Westinghouse Advanced Reactors Division

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MASTER

URANIUM-PLUTONIUM CARBIDE DEVELOPMENT
QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING SEPTEMBER 30, 1968

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INTRODUCTION

This eighth quarterly progress report on the Uranium-Plutonium Carbide Development Program covers the work performed during the first quarter of FY-1969.

SUMMARY OF OBJECTIVES

The Westinghouse (U,Pu)C program is directed toward the development of the sodium-bonded fuel element concept, the intents of which are to maximize heat transfer between fuel and cladding and to provide expansion space for fuel swelling in order to minimize fuel and mechanical interaction between cladding. In accordance with the overall objective and the use of this concept, the major specific objectives of this program are as follows:

- a. Develop and perform fabrication processes that produce suitable fuel elements for the irradiation program;
- b. Develop and perform characterization studies on fuel material and fuel elements to provide proper identification of irradiation samples;
- c. Design and perform appropriate steady-state and transient irradiation tests on fuel elements;
- d. Evaluate the irradiation performance of the sodium-bonded mixed-carbide fuel elements with particular emphasis on fuel swelling, fission gas release, compatibility of fuel and cladding, and structure of fuel and cladding;
- e. Evaluate the influence on irradiation performance of a range of fuel materials variables, that include:
 - (1) three types of carbide fuel--hyperstoichiometric, stoichiometric, and chemically modified with Cr_{23}C_6 ,
 - (2) two densities,
 - (3) an additional variant of 0.5 to 1.0 wt % oxygen in stoichiometric fuel,
 - (4) various power ratings resulting in different fuel center temperatures up to approximately 1000°C , and
 - (5) burnups ranging from 15,000 to 100,000 MWD/T;
- f. Develop theoretical and empirical models for the behavior of carbide fuel elements under steady-state and transient irradiation conditions.

The attainment of these objectives will form the basis for defining a meaningful engineering program on sodium-bonded carbide fuels in the post-1971 period, consistent with the long-range goal of the commercial development of LMFBRs.

PRIOR WORK

All work related to the present investigations and performed prior to the present period has been fully reported in:

WCAP-3791-2, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending December 31, 1966,"

WCAP-3791-4, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending March 31, 1967"

WCAP-3791-8, "Uranium-Plutonium Carbide Development Quarterly Progress Report for Period Ending June 30, 1967"

WCAP-3791-10, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending September 30, 1967"

WARD-3791-12, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending December 31, 1967"

WARD-3791-18, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending March 31, 1968."

WARD-3791-21, "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending June 30, 1968."

SUMMARY OF CURRENT PROGRESS

A summary of current progress is shown below. Detailed progress is reported in the individual task sections of this document.

Under Task CDAA-100, authorization for FY-1969 work was received from the Commission based on the work program submitted in June. Internal work authorizations and budgets for FY-1969 were established based on fiscal year and interim funding.

Under Task CDAA-200, development of acceptable processes for fabricating stoichiometric fuel continued. Preparation of experimental hyperstoichiometric pellet batches was initiated. Variables investigated for the five experimental batches of stoichiometric fuel and for the batch of hyperstoichiometric fuel prepared to date include initial carbon content, milling time, binder type, pressing pressure, and firing conditions. The available analyses, supported by process variable studies on available hypostoichiometric fuel, indicated that: (a) initial carbon contents of 4.45 and 4.70 wt % are required to yield stoichiometric and hyperstoichiometric pellets, respectively; (b) milling times should be increased from 2.5 to 6.0 hours to achieve better density control; and (c) maximum pressing pressures of 20 tsi are sufficient to yield desired densities while minimizing pellet defects, such as capping and lamination from this source. The latter defects also appear to be significantly dependent on powder characteristics. A statistical program designed to better define these areas and an acceptable fabrication procedure are in progress. The major work on Cr_{23}C_6 -modified fuels consisted of preparing a back-up lot for the EBR-II irradiation program. Further work on this fuel will be deferred in favor of preparing stoichiometric and hyperstoichiometric fuel.

