329
7-24-66

MASTER

FLUIDIZED-BED REPROCESSING OF STAINLESS STEEL CLAD UO₂ POWER REACTOR FUELS

C. B. BARTLETT, E. WIRSING, JR., S. J. WACHTEL,
R. JOHNSON, J. J. REILLY, AND L. P. HATCH

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

November 1965

BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.

under contract with the
UNITED STATES ATOMIC ENERGY COMMISSION

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

CFSTI PRICES

H.C. \$ 1.00; MN .50

FLUIDIZED BED REPROCESSING OF STAINLESS STEEL CLAD UO₂ POWER REACTOR FUELS

C. B. BARTLETT, E. WIRSING, JR., S. J. WACHTEL,
R. JOHNSON, J. J. REILLY, AND L. P. HATCH

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

November 1965

BROOKHAVEN NATIONAL LABORATORY
UPTON, NEW YORK 11973

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

PRINTED IN USA

PRICE \$1.00

Available from the
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards
U.S. Department of Commerce
Springfield, Virginia

June 1966

700 copies

ABSTRACT

A fluidized bed volatility process for the recovery of uranium from stainless steel clad UO_2 reactor fuels has been developed at Brookhaven National Laboratory. The process involves two steps:

- 1) A head-end step in which the cladding is removed from the fissile material by reaction with a mixture of HF and O_2 at 600°C .
- 2) A recovery step whereby the exposed uranium is converted to volatile UF_6 by reaction with fluorine at 450°C to 500°C .

Average penetration rates in excess of 60 mils per hour have been achieved in the decladding step and uranium recoveries have been greater than 99%.

FLUIDIZED BED REPROCESSING OF STAINLESS STEEL CLAD UO₂ POWER REACTOR FUELS

INTRODUCTION

The use of fluidized beds of inert granular material to control highly exothermic reactions between gases and massive solids has formed the basis for the development of a number of volatility separation processes¹⁻³ for the recovery of uranium and plutonium from spent nuclear reactor fuels of various types. These processes generally involve two steps:

- 1) A head-end step in which the fuel cladding or the matrix material is removed by volatilization.
- 2) A recovery step in which the exposed U and Pu are converted to their volatile hexafluorides.

The development of a head-end step suitable for decladding stainless steel clad fuels has presented unusual problems, however, since the major constituents of stainless steel do not form volatile compounds except at excessively high temperatures and under highly adverse conditions with respect to the corrosion resistance of process equipment. Moreover, the high temperatures have been found to promote undesirable side reactions between the reactant gas chlorine and uranium.

In the course of development studies on fluidized bed volatility processes at Brookhaven National Laboratory, it was found that rapid oxidation and subsequent disintegration of stainless steel coupons occurred on exposure to oxygen at high temperatures (~700°C) in a fluidized bed of granular alumina containing small quantities (~1%) of aluminum fluoride fines. The attack was characterized by the progressive formation of thick layers of oxide scale (Figure 1) composed primarily of α -Fe₂O₃. It was apparent that this fluorine-promoted oxidation provided a potentially promising means for decladding stainless steel clad fuels. This finding led to a broadening of the study to determine the "catalytic" effects of other metal fluorides, i.e., the fluorides of Al, Fe, Co, Ni, Cr, Na, and Mg. Of these, only Co, Fe, and Al fluorides were found to produce attack at significant rates.

At this point in the development, investigations at ORNL⁴ revealed that the same type of "catastrophic oxidation" of stainless steel could be produced by substituting gaseous hydrogen fluoride for the metal fluoride fines. Accordingly, the original approach was modified and it was found that, with the HF-O₂ gas mixture, higher reaction rates with stainless steel could be obtained and that lower reaction temperatures could be used than with the metal fluoride-oxygen system. In addition, use of the gaseous form of fluoride greatly simplified the problems of introducing the catalyst and maintaining its concentration at selected levels. This paper is concerned primarily with the results of bench-scale experiments on the application of the hydrogen fluoride-promoted oxidation reaction in the decladding of stainless steel clad UO₂ fuel rods in conjunction with the recovery of the U and UF₆. Results of the initial experiments with the metal fluoride system are presented in the Appendix.

