NDA 2145-1

CARBIDE FUEL DEVELOPMENT

Progress Report

Period of September 15, 1959 to January 31, 1960

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FOREWORD

The Carbide Fuel Development project is part of the AEC Fuel Cycle Development Program. The prime contractor is the Nuclear Development Corporation of America (NDA), and the subcontractor is The Carborundum Company. NDA is performing the conceptual design, fuel evaluation, fuel irradiation, and irradiated fuel reprocessing. The Carborundum Company is fabricating the fuel and reprocessing unirradiated fuel. Both companies are building plutonium handling facilities.

This report covers progress from September 15, 1959 to January 31, 1960. Previous progress was reported in Carbide Fuel Development – Phase I Report, NDA 2140-2 (October 15, 1959).

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1. INTRODUCTION

Fuel made of a combination of UC and PuC has a potential of reducing the fuel cycle cost of existing fast breeder reactors. The fuel cycle cost reduction is anticipated for two major reasons: increased burnup and increased power generation capability of PuC-UC compared to presently available metallic fuels. The effect of high burnup and high power generation rate on the stability of UC or PuC-UC is not known. However, based on UC irradiation tests, there is justifiable optimism that carbide fuels will be dimensionally more stable than metallic fuels. As a result of high melting point and good thermal conductivity of UC, there is further expectation that carbide fuels will be capable of high power generation rates.

The Carbide Fuel Development Program is studying the technology of the entire PuC-UC fuel cycle. The major goal of the program is to produce PuC-UC, and to obtain data on the irradiation behavior of PuC-UC for long burnups, and at high power generation rates. In addition, other areas of the fuel cycle are being explored to discover potential problems. The program is planned for a period of about $3\frac{1}{2}$ years and covers the following:

1. Conceptual Design (Completed). An analytical study of the effect of substitution of PuC-UC fuel in existing fast breeder reactors on heat transfer, physics, and cost was made. Conceptual, rod-type, fuel element configurations were proposed which can be substituted directly in existing reactors.
2. Facility Design and Fabrication. The experimental facilities to handle plutonium have been designed and are being built.
3. Fuel Fabrication and Evaluation. Various methods of fuel preparation and fabrication into cylindrical pellets are being explored. The pellets are being evaluated by density measurement, chemical analysis, x-ray diffraction, hardness, and metallography. Fuel cladding compatibility is being studied. Cermets of UC with small amounts of uranium metal will be cycled thermally.
4. Fuel Irradiation. Clad fuel samples will be irradiated, with burnup and maximum fuel temperature as variables; a post-irradiation examination will be made.
5. Fuel Reprocessing. A study of the reprocessing of both unirradiated and irradiated fuel will be made.
6. Full Scale Fuel Assembly. The design and construction of a full scale fuel element assembly for irradiation in an existing fast breeder reactor will be completed.

2. SUMMARY

2.1 FUEL FABRICATION AND EVALUATION

A 6 lb batch of good quality UC was synthesized by the UO_2-carbon reaction. Pellets with densities up to 13.0 g/cc, or 95% of theoretical, were produced by cold pressing and sintering. The effects of UC particle size, quantity of binder, sintering time, and temperature on final density were studied. As expected, reduction of particle size helped considerably in obtaining high densities. Pellets produced were evaluated by chemical analysis, x-ray diffraction, metallography, and hardness measurements.

A die has been made for the 0.191-in. diameter irradiation specimens, and has pressed pellets satisfactorily. The pellets, after sintering, have been ground to the desired tolerances.

Hot pressing of UC powder in an aluminum nitride die, rather than graphite die, reduced the reaction with the die material by a considerable factor.

Promising results have been obtained by limited efforts on the synthesis of UC by the ammonium diuranate-carbon reaction, and the simultaneous reacting and hot pressing of uranium metal and carbon. A density of 13.3 g/cc has been obtained by the latter method; the product contained a small amount of uranium metal.

Specimens for compatibility tests between UC and prospective cladding materials (Type 304 SS, $2\frac{1}{4}$% Cr-1% Mo, Inconel X, niobium, beryllium, Zircaloy-2) have been assembled, and are being tested at 820 °C.