Under Task CDAA-201, detailed evaluation of several batches of fuel, including GETR and EBR-II pellet lots, was conducted. This evaluation was based on metallography, chemical analysis and microprobe data from LASL. In general, all fuel lots appear to contain higher carbon contents than specified. Details of the evaluations are presented. A detailed review and comparison of N_2 analysis methods indicated that the Nitrox unit performs satisfactorily. Development of etching techniques for metallographic examination of stoichiometric and hyperstoichiometric fuels is in progress, and results to date appear satisfactory. Development of improved particle size characterization techniques was continued and thermogravimetric studies of binder-burnout kinetics were initiated.

Under Task CDAA-300, two pins containing $Cr_{23}C_6$ -modified fuel were loaded, encapsulated and inspected in preparation for irradiation in EBR-II. Profilometer diameter traces were taken for both pins. Analysis of two control pins (loaded in parallel) showed oxygen impurities in the sodium bond of 308 and 394 ppm. Equipment was installed for bonding six EBR-II pins simultaneously. Investigations of potentially improved eddy current equipment and methods for decreasing oxygen contamination levels in the sodium bond were continued.

Under Task CDAA-400, the nine fuel pins in GETR continued to operate satisfactorily throughout the quarter with pin W1GH nearing completion of the scheduled exposure and an accumulated average burnup greater than 45,000 MWD/T. The capsules were temporarily removed from the reactor between Cycles 98 and 99 for an interim examination by neutron radiography. Results of the examination are presented. The first draft of the design and fabrication report was completed, and review is in progress. An analysis of post-irradiation shipping costs was completed, and action initiated for making shipping arrangements. The facility for post-irradiation examination was identified as the Los Alamos Scientific Laboratory (LASL), with whom initial contact was made.

Under Task CDAA-500, two capsules containing $Cr_{23}C_6$ -modified (U,Pu)C, identified as W2X and W8X, were shipped to EBR-II. Detailed evaluation of the fuel composition and inspection records was conducted in conjunction with work under Tasks CDAA-200, -201, and -300 to ensure that the pins were satisfactory and that all EBR-II requirements were met. Evaluation of available additional $Cr_{23}C_6$ -modified fuels is in progress to determine if they are suitable for irradiation testing. Additional fabrication effort on this fuel type will be deferred as indicated previously. The thermal, nuclear, and mechanical design of the Phase I capsules was completed. The revised design and hazards report for these capsules was completed and is in reproduction. Approval-in-principle for the Phase I program was received from ANL.

CDAA-100 - PROJECT ADMINISTRATION

A. E. Duval, W. B. Heubel, P. Murray, P. L. Wyzenbeek

OBJECTIVES

The objectives of this task are to assure that the project objectives are accomplished on schedule, within budget, and to the satisfaction of the Atomic Energy Commission; to assure compliance with contracted obligations; and to coordinate this project with other AEC-sponsored and Westinghouse-sponsored LMFBR development projects.

Under this task, overall project direction and day-to-day administration will be provided. Plans and controls will be established and maintained; periodic reviews will be held with the Commission; correspondence and reports will be coordinated; day-to-day technical and administrative liaison with the Commission will be provided.

PRIOR WORK

Work under the contract started on October 3, 1966. FY-1967 administrative actions included submission and approval of the FY-1967 work program, negotiation and execution of the contract, the obtention of the license to operate the Plutonium Fuels Development Laboratory (Cheswick), and procurement of plutonium metal and enriched uranium. Detailed schedules, budgets, shop orders, IWRs, and subcontracts were prepared and issued; progress was monitored and evaluated; all aspects of the project were coordinated; and day-to-day liaison with Commission was provided. The following reports were published during FY-1967; two quarterly progress reports (WCAP-3791-2 and WCAP-3791-4), a topical report on the compatibility of mixed carbide with stainless steel (WCAP-3791-3), and the Irradiation Test Program (WCAP-3791-6).