DECLADDING STUDIES

Studies on the HF-O₂ reaction with stainless steel were carried out in a 1½-in.-diameter fluidized bed reactor. A schematic diagram of the apparatus is shown in Figure 2. The reactor, constructed of nickel 200, consisted of 1½-in.-diameter by 24-in. reaction section topped by a 3-in. by 18-in. disengaging section. The upper flange of the reactor was fitted with a porous nickel filter (1-in. diameter x 12 in.) having a rated mean pore size of 20 microns. To prevent excessive pressure drop due to the accumulation of fines on the filter, a pulsed nitrogen blowback system was used in which both the pulse frequency and the blowback pressure could be varied.

Figure 1. Catastrophic oxidation of stainless steel in a high temperature oxygen-fluoride environment

The reactant gases (HF-O_2) were introduced at the bottom of the reactor through a conical section which contained a ball check to prevent any backflow of solids. Electrical heating was used to maintain reaction temperatures. Temperatures at the reactor wall and the bed centerline were measured by means of thermocouples, and taps were provided to measure pressure differences across both the bed and the filters.

The off-gases from the reactor were conducted in heated lines to a caustic scrubber which served to neutralize the HF or excess fluorine. In experiments which involved recovery of uranium, the off-gas was first passed through a series of three cold traps in which the uranium was removed from the gas stream. The first trap was made up of a 3-in.-diameter nickel pipe packed with Ni helices and maintained at -60°C with refrigerated trichloroethylene. The other two traps were 3-in.-diameter copper vessels immersed in a mixture of dry-ice and trichloroethylene, maintained at $\sim -90^\circ\text{C}$.

The effects of variations in the HF/O_2 ratio on the oxidation rate of type 348 stainless steel were determined at 600°C in a fluidized bed of 60 mesh Norton-type RR alumina. A rapid rise in rate of attack (Figure 3) was obtained with increasing HF concentration with a maximum rate at 40% HF; beyond this point the rate decreased gradually with higher HF concentrations. Reaction rates were also determined using the

optimum 40% HF–60% O_2 mixture, over the temperature range 500°C to 700°C . The results (Figure 4) show a rapid rise in rate to a maximum (60.5 mils/hr) at 650°C . At temperatures above 700°C , bed caking with loss of fluidization was evident. The caking was subsequently shown to be due to the action of HF on the bed material (Norton grade RR alumina). Experiments with other types of alumina, however, showed that the Norton abrasive grain, "blocky type", could be used with the 40% HF–60% O_2 mixture at

Figure 2. Bench-scale fluidized bed reactor system for stainless steel clad UO_2 fuels

temperatures as high as 800°C without loss of fluidization. The effects of both temperature and gas composition on the rate of oxidation of type 348 stainless steel, using the blocky-type abrasive grain alumina, were comparable to those obtained with the RR alumina (Figure 5).

EXPERIMENTS WITH YANKEE FUEL

Five experiments were carried out on the decladding of 15-in. lengths of Yankee fuel rods. These fuel rods consisted of .340-in.-o.d. x .021-in. wall, type 348 stainless steel tubing containing sintered UO_2 pellets (.5-in. x .295-in. diam). Using the 40% HF-60% O_2 gas mixture at 600°C, it was found that complete disintegration of the fuel rod had taken place within a 2-hr reaction period. Examination of the bed after each run showed that it contained Fe_2O_3 scale and uranium reaction products, identified by x-ray diffraction analysis as being composed of a mixture of U_3O_8 , UO_2F_2 , and UF_4 . Details of uranium recovery experiments are presented in a following section.

Figure 3. Penetration rate of type 348 stainless steel vs reactant gas composition at 600°C in a fluidized bed of 60 mesh type RR alumina

Figure 4. Penetration rate of type 348 stainless steel vs temperature with a 40% HF-60% O_2 reactant gas mixture in a fluidized bed of 60 mesh type RR alumina

Experiments were then carried out in a 3-in.-diameter fluidized bed reactor using small simulated fuel rod assemblies. A typical assembly, shown in Figure 6, was made up of nine, 15-in.-long sections of Yankee fuel rods welded to 1/8-in. type 304 stainless steel end plates. The rods were spaced in square array, 0.422-in. on centers, as in Yankee Core I. The total weight of UO_2 in these assemblies was approximately 1.5 kg.