Chemical analysis procedures have been investigated and some modifications have been made.

The availability of plutonium dioxide and plutonium metal was investigated by the New York Operations Office of the AEC.

2.2 IRRADIATION TEST DESIGN AND PROTOTYPE FABRICATION

The design of the irradiation capsules has been completed. A pressure probe for fission gas release measurement has been built and is ready for testing. Materials for the irradiation specimens have been ordered.

2.3 REPROCESSING STUDIES

Dissolution studies of unirradiated UC with simulated fission products ("fisside") showed that 99.97% uranium recovery is possible. Extrapolation of this data to irradiated fuel which has operated at high temperatures for a long time is not possible.

2.4 PLUTONIUM FACILITY CONSTRUCTION

2.4.1 Facility at The Carborundum Company

The laboratory and its services are nearly completed. The components of the helium purification system have been completed and are ready to be assembled. The plutonium handling boxes are expected to be delivered in February. Equipment to go in the boxes has been received, or is under construction. Equipment to be used for chemical analysis has been checked out satisfactorily. The plutonium laboratory is planned for completion in March.

2.4.2 Facility at NDA

The laboratory and its services are more than half complete. Construction of the gas purification system has begun. Three plutonium handling boxes have been equipped and used for chemical analysis. The remaining boxes are under construction. Much of the equipment to go in the boxes has been received and is being assembled. The plutonium laboratory is planned for completion in April.

3. FUEL FABRICATION AND EVALUATION

3.1 INTRODUCTION

The goal of the fabrication studies is to produce a high density combination of stoichiometric PuC and UC by powder fabrication techniques. The monocarbides are the most desirable of the fuel carbides, since they have the highest fuel density. A high bulk density (about 95% of theoretical) is desired to minimize fission gas release, as well as give a high fuel density. Some fuel is planned to be fabricated with excess uranium metal, to increase sinterability and achievable density as well as to increase thermal conductivity.

The goal of the evaluation tests is to identify the material as well as possible, by density measurement, chemical analysis, x-ray diffraction, metallography, and hardness. Additional out-of-pile tests of fuel-clad compatibility and thermal cycling of fuel containing excess uranium metal will survey some of the properties, knowledge of which is valuable for in-pile tests.

Current studies are limited to UC for lack of plutonium handling facilities. Fabrication and evaluation procedures are being developed for UC and will be applied to PuC-UC as soon as the plutonium handling facilities are completed.

3.2 CARBIDE POWDER PREPARATION

3.2.1 Uranium Oxide-Carbon Reaction

Based on the results of development work reported previously,[1] a 6 lb batch of UC was synthesized by reacting UO_2 and carbon in vacuum. Pellets for the reaction were about 1 in. in diameter, 1 in. long. The reaction was carried out at 1800°C for 4 hr. Analysis of two samples of the UC powder obtained gave the following results:

U	94.51 w/o;	94.40 w/o
Total C	4.82 w/o;	4.83 w/o
Free C	0.05 w/o;	0.06 w/o
Fe	0.11 w/o;	0.21 w/o
O	0.361 w/o;	0.342 w/o
N	0.021 w/o;	0.018 w/o
H	0.005 w/o;	0.005 w/o

X-ray diffraction patterns showed major UC, faint UO_2, and faint UC_2 lines.

Synthesis of PuC-UC will be done in atmospheric pressure inert gas. Since a good deal of the past work was done in vacuum, the procedure of synthesizing UC by the UO_2 + C reaction was repeated in argon at atmospheric pressure. A good purity of UC was obtained.

