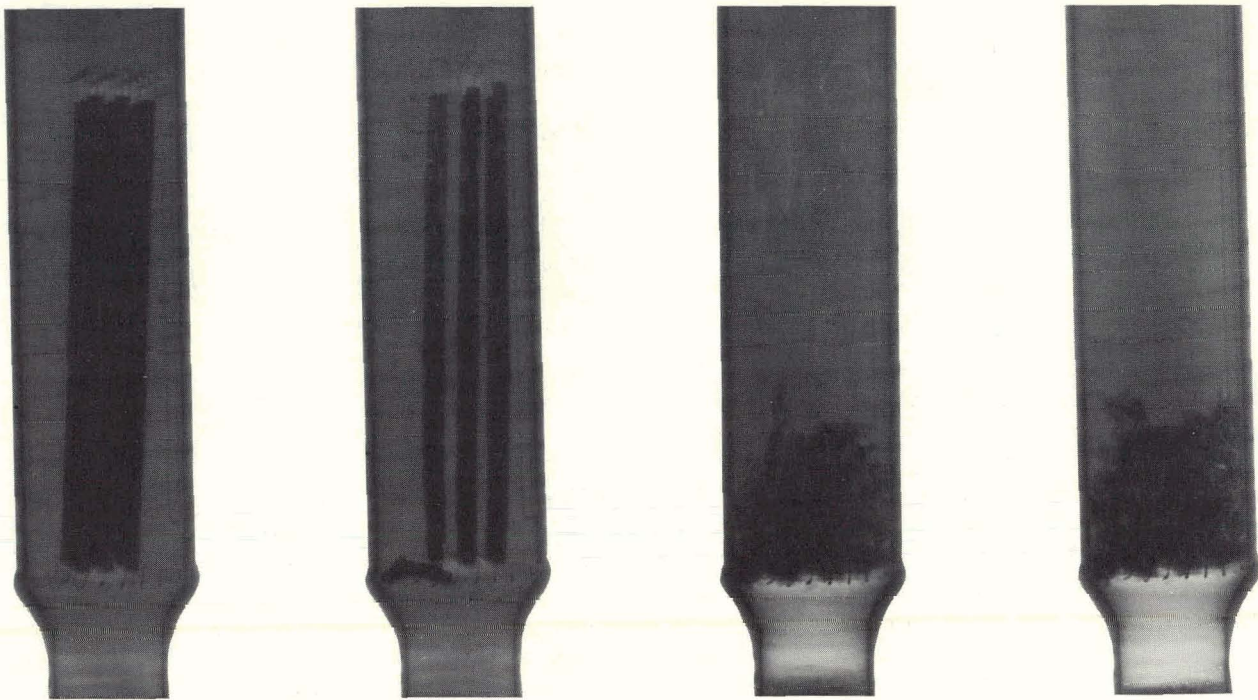


Figure 7. Decladding of 9-rod Zircaloy clad UO_2 fuel assembly with $\text{HF}-\text{O}_2$ in a fluidized bed of alumina.

Decladding and uranium recovery studies have been carried out in laboratory scale equipment using both single rod and multiple fuel rod assemblies. The progress of the decladding operation with multirod assemblies has been observed using gamma radiography as reported by Bartlett *et al.*¹⁶ for 9-rod assemblies of stainless steel clad UO_2 fuel. Radiographs made during the decladding of a 9-rod Zircaloy clad UO_2 fuel assembly in a 3-in.-diameter fluidized bed of alumina at 625°C with 40% $\text{HF}-60\%$ O_2 are shown in Fig. 7. The first view shows the fuel before reaction. After 30 min of oxidation (View 2) the assembly had begun to swell because of the formation of a thick ZrO_2 layer, and the presence of UO_2 pellets on the fuel support grid indicates that at least one rod had ruptured. After an additional 30 min of reaction (View 3) the decladding was almost complete. The last radiograph, made after a total of 90 min reaction time, shows that the assembly had been completely disintegrated.

After completion of the studies on graphite matrix fuels, the BNL pilot plant was modified

in order to carry out larger scale experiments with stainless steel clad and Zircaloy clad UO_2 fuels.^{18,19} Fig. 8 shows a view of one of the assemblies, a 16-rod cluster made up with 15-in.-long "Yankee"-type fuel rods arranged in a 4×4 array. The assembly contained 2.5 kg UO_2 and 0.8 kg stainless steel. Fig. 9 shows a flow diagram for the process, and Table 1 summarizes the results of the experiments with the clad UO_2 fuel. Both the $\text{HF}-\text{O}_2$ decladding step and the fluorination step were performed in the modified 4-in.-diameter fluidized-bed reactor. Decladding experiments were carried out at 600° to 650°C as a two stage operation with the HF/O_2 ratio 40/60 in the first stage and 25/75 in the second. This provided for a high rate of attack on the cladding (in the first stage) followed by a moderate attack with breakup of the UO_2 pellets into U_3O_8 fines* (in the second stage). Fluidization quality and

* U_3O_8 fines are fluorinated readily and with little chance of bed caking whereas larger pieces of the $\text{UO}_2\text{F}_2-\text{UF}_4$ reaction product, which form when decladding is carried out with the 40% $\text{HF}-60\%$ O_2 mixture, do not.