The FY-1968 work program (WCAP-3791-5) was prepared and submitted in May, 1967. It was revised in August, 1967 to conform with the FY-1968 ceilings, and was approved by the Commission. Other FY-1968 administrative actions included the negotiation of additional contract scope, schedule and financial controls, issue of FY-1968 shop orders and IWR's, etc. In January, 1968, the FY-1968 ceilings were reduced, requiring a revision of the FY-1968 scope. During FY-1968, quarterly progress reports WCAP-3791-8, WCAP-3791-10, WCAP-3791-12, and WCAP-3791-18, were published. The FY-1969 work program was prepared and submitted in June, 1968.

CURRENT PROGRESS

FY-1969 work was authorized by the Commission based on the work program submitted in June. Contract modifications 9 through 11, which funded work through September, were received and executed. FY-1969 project plans and controls were established. Based on RDT program guidance, the FY-1969 work program (WARD-3791-23) was revised and issued. Work is proceeding on schedule and within budget.

CDAA-200 - FUEL MATERIAL FABRICATION DEVELOPMENT

M. G. Bale, A. Boltax, W. R. Jacoby, T. W. Wallace, N. G. Wolcott, Jr.

OBJECTIVES

The principal objectives are to develop uranium-plutonium carbides with and without modifiers for use as fast breeder reactor fuels in sodium-bonded fuel elements, and to develop process techniques for fabrication of fuel pellets for fuel test assemblies. The test assemblies will be utilized to evaluate irradiation performance of these fuel materials as defined in Tasks CDAA-400, CDAA-500, and CDAA-600.

PRIOR WORK

Investigation of Process Variables

Initial work on the use of carbowax as a binder indicated that 0.5 wt % was the optimum amount. Smaller amounts did not adequately protect the powder from oxidation, and amounts over 0.5 wt % caused lamination and cracking of the pellets during the burn-out cycle. Later, it was noted that fairly consistent increases of 2,500 ppm oxygen occurred between the powder and pellet analyses; this value is approximately equal to the oxygen content of the carbowax addition. Work continued using oxygen-free binders (naphthalene, paraffin wax), and the initial results with these materials indicated that lower oxygen contents were being obtained. Subsequent analyses, however, showed that these binders were not protecting the powder adequately, and consequently, the carbide did not press satisfactorily.

During part of this work, high nitrogen results were recorded on buttons, powders, and pellets. The values appeared to be erratic, and all possible sources of contamination and error were checked. From the available results, it appeared that absorption of nitrogen into the carbide powder was the most likely cause of the problem, but the condition required for such absorption could not be identified. To minimize the amount of waste material generated, work commenced on the reclamation of out-of-specification pellets. Scrap Cr_{23}C_6 -modified (U,Pu)C pellets were arc-melted, and button and pellet samples were taken for metallographic examination. While no problems were experienced in processing the material, evaluation data were not available at that time.

Fabrication of EBR-II Irradiation Test Specimens

In the last period, the initial effort was devoted to the fabrication of Cr_{23}C_6 -modified fuel. Seven batches of material were processed, and two were subsequently loaded into pins for irradiation testing. Chemical analyses and processing conditions were tabulated for reference purposes.

Three batches of stoichiometric fuel were also processed during the period, but the pellets were sintered to densities of 78 to 85% T.D. These values were too low for the fuel requirements, and available chemistry results indicated that hyperstoichiometric material had been prepared. These pellets were sent for re-processing.

CURRENT PROGRESS

Work performed during this period is reported in two parts: investigation of process variables and fabrication of EBR-II Phase I fuel pellets.