3.2.2 Ammonium Diuranate-Carbon Reaction*

Ammonium diuranate is a salt formed in the process of making UO_2 from uranyl nitrate. Cost savings would be possible if UC could be made directly from ammonium diuranate instead of UO_2. The ammonium diuranate-carbon reaction has not been studied before, and certain assumptions had to be made concerning its probable course. Ammonium diuranate decomposes on heating as follows:[2]

$$9(NH_4)_2U_2O_7 \rightarrow 6U_3O_8 + 14NH_3 + 15H_2O + 2N_2$$

The course of the simultaneous decomposition of ammonium diuranate and reaction of resulting uranium oxide with carbon is not known. Addition of a stoichiometric amount of carbon for the reaction $U_3O_8 + 11C \rightarrow 3UC + 8CO$ gave excess carbon. The amount of carbon was decreased until the UC product had less than the stoichiometric amount of carbon. The carbon addition in that test amounted to less than the stoichiometric amount required for the reaction $UO_2 + 3C \rightarrow UC + 2CO$. The best product to date was obtained by this latter mixture using the following procedure.

Ammonium diuranate and carbon were mixed for 4 hr, cold pressed, and reacted in an alumina tube furnace at 1800°C for 1 hr, in an argon atmosphere. The chemical analysis of the resulting powder was:

U	94.53	w/o
Total C	4.16	w/o
Free C	0.16	w/o
Fe	0.003	w/o
N	< 0.1	w/o

The carbon content was not sufficient to tie up all the available uranium as carbide. The remaining small amount of uranium was probably in the form of uranium oxide and/or uranium metal. X-ray diffraction showed strong UC, faint UO_2, and a trace of UC_2 lines. The results are encouraging, in view of the small number of trials made. Further work is planned.

3.3 CARBIDE PELLET FABRICATION

3.3.1 Pressing and Sintering

All the pellets described were fabricated from the UC batch made by the reaction of UO_2 and carbon, described in Section 3.2.1. Unless otherwise noted the pellets were 0.50 to 0.55 in. in diameter and of similar length.

The effect of particle size and temperature on final density was studied. Pellets were cold pressed at 40,000 psi with 1/2% Carbowax binder. The results are presented in Table 3.1.

An experiment was conducted to determine the effect of milling time on sintered density. The data shown in Table 3.2 indicate that there is little effect past 24 hr.

In order to determine the best percentage of Carbowax 6000 to use as a temporary binder, pellets were fabricated with 1/4, 1/2, and 3/4% additions. Although the following data (Table 3.3) show little effect, the pellets with 1/2% Carbowax added had sufficient strength for easy handling; this is probably the best amount to use.

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* Work done under AEC Contract AT(40-1)-2558.

Table 3.1 — Effect of Particle Size on Density of UC

Particle Size	Density, in g/cc, Obtained at 1850°C	1950°C	2050°C	X-Ray Analysis	Chemical Analysis, w/o	
-80 mesh	—	10.30	9.93	—	—	
~2 μ	—	10.65	10.60	—	—	
~0.5 μ*	12.50	—	—	Major phase, UC	U	94.96
				Minor phase, UC_2	Total C	5.26
					Free C	0.04
					Fe	0.04
					N	< 0.10

*The fine particle size was obtained by increasing the ball milling time to 24 hr (from the previously used 4 hr).

Table 3.2 — Effect of Milling Time on Density of UC

Milling Time, hr	24	48	72
Green Bulk Density, g/cc	8.75	8.70	8.80
Sintered Bulk Density, g/cc	12.83	12.67	12.69

Table 3.3 — Effect of Binder Percentage on Density of UC

Carbowax, %	1/4	1/2	3/4
Green Bulk Density, g/cc	8.59	8.75	8.44
Sintered Bulk Density, g/cc	12.40	12.83	12.53

All the above pellets were pre-sintered at 1700℃ in argon, and sintered at 1850℃ in vacuum for 1 hr.

Experiments are in progress to determine the effect of sintering time and temperature on density. Initial indications are that increasing the temperature above 1850℃ has little effect on density.

Two larger batches of pellets were made for evaluation by chemical analysis, x-ray diffraction, metallography, hardness, and fuel-cladding interaction studies. Densities of up to 95% of theoretical were achieved. The fabrication process used and the results are given in Table 3.4.

The reason for the two sintering steps is one of equipment limitation. The SiC-resistance-element-heated, alumina tube, argon atmosphere furnace is limited in temperature, but can be used for slow heating for pre-sintering. The induction-heated, vacuum furnace is not temperature-limited but is not adaptable for slow heating of samples. The combination of furnaces serves the sintering requirements. The PuC compacts will be sintered in a graphite pin resistance furnace in one firing.