The oxidation step was carried out at 625°C using the 40% HF - 60% O_2 mixture, and the assembly was completely disintegrated in a 4-hr reaction period. In a similar experiment, gamma radiography was used to determine the extent of disintegration of the assembly at intervals during the course of the reaction. An Ir^{192} gamma source was positioned adjacent to the reactor at the level of the fuel support grid, and 5-min film exposures were made. The radiographs are presented in Figure 7. Figure 7-A, taken after 1 hr 27 min of reaction time, shows a marked distortion of the rod near the lower end. Figure 7-B, taken after 1 hr 52 min, indicates that the lower end plate and part of the upper section of the assembly were broken up and that several rods were resting independently on the support grid, with one rod actually passing through the grid and into the conical bottom.

Figure 5. Effect of temperature and gas composition on penetration rate of type 348 stainless steel in a fluidized bed of 60 mesh Norlon blocky-type alumina

Figure 6. Stainless steel clad-UO₂ fuel rod assembly

Figure 7. Progressive oxidation of stainless steel clad- UO_2 fuel rod assembly during reaction with the HF-O_2 gas mixture

- A After 1 hr 27 min.
- B After 1 hr 52 min.
- C After 2 hr 40 min.
- D After 3 hr 20 min.

An accumulation of U reaction products is shown on the support grid. Figure 7-C, taken after 2 hr 40 min, shows that disintegration of the assembly was nearly complete but that a section on one fuel rod remained almost intact. Figure 7-D, made after 3 hr 20 min, shows that complete disintegration of the assembly and of the individual fuel rods had taken place.

The accumulation of U and stainless steel reaction products on the grid was estimated to be ~ 5 in. deep. Previous experience had demonstrated that reaction of such accumulations, or the uranium compounds therein, with fluorine results in high localized temperatures and bed caking. Therefore, the decladding step must be carried out under conditions which will not only give acceptable reaction rates but will also produce a more finely divided uranium product.

To determine the conditions which would result in maximum disintegration of the uranium

reaction products, a series of boat experiments was performed in which single pellets of UO_2 were exposed to HF-O_2 over a range of concentrations and temperatures. The results, presented in Table 1, show that high HF concentrations (40 to 50%) gave the coarsest U product. The chemical composition of the product also varied, U_3O_8 being formed at the lower HF concentrations (20 to 30%), and a mixture of UO_2F_2 and UF_4 at the higher HF concentrations (40%). These results indicated that the reaction of stainless steel clad UO_2 fuel with a 30% $\text{HF-70\% O}_2$ mixture at 650°C would give both a rapid attack on the stainless steel and good dispersion of the uranium reaction products. This tentative conclusion was confirmed in fluid bed experiments wherein sections of Yankee-type fuel were completely disintegrated in a 90-min oxidation period and 80% of the residual bed material was found to be finer than 60 mesh.

Table 1

Particle Size Distribution of Single UO_2 Pellet
Reaction Product After Exposure to HF-O_2 Mixtures

U.S. Mesh	20% $\text{HF-80\% O}_2$ 650°C, 1½ hr wt % of product	20% $\text{HF-80\% O}_2$ 600°C, 2 hr wt % of product	30% $\text{HF-70\% O}_2$ 600°C, 1½ hr wt % of product	40% $\text{HF-60\% O}_2$ 600°C, 1½ hr wt % of product
1 to 8	10	1.2	13	33
8 to 16	2.3	0.3	2.3	12
16 to 40	6.2	1.7	7.4	14
40 to 60	6.4	2.8	6.7	7.0
60 to 120	15	12	13	10
120 to 200	20	27	26	10
Through 200	31	43	21	6.3
Recovery*	91	88	89	92

* With the small quantities involved, low recovery is believed to be due mainly to loss of fines.

URANIUM RECOVERY

A series of five runs was carried out to demonstrate the recovery of uranium from Yankee-type fuel clad by the HF-O_2 method. In each run, 15-in. sections of Yankee fuel rods were first reacted with a 30% $\text{HF-70\% O}_2$ mixture at 650°C in the 1½-in.-diameter fluidized bed reactor and the uranium was volatilized by reaction with F_2 .