Investigation of Process Variables

a. Reprocessing of Materials

In an effort to reclaim out-of-specification Cr_{23}C_6 -Modified (U,Pu)C, scrap pellets were returned to the arc-melting stage for reprocessing. A sample of the arc-melted button was taken for metallographic examination prior to processing the remainder into pellets. A fired pellet was also sampled for metallography, and button and pellet were compared with previously processed Cr_{23}C_6 -modified material. The comparisons showed abnormally complex structures, indicating that it is not feasible to reclaim modified material by this route. A similar method has been used for the reprocessing of green and fired unmodified pellets. The oxygen content of the material can be reduced by repeated meltings, and a corresponding decrease in the carbon content normally occurs. This is shown in Table 1. However, the metallographic structure resulting from this treatment has shown porous grains. A possible explanation of the porosity is the formation and trapping of CO within the grains during the initial stages of solidification. During processing, this material does not behave the same as that prepared from elemental stock; additional effort is required to determine the optimum parameters for the production of acceptable pellets.

b. Effects of Binders

Experimental batches have been processed to compare the effects of carbowax and acryloid/stearic acid mixtures as binders. Complete analyses are not yet available. The initial results indicate that, for similar processing conditions, the acryloid/stearic acid additions increase firing shrinkage and produce a higher carbon content in the final pellets. The latter effect may be caused by the larger percentage of carbon in the acryloid/stearic acid mixture combined with a binder burn-out cycle that is too short. Based on recent studies on the decomposition of carbowax, a slower binder burn-out cycle is being used, so that the furnace reaches 700°C after three hours rather than the 1000°C previously attained.

Table 1

Arc-Melted Powder Results

Material	Number of Melts	Carbon (wt%)	O ₂ (ppm)
UC	--	4.69	3,402
^a Button C	5	4.41	419
^a Button B	12	4.24	159
^a Button A	12	4.74	127

^aButtons made by pressing powder into pellets and arc-melting.

c. Effect of Pressing Pressure

Throughout the work, continued efforts have been made to decrease the pressing pressure. In this way, pellet defects previously observed in green and fired pellets (capping, cracking, etc.) have been minimized without greatly affecting the final density. Figure 1 indicates the effect of pressure on the final densities of UC and (U,Pu)C hypostoichiometric pellets. A pressure of 20 tsi is currently used for the fabrication of most pellets.

More recent work on the problem of defects suggests that slight variations in the powder characteristics also have a significant effect on the susceptibility to pellet laminating and capping.

Fabrication of EBR-II Phase I Fuel Pellets

a. Powder Preparation

The powder preparation method has been altered by varying the milling time and mixing method. The new procedure, which will be used for fabrication of stoichiometric and hyperstoichiometric fuel, is shown in Figure 2. The milling time has been increased from 2.5 to 6.0 hours since previous work showed that denser pellets could be obtained from powders that had been milled for longer times. The final dimensions and densities of pellets are now controlled by varying the firing cycle. Problems associated with spattering of carbide powder while removing alcohol under a dynamic vacuum have been eliminated by hand mixing the powder until dry. Initial results indicate that the final blended powder is homogeneous.

b. Cr₂₃C₆ - Modified Fuel

Four stack lengths of this fuel type have been loaded into pins; two of these (batches 502 and 503) have been encapsulated and shipped to the EBR-II test reactor site. The initial analyses and processing conditions for batches 514 and 518 are given in Table 2; complete chemical analyses are given under CDAA-201.

The third Cr₂₃C₆-modified batch, 520, was originally processed as back-up material for the irradiation test program, but it has subsequently been scrapped since it did not fire to the specified density. Further work on Cr₂₃C₆-modified fuel will be deferred in the interest of preparing stoichiometric and hyperstoichiometric EBR-II fuel pins.

c. Stoichiometric Fuel

Work continued through the quarter on determining the optimum processing conditions for stoichiometric fuel. Ranges of variables used while processing the five batches of material were:

Figure 1 Pressing Pressure vs. Percent Fired Theoretical Density

Figure 2 Powder Preparation Flow Diagram

Table 3
Cr₂₃C₆-Modified Irradiation Fuel Batch Status

Batch No.	Chemical Analysis (wt%)						Mill Time (hr)	X Additions	Screen Size (mesh)	Pressure (psi)	X T.D. (green)	Fire Time (hr)	Fire Temp. (°C)	X T.D. (fired)
	Powder			Pellet										
	C	O ₂	N ₂	C	O ₂	N ₂								
514	- Not Available -			4.43	3,425	227	2.5	0.5 Carbowax 2.0 Cr ₂₃ C ₆	200	20		2	1550	87-91
^a 518	- Not Available-			4.52	3,800	235	4.0	0.5 Carbowax 0.5 Stearic Acid 2.5 Cr ₂₃ C ₆	200	10	75	2	1550	85-86
^a 520				5.09			6.0	0.5 Carbowax 3.0 Cr ₂₃ C ₆ 0.25 Stearic Acid	400	10	73	2	1550	86

^a A number of test fires, with different powder processing conditions and firing cycles, were used on these batches. The reported results represent those obtained on the main branch.

Carbon	- 4.30 to 4.65 wt %
Binder	- Carbowax, with and without stearic acid addition and acryloid/stearic acid mixture.
Milling Time	- 2.5 and 6.0 hours
Applied Pressure	- 10 to 30 tsi
Firing Temperature	- 1500 to 1700°C
Firing Time	- 1 to 12 hours

Typical results are given in Table 3, with many of the other values recorded still being evaluated. It should be noted that the available analyses indicate the pellets to be hyperstoichiometric, although the initial "weighed-in" carbon value was below 4.8 wt%. It is currently believed that a starting carbon figure of 4.45 wt% should yield pellets with C_E in the range 4.8 to 4.9 wt%. This is based on the following calculation:

Carbon weighed into button	= 4.45 wt%
Oxygen content of pellet	= 0.23 wt%
Nitrogen content of pellet	= 0.04 wt%
Carbon picked up from binder	= <u>0.12</u> wt%
TOTAL (C_E value of pellet)	= 4.84 wt%

All analyses will subsequently be correlated with metallographic examinations.

d. Hyperstoichiometric Fuel

To date, one batch of hyperstoichiometric material has been fabricated and some of the results are given in Table 4. As with the stoichiometric carbides, this was an experimental batch, and some variations to the processing conditions were employed. The applied pressure was varied between 10 and 30 tsi, but this did not appear to affect the final density. Similar effects were noted with one and two hours firing time and 1500 and 1550°C firing temperature. C_E values on the pellet were higher than anticipated, 5.3 to 5.4 wt%, indicating that lower "weighed-in" carbon values should be used in future work.

A statistically designed program is to be conducted to determine the major effects of process variables both for stoichiometric and hyperstoichiometric fuel. For this work, starting carbon contents of 4.45 and 4.75 wt% will be used for the stoichiometric and hyperstoichiometric compositions respectively. Results from this experiment will be correlated with values previously obtained.

Table 3

Unmodified Stoichiometric Fuel Batch Status

Batch No.	Chemical Analysis (wt%)						Mill Time (hr)	% Additions	Sieve Size (mesh)	Pressure (tsi)	% T.D. (green)	Fire Time (hr)	Fire Temp. (°C)	% T.D. (fired)
	Powder			Pellet										
	C	O ₂	N ₂	C	O ₂	N ₂								
513	- Not Available -			4.26	3,565	190	2.5	0.5 Carbowax	200	20	--	2	1550	76-81
516	- Not Available -			4.73	2,606	1347	6.0	0.5 Carbowax	200	20	69.0	2	1550	90-94
517A	- Not Available -			4.37			6.0	0.5 Carbowax	200	20	74.5-75.0	12	1500	- 85
517B	- Not Available -			4.59			6.0	0.5 Acryloid/ Stearic Acid	200	20	70.5-71.5	12	1500	- 89
519	4.75	2976	449				6.0	0.5 Carbowax	200	20	72.0-	2	1500	86-88
521	4.80			4.56			6.0	0.5 Carbowax	200	20	73.5	2	1500	90-91

Note: All the above batches have been processed or fired under varying conditions. The above results are typical of the values obtained.