The differences between the two batches are slight. The densities of Batch No. 2 pellets are higher. The reason for the higher densities is not obvious. The only difference in processing was a lower pre-sintering temperature for Batch No. 2. This batch also appeared to have a slightly lower particle size, which may have contributed to the higher density.

Metallography of samples from both batches of pellets indicated a structure of UC with a small amount of second phase present. Figs. 3.1 and 3.2 show photomicrographs of samples taken from each batch. Based on previous publications on the photomicrography of uranium carbide, the appearance of the second phase is that of UC_2. Since the carbon content is slightly below stoichiometric for UC and the oxygen content is about 0.3 w/o, the structure cannot be pure UC-UC_2. The most probable interpretation is that either one or both of the phases have a small amount of oxygen in solid solution. Fig. 3.1 shows a rounded third phase which has not been identified; it may be UO_2.

X-ray diffraction data obtained by The Carborundum Company and Brooklyn Polytechnic are in essential agreement. The sensitivity of the x-ray diffraction measurements is on the borderline for the quantities of impurities and second phase involved.

Diamond pyramid hardness readings on Batch No. 1 with 15 kg (superficial Rockwell), 1 kg (Newage microhardness), and 200 g (Bergman microhardness) produced a scatter greater than would be expected from UC alone. Cracking and porosity were the probable cause of the scatter. Microhardness readings on Batch No. 2 with 100 g and 200 g loads gave reproducible values that averaged 600 Vickers hardness. The 200 g loads caused hairline cracks.

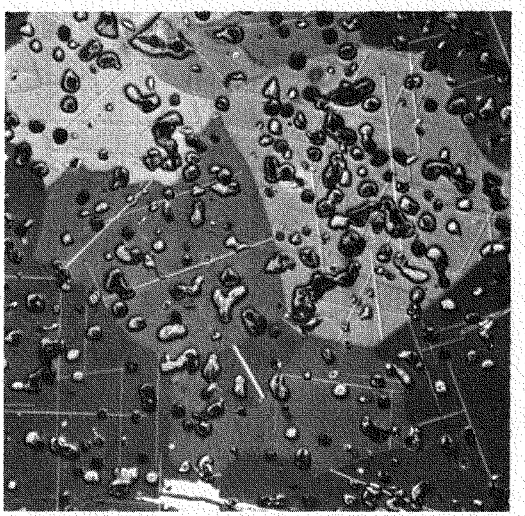
The irradiation specimens and all subsequent plutonium-containing specimens will be 0.191 in. in diameter rather than 0.50 in. diameter pellets. A die was made up for this size and tested successfully. Using UC powder which had been ball milled 24 hr, several pellets were pressed at 16,000 psi with single plunger action. The temporary binder was Carbowax. The unfired bulk density was 8.95 g/cc. The pellets were pre-sintered to 1375℃ in an argon atmosphere and then fired for 1 hr at 1850℃ in vacuum. The sintered bulk density was about 12.2 g/cc. A typical as sintered pellet had the following dimensions: ends concave by 0.0015 in. diameter; one end, 0.195 ± 0.001 in.; center 0.193 ± 0.001 in.; other end 0.197 ± 0.001 in. In future experiments, double plunger action will be used in cold pressing. It is believed that this will produce more uniform pellets. Subsequent centerless grinding of the pellets produced the desired dimensions of 0.191 + 0.000, - 0.001.