The following fluorination procedure was used in each experiment:

- 1) 15 min at 400°C with 20% F_2 -80% N_2 ;
- 2) 20 min at 450°C with 30% F_2 ;
- 3) 40 min at 500°C with 50% F_2 ;
- 4) 30 min at 550°C with 100% F_2 .

This sequence was employed in accordance with the procedure suggested by Argonne National Laboratory for maximizing Pu recovery, although no Pu was present in the BNL experiments.

Final residual uranium concentration in the bed was determined by means of x-ray fluorescence analysis. The results of these experiments, presented in Table 2, show that greater than 99% recovery of uranium, as UF_6 , was achieved.

It has been reported⁶ that the recovery of Pu in yields as high as 99% would require recycling the bed several times. Therefore, the problems associated with the recycling of alumina beds containing gross quantities of reaction products were investigated in conjunction with the determinations of U recovery.

At the end of the five uranium recovery runs, noted above, with reuse of the same bed, 27 wt% stainless steel reaction products had accumulated in the bed. It was noted that the thick Fe_2O_3 scale, as formed during the de-cladding steps, had been converted to fine (~90 mesh) particles of FeF_3 during the fluorination step. No evidence of bed caking was noted

Table 2

Uranium Recovery From Yankee Fuel Rod Sections 1000 g Bed of 100 Mesh Abrasive Grain Alumina. Initial Bed Charge = 1000 g of 100 mesh abrasive grain Al_2O_3 . Residual U in Bed After 5th Cycle = ~ 750 ppm U or 0.96 g U. U Volatilized From Bed in 5 Cycles = 99.9%.

Cycle	UO ₂ charge/cycle (g)	SS charge/cycle (g)	Calculated % SS product in bed	Residual U in bed (ppm)*	U recovery** (%)
1	154.2	41.5	7	200	99.6
2	153.2	41.2	13	320	99.8
3	153.9	41.4	18	225	99.8
4	135.8	36.3	22	250	99.8
5	154.5	40.3	27	750	99.4
Total	751.6	200.7	27	750	99.9

*Determined by x-ray fluorescence analysis.

**Per charge.

during the runs. However, when an additional wt% (120 g) of stainless steel tubing was then charged to the reactor and the cycle repeated, bed caking was observed. Efforts to restore fluidization were unsuccessful and the bed, on removal from the reactor, was found to contain some unreacted stainless steel, iron oxide scale, loosely caked bed material, and smaller pieces of sintered bed material. It was apparent, therefore, that the build-up of stainless steel reaction products in a recycled bed should be limited to 27 wt% when a subsequent high temperature fluorination step was to be employed.

Analysis of the contents of the UF₆ cold traps during the uranium recovery experiments indicated that a substantial quantity of chromium was volatilized during the fluorination step. To

determine the extent of Cr volatilization, a series of five experiments was carried out in which single sections of stainless steel tubing were dechlorinated with the HF-O₂ gas mixture, the same bed being used throughout the series. After each oxidation, the bed was exposed to a 25% F₂-75% Ar mixture for 2 hours at a temperature of 450°C. The results of these experiments, presented in Table 3, show that relatively equal amounts of Cr were volatilized in each of the first three fluorination steps. In cycle No. 4, however, a 6-hr fluorination was used at temperatures ranging from 450°C to 550°C, and a three-fold increase in the quantity of Cr volatilized occurred. A sample of the volatile Cr species was isolated and identified as chromyl fluoride (CrOF₂ subl. 30°C) by x-ray diffraction analysis.

Table 3

Cyclic HF Oxidation-Fluorination of Single Rods of Stainless Steel Type 348 Clad UO_2
in a Fluidized Bed of 60 Mesh Abrasive Grain Alundum

Cycle	SS added (g)	Cr in SS	Net Cr in bed	Cr in UF_6 trap (g)	As % of Cr in bed	Cr volatilized	Fluorination hr °C
						As % of Cr charged in each cycle (%)	
1	45.0	8.1	8.1	1.4	17	17	2 450
2	45.0	8.1	14.8	1.4	9.5	17	2 450
3	41.4	7.5	20.9	1.4	6.7	19	2 450
4	42.5	7.5	27.0	4.5	17	60	6 450 to 550
Total	173.9	31.2		8.7	28		

Stainless steel reaction products in 4th cycle bed = 315 g
(assume Fe as FeF_3 , Ni as NiF_2 , and residual Cr as Cr_2O_3).