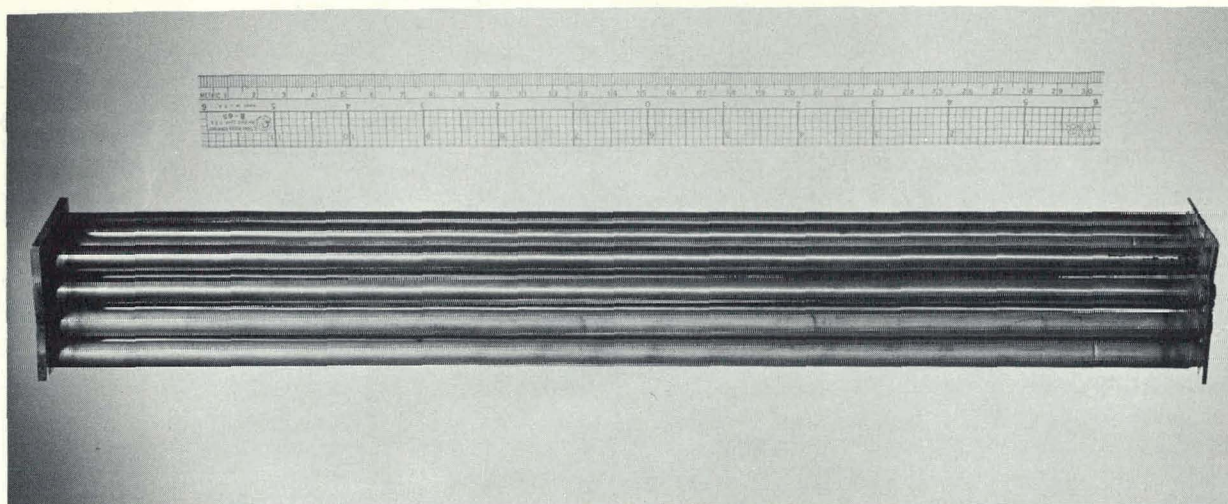


Figure 8. 16-rod "Yankee"-type fuel assembly used in pilot plant decladding fluorination studies.

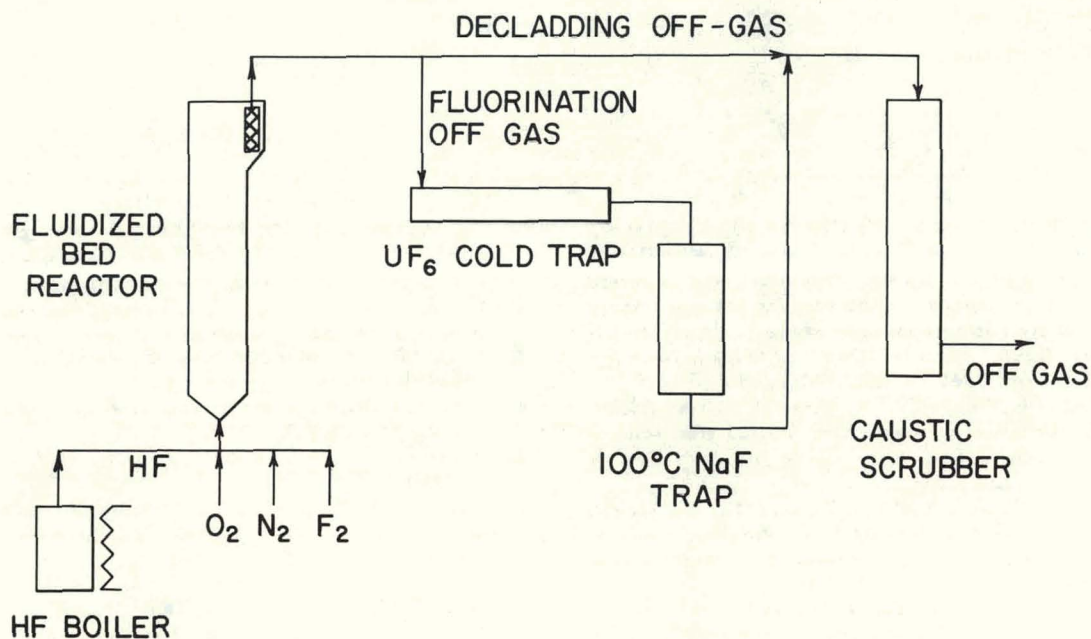


Figure 9. Simplified pilot plant flow diagram for decladding-fluorination of clad power reactor fuels.

temperature control were good during the decladding of both stainless steel clad and Zircaloy clad UO_2 fuel assemblies. The oxidation products were readily reacted at temperatures of 450° to 500°C with fluorine-nitrogen mixtures containing 10 to 50% F_2 , and approximately 99% of the uranium was volatilized from the bed.