Table 3.4 — Fabrication Procedure and Properties of UC Pellets

Item	Batch No. 1	Batch No. 2
Milling time, hr	24	24
Particle size, μ	~1	<1
Binder, % Carbowax	1/4	1/4
Cold pressing pressure, psi	16,000	16,000
Pre-sintering		
Temperature, °F	1,750	1,500
Time, hr	1	1
Atmosphere	Argon	Argon
Final sintering		
Temperature, °F	1,850	1,850
Time, hr	1	1
Atmosphere	100 μ vac.	100 μ vac
Number of pellets	14	15
Density		
Range		
g/cc	12.2-12.5	12.4-13.0
% of theoretical	89.5-91.8	91.3-95.5
Average		
g/cc	12.3	12.7
% of theoretical	90.4	93.3

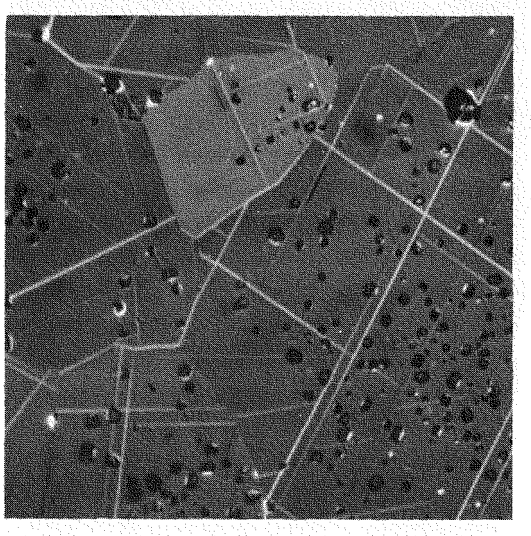
Chemical analysis		Lowest Density	Highest Density
U, w/o	94.87	95.01	94.94
Total C, w/o	4.71	4.62	4.58
Free C, w/o	0.05	0.03	0.03
Fe, w/o	0.01	—	—
N, w/o	<0.10	0.09	by vacuum fusion
O, w/o	~0.25, by difference	0.35	by vacuum fusion
X-ray diffraction*			
The Carborundum Co.	Major, UC	Major, UC	Major, UC
	Faint, UC_2	Faint, UC_2	Faint, UC_2
	Faint, UO_2	Very faint, UO_2	Very faint, UO_2
Brooklyn Polytechnic Institute*	Major, UC	Major, UC	
	Faint, UC_2	Very faint, UC_2	
	Faint, UO_2	Very faint, UO_2	
Lattice constant, Å	4.963 ± 0.001	4.963 ± 0.002	
Metallography	See Fig. 3.1	See Fig. 3.2	

*X-ray patterns were taken on different samples from the same batch.



NEG. NO 1197B

Fig. 3.1 — UC from Batch No. 1 – 500 × – nitric acid-acetic acid-water etch. The matrix phase is UC. The acicular phase is UC_2 and the globular phase may be UO_2. The UC and UC_2 may contain some oxygen in solid solution.



NEG NO. 1190RE

Fig. 3.2 — UC from Batch No. 2 – 500 × – nitric acid-acetic acid-water etch. The matrix phase is UC. The acicular phase is UC_2. The UC and UC_2 may contain some oxygen in solid solution.

In order to improve sintered density, experiments were started on sintering UC pellets containing additions of uranium metal. One percent uranium metal (100 to 250 mesh shot made by the National Lead Company) was added to the UC powder and the mixture ball milled for 24 hr. The pellets were cold-pressed at 16,000 psi with Carbowax binder, pre-sintered at about 1375°C in argon and then vacuum sintered at 1850°C for 1 hr. All sintered pellets were cracked near the ends. The nature of the cracks indicated that they may have started during cold pressing. Work on metal additions is being continued.

3.3.2 Hot Pressing UC Powder

Some 80 mesh UC powder made by the UO_2 + C reaction was hot pressed in an aluminum nitride die at 1800°C and at 2000 psi for 1 hr in an argon atmosphere. The resulting pellet had a density of 12.4 g/cc. Chemical analysis of the pellet was as follows:

U	94.32 w/o
Total C	4.92 w/o
Free C	0.07 w/o
N	< 0.10 w/o
Al	0.21 w/o

The analysis indicates there was only a slight reaction with the die material. Aluminum nitride is superior to graphite as a hot pressing die for UC. Experience on this project and literature references indicate that UC reacts with graphite dies during hot pressing, to form UC_2.

Further work on hot pressing was discontinued because of the good success in obtaining high densities by the more economical cold pressing and sintering process.