Initial charge = 1000 g of 100 mesh abrasive grain Alundum.

Bed loading = $(315 \text{ g}/1000 + 315) = 24\%$ SS reaction products in the bed.

CONCLUSIONS

This study has demonstrated that uranium can be quantitatively recovered from stainless steel clad UO_2 fuels using a two-step fluidized bed process whereby the fuel elements are first de-clad by an HF-promoted oxidation and the U is subsequently recovered as UF_6 by a reaction with elemental fluorine.

The Brookhaven fluidized bed pilot plant, which was designed specifically for experiments with graphite matrix fuels,² has been modified to carry out reprocessing studies on stainless steel clad UO_2 fuels using the fluoride-promoted oxidation technique. The operation of the pilot plant will be based on the results of the bench-scale studies and will be carried out to investigate the decladding step as well as the uranium recovery from assemblies containing as many as 49 Yankee-type fuel rods with lengths up to 30 in.

APPENDIX

As noted in the introduction, it was found that certain metal fluorides produced rapid catastrophic oxidation of stainless steel. Of the metal fluorides tested, only four produced significant rates of attack on type 304 stainless steel, i.e., FeF_3 , AlF_3 , CoF_3 , and CoF_2 . However, with CoF_2 and CoF_3 elemental fluorine was liberated during the reaction, and at the high temperatures (700°C) F_2 would be too highly corrosive even to nickel or high nickel alloys. For this reason the study was narrowed to include only the effects of AlF_3 and FeF_3 .

A series of scouting experiments was carried out to determine the effects of varying fluoride concentrations and temperatures on the reaction rates. To simplify the experimental procedure, the reactions were carried out in small rotating tube reactors, made up, as shown schematically

in Figure 8, of a short section of 1-in.-diameter Inconel tube fitted at each end with a Rigi-mesh filter. The reactor was attached at each end to a concentric $\frac{1}{2}$ -in.-diameter nickel tube which served both to conduct the gas and as a shaft for rotation. With this apparatus, good contact between the reactants could be effected with low gas flow rates and with negligible loss of the fluoride catalyst fines. The results of the experiments, presented in Figure 9, showed that significant attack rates occurred in each case with fluoride concentrations higher than 1.5% wt%. A maximum rate was obtained with AlF_3 at ~ 4 wt%, but the rate dropped off sharply with higher concentrations. With FeF_3 , a maximum

rate was obtained at 6 wt%; however, the rate dropped off more gradually (than before) with higher concentrations. The effect of temperature on penetration rate was also determined and the results of these experiments are noted in Figure 10. The data show that with both FeF_3 and AlF_3 , the maximum rate occurred at 700°C to 730°C . At this point the study was further narrowed to center on the use of FeF_3 only, since it appeared that a wider latitude in fluoride concentration was possible with this system. Experiments were carried out with different types of stainless steel and the results showed that with the FeF_3 catalyst comparable rates of attack were obtained with types 304, 316, 347, and 348 stainless steel.

Figure 8. Rotating capsule used in metal fluoride-stainless steel experiments

Figure 9. Type 304 stainless steel wt loss vs metal fluoride concentration at 600°C in a rotating capsule with 100% O_2

Figure 10. Type 304 stainless steel wt loss vs temperature using 100% O_2 in a rotation capsule containing 4 wt% metal fluoride

The $\text{FeF}_3\text{-O}_2$ system was then investigated in fluidized beds of 60 mesh RR alumina to determine recovery of uranium from sections of unirradiated Yankee fuel rod.

Four experiments were carried out in the fluidized bed unit in which single 15-in. sections of Yankee fuel rod containing 150 g of UO_2 were reacted with 100% oxygen at 750°C in a fluid bed of Norton RR alumina containing 5 w/o FeF_3 for a period of 5 hours. After this decladding step, the uranium-bearing bed was exposed to elemental fluorine (25% in Ar) for a period of 5 to 6 hours at 450°C . The results of these experiments, presented in Table 4, show that >99% of the uranium was volatilized from the bed during the fluorination step.