Presently, a 6-in.-diameter reactor vessel is being installed in the pilot plant to be used in

decladding experiments with stainless steel clad and Zircaloy clad UO_2 fuel assemblies made up with as many as 49 rods (7×7 array), 30 in. long.

These BNL pilot plant decladding and fluorination studies are being carried out with unirradiated fuel to obtain basic engineering information to be used in support of the ORNL hot pilot plant program.²⁰

Table 1
PILOT PLANT RUN SUMMARY

Run	Yankee Fuel				Zircaloy-2 Fuel
	SS-1	SS-2	SS-3	SS-4	Zr-1
Fuel	9-rod 15-in. long	9-rod 30-in. long	16-rod 15-in. long	25-rod 15-in. long	9-rod 30-in. long
Contained UO ₂	1.410 kg	2.930 kg	2.509 kg	3.9 kg	3.357 kg
Total stainless steel or Zircaloy-2	0.440 kg	0.838 kg	0.792 kg	1.2 kg	0.916 kg
Total wt of fuel	1.850 kg	3.768 kg	3.301 kg	5.1 kg	4.273 kg
Alumina charge	18.2 kg	23.6 kg	18.2 kg	18.2 kg	23.6 kg
U/Al ₂ O ₃ ratio	0.068	0.110	0.121	0.189	0.125
SS Fluoride/Al ₂ O ₃ ratio	0.044	0.064	0.079		0.062
HF-oxidation					
40% HF-60% O ₂	39 min	1 hr	1 hr 15 min	35 min	30 min
25% HF-75% O ₂	1 hr 51 min	3 hr 20 min	3 hr 28 min	1 hr	2 hr 30 min
Total	2 hr 30 min	4 hr 20 min	4 hr 43 min	1 hr 35 min	3 hr
Fluorination					
25% F ₂ -75% N ₂		2 hr 20 min	2 hr 20 min		2 hr 28 min
50% F ₂ -50% N ₂		1 hr 5 min	1 hr 0 min		30 min
Total time	2 hr 20 min	3 hr 25 min	3 hr 20 min		2 hr 58 min
Total F ₂ charge	6 lb	10 lb	10 lb		7.5 lb
U in bed after fluorination	0.025%	0.107%	0.16%		0.17%

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07A CHEMICAL ENGINEERING	8G GEOLOGY	04A PHYSICS (ATMOSPHERIC)	18I REACTOR SAFETY
07 CHEMISTRY (ANALYTICAL)	08 GEOPHYSICS	20M PHYSICS (LOW-TEMPERATURE)	18K REACTOR THEORY
07B CHEMISTRY (INORGANIC)	6R HEALTH AND SAFETY	20H PHYSICS (PARTICLE)	18L REACTORS--POWER
07C CHEMISTRY (ORGANIC)	20M HEAT TRANSFER	20I PHYSICS (PLASMA)	18M REACTORS--PRODUCTION
07D CHEMISTRY (PHYSICAL)	08H HYDROLOGY	20L PHYSICS (SOLID-STATE)	18M REACTORS--RESEARCH
07E CHEMISTRY (RADIO- AND RADIATION)	18D INSTRUMENTATION	18C PLOWSHARE	11B REFRACTORIES
09B COMPUTERS	18B ISOTOPES	PROGRESS REPORTS	08K SEISMIC PHENOMENA
11F CORROSION	14B LABORATORIES AND TEST FACILITIES	21 PROPULSION	07A SEPARATION
20H COSMIC RADIATION	20I MAGNETOFLUIDDYNAMICS	06Q PROTECTIVE EQUIPMENT	18N SNAP TECHNOLOGY
18H CRITICALITY STUDIES	11 MATERIALS	08 RADIATION BELTS	22 SPACE TECHNOLOGY
20B CRYSTALLOGRAPHY	12 MATHEMATICS	RADIATION EFFECTS	13M STRUCTURES
07A DECONTAMINATION	MEETINGS	18F RADIATION SHIELDING AND PROTECTION	18 TRACER STUDIES
06R DOSIMETRY	11F METALLURGY	18G RADIOACTIVE WASTES	TRANSLATIONS
06F ECOLOGY	04B METEOROLOGY	06R RADIOBIOLOGY	19 WEAPONRY
09 ELECTRONICS AND ELECTRICAL ENGINEERING			