3.3.3 Simultaneous Carbide Synthesis and Pellet Fabrication

The lower reaction temperature of the reaction U + C → UC compared to UO_2 + 3C → UC + 2CO may make the simultaneous synthesis and fabrication of UC possible. Considerable crushing and milling, as well as additional cold pressing and sintering, would be eliminated. Higher densities may also be obtained. The advantages are offset by the higher cost of metal vs oxide.

An experiment was made to react uranium metal and carbon by hot pressing the mixture. The 100 to 250 mesh uranium metal shot was milled with a stoichiometric amount of carbon for 24 hr and hot pressed in a graphite die at 1500 psi in an argon atmosphere. The temperature was held at 800°C for 1 hr, then gradually raised to 1400°C and held for 2 hr. The resulting pellet had a density of 13.3 g/cc. X-ray diffraction indicated major UC and faint UO_2 lines. The chemical analysis of the pellet was as follows:

U	95.26 w/o
Total C	4.35 w/o
Free C	0.13 w/o
Fe	0.04 w/o
N	< 0.01 w/o

The above analysis indicates that there is a small amount of uranium metal still present. Considering the encouraging results obtained, work on this method of fabrication will be continued.

3.3.4 Fuel Cladding Compatibility Tests

In order to study possible fuel-cladding interactions under conditions similar to those which would prevail in an actual fuel element, a series of diffusion couples are being tested. The carbide fuel is held in contact with various cladding materials at 820°C in a helium atmosphere. Following this, the couples will be sectioned and the carbide-cladding interface examined by metallography, x-ray diffraction, and autoradiography. The tests have run for 400 hr to date.

The cladding materials being tested and the planned test periods are tabulated below. Samples are in duplicate.

	Type 304 SS	$2^1/_4$% Cr-1% Mo	Inconel X	Niobium	Zircaloy-2	Beryllium
1000 hr	X	–	X	X	–	–
2000 hr	X	–	X	X	–	–
4000 hr	X	X	X	X	X	X

3.3.5 Chemistry

Current and proposed methods of chemical analysis are described in Reference 1. As a result of additional investigations some modifications are being made.

Plutonium analysis by alpha counting will be used. Two sets of plutonium counting standards will be made up by the New Brunswick Laboratory of the AEC, one each for NDA and The Carborundum Co.

Various means for determining iron impurities in uranium (and in the future plutonium) were studied, since iron interferes with the usual methods of determining uranium content. At NDA solvent extraction and ion exchange methods were tried to separate uranium and iron. Solvent extraction of iron from 9M HNO_3, containing 3% H_2O_2, by a 0.5M solution of TTA in xylene proved to be too slow for glove box operation. The ion exchange separation proved to be more convenient and efficient. A 10M HNO_3 solution of uranium and iron was passed through a Dowex 1-8x resin column. The iron passed through the column and uranium was subsequently removed by dilute acid or water. At The Carborundum Co. the fluorimeter used for uranium microanalysis is being adapted for colorimetric determination of iron.

3.3.6 Plutonium Procurement

The New York Operations Office of the AEC has located sources of plutonium dioxide and plutonium metal, to be used as starting material in the fabrication of plutonium carbide.

An order for plutonium will be placed as soon as the feasibility reports currently being written by NDA and Carborundum are approved.

4. IRRADIATION TEST DESIGN AND CAPSULE PROTOTYPE FABRICATION

4.1 INTRODUCTION

In order for PuC-UC fuel to reduce fuel cycle cost, several conditions must be met. The fuel has to be able to achieve high burnup, high operating temperatures, and high power. At least 2 a/o burnup is desirable at fuel temperatures above 1200°F with maximum dimensional stability of the fuel and minimum release of fission products. Power should be at least equivalent to presently planned fuels. The objective of the irradiation program is to determine whether the high burnup, temperature, and power required can be achieved.

The test will measure temperatures, burnup, power, and dimensional stability of the fuel. In addition, measurements of in-pile effective thermal conductivity, fission gas pressure, and fission gas release will be attempted. The first two capsules containing UC specimens will go into the test reactor during the Fall of 1960.