In parallel experiments, it was determined that the reaction rate decreased with time (Figure 11). This phenomenon was attributed in part to the elutriation of the fluoride catalyst fines from the reaction zone. The high initial rates could be re-established, however, by the addition of fresh catalyst to the bed. Efforts to sustain the reaction rate by using a higher initial catalyst

loading resulted in caking of the bed, i.e., with FeF_3 concentrations higher than 8%. Intermittent injection of fresh catalyst during the course of the reaction also produced caking, presumably as a result of high local concentrations of FeF_3 . It was evident from previous studies that such bed caking would adversely affect uranium recoveries.

Although the fluoride-promoted oxidation method of decladding using metal fluoride fines as the catalyst showed promise for further development, the advantages of using gaseous HF as the catalyst were apparent.

Several short term corrosion tests were carried out in the metal fluoride-oxygen system and the results are presented in Table 5. These data suggest that for such high temperature metal-fluoride oxygen application, Inconel is the preferred material of construction.

Acknowledgments: The authors wish to express their appreciation to Dr. R.H. Wiswall, Jr. for his many valuable suggestions and to Messrs. R.A. Feindt, W.C. Freese, and M.E. Stone for their assistance in carrying out the experiments.

Table 4

Uranium Recovery from Yankee Fuel Rods.

1000 g RR Alumina, 50 g FeF_3 ,100% O_2 at 725°C , 25% F_2 in Ar at 475°C .

Run	U charged (g)	Oxidation Hours	Fluorination Hours	Residual U in bed (ppm)	U recovery (%)	Material balance (%)
1	158	4.75	2	20,000	83	80
2	75	4.5	6.5	300	113	100
3	75	4.25	4.5	294	>99	98
4	75	5	4	277	>99	60

Figure 11. Average stainless steel penetration rate in a fluidized bed as a function of reaction time

Table 5

Short-Term Corrosion Tests

2% FeF₃ in 1000 g Al₂O₃ Bed100% O₂ at 700°C, 2-Hr Test

Run	Specimen Corrosion Rate (mils/hr)		
	Inconel #1-1	Inconel #2	Nickel #1
1	0.5	0.8	0.13
2	0.11	0.16	0.08
3	—	0.005	0.26
4	0.001	0.002	0.32
5	0.005	0.005	0.17
N-1	0.1	0.7	0.48
N-2	0.02	0.01	0.5
N-3	0.01	0.003	1.7
N-4	0.002	0.05	0.4

1% AlF₃ Added to 1000 g Al₂O₃ Bed100% O₂ at 700°C, 2 Hr O₂

Run	Specimen Corrosion Rate (mils/hr)		
	Inconel #1	Nickel #1	Nickel #2
R-1 ^a	0.03	1.0	1.1
R-2	0.17	0.3	0.3
R-3	0.11	0.21	0.21
R-4	0.06	0.55	0.43

^aBed and specimens exposed to 450°C F₂ for 15 min prior to oxidation.

REFERENCES

1. Reilly, J.J., Wachtel, S.J., Johnson, R., Wirsing, E., and Hatch, L.P., *Reprocessing of Reactor Fuels by Volatilization Through the Use of Inert Fluidized Beds*. BNL 663 (classified).
2. Reilly, J.J., Wachtel, S.J., Johnson, R., Wirsing, E., and Hatch, L.P. Fluidized bed reprocessing of graphite matrix nuclear fuel. *Ind. Eng. Chem. Process Design Develop.* 5, 51-9 (1966).
3. Bartlett, C.B., *Progress Report Nuclear Engineering Department*. Pp. 33-44, Sept. 1 - Dec. 21, 1962.
4. *Oak Ridge National Laboratory Status and Progress Report*. ORNL-3164, pp. 12-13, April 1963.
5. *Argonne National Laboratory Chemical Engineering Division Semi-Annual Report*. ANL 6900, pp. 144-54, Jan. - June 1964.
6. *Argonne National Laboratory, Reactor Development Progress Report*. ANL 6977, p. 67, Nov. 1964.