Table 4

Unmodified Hyperstoichiometric Fuel Batch Status

Batch No.	Chemical Analysis (wt%)						Mill Time (hr)	% Additions	Sieve Size (mesh)	Pressure (tsi)	% T.D. (green)	Fire Time (hr)	Fire Temp. (°C)	% T.D. (fired)
	Powder			Pellet										
	C	O ₂	N ₂	C	O ₂	N ₂								
515	4.99	231	260	5.00	3400	400	6	0.5 Carbowax	200	20	68-69	2	1550	94

Note: Other firing times and temperatures were also used on portions of the above batch. The results quoted are typical of the values obtained.

CDAA-201 - FUEL CHARACTERIZATION

A. Boltax, P. M. French, M. H. Hodge, W. R. Jacoby, P. D. Shalek

OBJECTIVES

The principal objectives of this task are to develop characterization techniques and to characterize the uranium carbide and mixed uranium-plutonium carbide materials (powders and pellets) developed and fabricated under Task CDAA-200.

PRIOR WORK

Contract Work

Evaluation equipment and techniques were under continued development. Continued statistical review of analytical chemistry data indicated reductions in total error at the 95% (2σ) confidence level had occurred. Powder samples were examined in transmitted light for size and shape analysis. The Fisher Sub-Sieve Sizer measurement of average particle diameter was being revised to accommodate smaller samples. Sub-micron sieving was also investigated. An immersion density technique for fuel pellets was established utilizing dried xylene as the immersion liquid. Porosity characteristics were also observed microscopically.

Numerous etches were used to aid in the microscopic evaluation of the pellets. An orthophosphoric acid etch and vacuum cathodic etching were found to be satisfactory for delineating grain and phase boundaries. Acetic-nitric, oxalic, and bromine-methanol etches were used to give more specific effects. Point counting of representative photomicrographs was used in conjunction with a projected M-Cr-C phase diagram to confirm analytical compositions. Some Cr_{23}C_6 - and Fe -modified specimens were sent to Los Alamos Scientific Laboratory (LASL) for microprobe and x-ray analysis.

Nine compatibility capsules containing carbide fuels were started on 1,000-hour tests.

Related Work Not Charged to the Contract

Nine mock-up compatibility capsules were used to finalize loading, bonding, and soaking procedures. A sodium migration problem was eliminated by design and procedural changes.

CURRENT PROGRESS

Work performed during this period is reported in seven sections, namely: Analytical Chemistry, Metallography and Point Counting, Powder Characterization,

Immersion Densities, Thermogravimetric Analyses, Presentation and Discussion of the Microprobe Evaluations Undertaken at LASL, and Related Work not Charged to the Contract. The bulk of the characterization effort continues in the fields of analytical chemistry and metallography.

Analytical Chemistry

Routine analytical chemistry support has continued throughout this period. During the early part of the period, analyses on Cr_{23}C_6 -modified pellets gave nitrogen values up to 3,000 ppm, indicating that either gross contamination of samples was occurring or the Nitrox unit was not functioning correctly. The results of sampling numerous batches under varying atmosphere conditions did not permit definite conclusions to be drawn regarding the contamination, and, consequently, it was decided to run a check-out on the Nitrox unit.

Samples of UN, which were supplied by AEC, New Brunswick Laboratory, were analyzed in the Nitrox unit in the Plutonium Laboratory. New Brunswick estimated the nitrogen content to be 8% plus, and Kjeldahl analyses undertaken at the WARD Waltz Mill laboratory gave results of 8.57 and 8.53%, respectively. BrF_3 -analysis gave a result of 8.59% nitrogen.

Five nitride samples were then analyzed on the Nitrox unit and gave nitrogen figures ranging from 5.9 to 9.0% with an average value of 7.4%. At the 95% confidence level, this would mean that the coefficient of variation would be + 35.2%, and the nitrogen recovery, based on a nitrogen content of 8.55%, is 87%.