4.2 DESIGN

The design of the irradiation specimens and capsules was completed. The capsules and test conditions are essentially as described in Reference 1. The major variances made from the reported design involved increasing the reliability of the capsule heater, the function of which is to keep the capsule at constant temperature in spite of reactor power fluctuations and loss of fission heat with increased burnup. First, the enrichment of the UC fuel was increased from $3\frac{1}{2}\%$ to 24%; the increased enrichment will decrease the power load on the heater at 2 a/o burnup, since only 8% of the fission heat will be lost instead of 57%. Second, the overall OD of the heater was increased to 1/8 in., a size which has performed more reliably than the smaller sizes. Third, a specification was made for close inspection and performance testing of heaters by the vendor prior to acceptance by NDA.

The 24% U^{235} enrichment of UC was determined by taking the uranium enrichment required for fission power equivalent to 20% Pu^{239} enrichment of PuC-UC. The 20% Pu^{239} enrichment is required for the substitution of a PuC-UC core for U-10% Mo in the EFFBR, according to the conceptual design study.[1] The UC irradiation capsule design will thus be directly applicable to the PuC-UC irradiation.

The capsule design for the 24% enriched specimens is the same as for the $3\frac{1}{2}\%$ enriched specimens since it is planned to use the same heat generation rate by decreasing the neutron flux.

4.3 PROTOTYPE CAPSULE FABRICATION

A prototype of the pressure probe for the measurement of fission gas release has been built. Out-of-pile testing will be started shortly.

Enriched fuel and cladding tubing has been ordered for the irradiation specimens.

Some of the fuel pellets will require an axial hole to accept a thermocouple well. A 0.070 in. hole was drilled successfully in a 0.53-in. diameter UC pellet, using a tungsten carbide drill. Drilling of 0.191-in. diameter pellets (actual size of the irradiation samples) will be tried shortly.

5. FUEL REPROCESSING

5.1 INTRODUCTION

The major problem in reprocessing uranium carbide-plutonium carbide fuels by liquid-liquid extraction methods is expected to be dissolution of the spent fuel. While UC is expected to dissolve readily in conventional solvents such as nitric acid, the solubility of PuC-UC is unknown. The probable behavior of the irradiated fuel containing fission products is still more difficult to predict. Laboratory studies on the dissolution of pellets of UC and PuC-UC with selected simulated fission products may provide guidance for methods of attack on the difficult problems of dissolution of the irradiated fuel. Reprocessing studies were discontinued for the present contractual period, but will be resumed in the next contractual period.

5.2 DISSOLUTION STUDY

A few UC pellets were made up containing simulated fission products to find out whether they affect the solubility of the pure UC. The "fisside" was not expected to reproduce the solubility problems of a real irradiated carbide; the latter is probably more difficult to dissolve. It was hoped that the "fisside" will give some measure of the problems that might arise with irradiated fuel.

The simulated fission products were limited to relatively stable carbide formers. The amounts to be present after 40% plutonium atom burnup of 20 w/o PuC-80 w/o UC were calculated.[1]

The composition of pellets by weight percent is shown below.

Constituent	%	Constituent	%	Constituent	%
UC	96.28	NbC	0.02	ZrC	0.64
LaC_2	0.25	CeC_2	0.58	MoC	0.72
YC_2	0.06	Pd+C	0.61	RuC	0.84

The methods of dissolution were described in Reference 1.

The examination of the residues of the "fisside" pellets has been completed. The uranium content of the insoluble residues from various dissolution steps was as follows:

	8N HNO_3	HF-HNO_3	HCl-HNO_3
Total Residue, w/o (3.72% Total Simulated Fission Products Added)	4.24	2.10	7.55
U in Pellet Left in Residue, w/o			
X-ray spectrographic analysis (The Carborundum Co.)		<0.01	Major
Fluorescence analysis (Brooklyn Polytechnic)	Small amount	Not detectable	—
Chemical analysis (NDA)	1.2	0.03	—

The $HF-HNO_3$ treatment appears to be the most effective, and high uranium recovery from the unirradiated fisside is possible. This indicates that reprocessing problems are not immediately apparent. The results cannot be extrapolated to irradiated fuel, which has operated at a high temperature for a long time.

6. PLUTONIUM FACILITY CONSTRUCTION

6.1 INTRODUCTION

The Carborundum Company is constructing a facility to be used for fabricating the fuel and studying "cold" reprocessing. The design of the facility is complete and its components are being built. The construction of the facility and assembly of the equipment is expected to be complete in March 1960. NDA is constructing a facility to be used for fuel evaluation, specimen preparation, irradiation, post-irradiation examination, and "hot" reprocessing. The design of the pre-irradiation facility is complete and its components are being built. The construction of the pre-irradiation facility and equipment is expected to be completed in April 1960. The equipment for post-irradiation examination will be designed and built in 1961.

6.2 FACILITY FOR FUEL CARBIDE FABRICATION AT THE CARBORUNDUM COMPANY

6.2.1 Laboratory

The laboratory has been nearly completed. The steel walls, doors, and celing are erected and sealed. The vinyl tile floor is completed and sealed. Lighting has been installed. The room ventilation system (blowers, filters, air conditioner, and ductwork) has been installed. The shower, the sink, and the waste water storage system are complete and installed. The power lines to the laboratory are currently being put in. The emergency generator has been installed. The helium mass spectrometer leak tester and the dielectric plastic sealer have been received.

6.2.2 Helium Purification System

The components of the helium purification system have been completed by the Liquid Metals Corporation and shipped to The Carborundum Company. Installation of the gas system will begin shortly.

6.2.3 Plutonium Handling Boxes and Laboratory Equipment

The plutonium boxes are being built by the Liquid Metals Corp. The components for the boxes have been completed; however, delivery of the boxes has been delayed due to problems in aluminum welding. The chief problem, meeting strict weld porosity specifications, is believed to be under control now and the boxes are expected to be completed during February.

All the equipment to go in the boxes has been ordered, and nearly all of it has been received. The equipment for chemical analysis has been checked out. The Fisher Carbon Apparatus was checked out with Bureau of Standards silicon carbide. The fluorimeter was checked and found to respond linearly over the range 10^{-2} to 1 μg of uranium per sodium fluoride pellet. Uranium carbide samples were analyzed for uranium and carbon on the plutonium laboratory equipment; the analyses corresponded closely to check analyses carried out by the central chemistry labor-

atory. Equipment for fabrication such as press, mill, grinder, etc. have been received. The graphite resistance furnace is under construction.

6.3 FACILITY FOR CARBIDE EVALUATION AT NDA

6.3.1 Laboratory

The laboratory is more than half complete. Dividing walls to separate the plutonium area from the remainder of the laboratory have been erected. Internal sheet rock walls and lowered ceilings have been installed. Lighting and power for equipment has been installed. The building to house the gas purification system and emergency power source has been constructed. The foundation for the waste disposal building, and the drain line leading to it have been laid.

6.3.2 Helium Purification System

Construction of the gas purification system has started. To date the main headers have been completed.

6.3.3 Plutonium Handling Boxes and Laboratory Equipment

The box layout of the laboratory has been changed from two L-shaped lines to two straight lines of four boxes each. One decontamination box and one helium transfer box will stand separately. S-207, the air transfer box, has been eliminated.

Of the four boxes built in Phase I, three have been equipped with equipment for chemical analysis. Uranium-iron separations and uranium analyses have been carried out satisfactorily in the boxes. Carbon analyses have been made, but the desired degree of accuracy has not yet been obtained. The remaining box is being outfitted for cutting and grinding operations.

The remaining six boxes are being built by Boyle Metalcraft Corporation. The entry ports, glove ports, and window frames have been completed by NDA and shipped to Boyle for assembly. Two hoods have been ordered.

Much of the equipment to go into the boxes has been received. The furnace for fuel-cladding compatibility studies is nearly complete.

7. REFERENCES

1. Carbide Fuel Development – Phase I Report – Period of May 15 to September 15, 1959, NDA 2140-2 (Oct. 15, 1959).
2. J. Katz and E. Rabinowitch, "The Chemistry of Uranium," McGraw-Hill Book Company, Inc. New York, 1951.

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