It was felt that these figures indicated the Nitrox unit to be performing satisfactorily, and the variation in nitrogen figures found with UC and (U,Pu)C samples was genuine.

Since that time, faults have developed in the Nitrox unit, necessitating its shutdown. The faults were found to be in the analyzing circuits and required the replacement of several complex components. This has been undertaken, and the Nitrox is now operational. However, due to this shutdown, there is a backlog of oxygen and nitrogen analyses.

No problems have been encountered in the air box, and C determinations, oxidation of carbide scrap, and preparation of fusion samples for x-ray fluorescence analysis have not been interrupted throughout the period. Analytical chemistry results obtained on Cr_{23}C_6 -modified (U,Pu)C fuel batches for EBR-II pins are shown in Table 5. Examination of the fabrication histories of these batches is currently being undertaken in an attempt to determine the cause for the considerable increase in W content observed in the last three batches.

Metallography and Point Counting

Metallographic examination of potential EBR-II fuel batches for irradiation has continued during this quarter. Recent emphasis on the metallography of hyperstoichiometric and stoichiometric (U,Pu)C fuel has necessitated the development of new etching techniques and modification of existing ones. A distinct difference in etchant response of the two fuel types has been observed.

Table 5

Analytical Chemistry Data on Cr₂₃C₆- Modified (U,Pu)C Fuel Batches

Batch No.	C (wt%)	O (ppm)	N (ppm)	C _E (wt%)	Pu (wt%)	Cr (wt%)	W (wt%)	Total Metallic Impurities Excluding W (ppm)
502	4.72	3,015	281	4.97	17.61	2.71	0.29	798
503	4.59	4,660	960	5.06	18.18	3.30	0.37	871
511	4.56	2,690	548	4.81	16.67	3.89	2.83	900
514	4.43	3,425	227	4.71	17.31	1.66	1.35	726
518	4.52	3,800	235	4.83	16.77	3.12	1.32	857

An electrolytic etch in 2:1 acetic:nitric acid superimposed over a chemical swab etch of 85% orthophosphoric acid has given good grain delineation with hyperstoichiometric fuels. Grain contrast has been further improved by giving samples a final swab etch with wet methanol. This latter etch gives grain coloration. A typical microstructure of such a sample is shown in Figure 3, which has a magnification of X600. It is obviously difficult to obtain an accurate point count of the volume of secondary phases, but it is estimated that there is between 15 and 20%. This batch has a carbon equivalent (C_E) of 5.05 wt% and consequently contains more secondary phase than would be expected. However, this sample does contain W, which appears as large white inclusions, as shown in Figure 4 (magnification X300), and this would undoubtedly complicate the structure. The W content of this batch is not known as yet.

Stoichiometric fuel samples to be etched have not responded very satisfactorily to the above approach, however, experience to date indicated that a light electrolytic etch in 2:1 acetic:nitric acid for cleaning and polishing purposes prepares the sample for a vacuum cathodic etch, which defines grain boundaries.

Typical microstructures of five $Cr_{23}C_6$ -modified (U,Pu)C batches are shown in the next sequence of illustrations (figures 5 through 9). These are potential EBR-II fuel batches, and their analytical chemistry data are given in Table 5.

The average grain size of $Cr_{23}C_6$ -modified (U,Pu)C samples is noticeably higher than that for the unmodified. Typical figures for the modified and stoichiometric are 15 to 30 μ and 4 to 5 μ , respectively.

In addition, the average grain size of the unmodified batches is apparently dependent on stoichiometry. Sufficient data are not yet available to definitely confirm this dependency, but it is apparent that grain size decreases with increasing C_E over the range $C_E = 4.70$ to 5.25 wt%.

Turning to the microstructures of batches 502 and 503 (Figures 5 and 6), both have similar appearances. This is reasonable, as point count analysis indicates the composition of both lie in the MC- Cr_7C_3 - $Cr_{23}C_6$ phase triangle. Both exhibit a uniform grain size and porosity distribution, while the minor phase, which surrounds the (U,Pu)C matrix grains, comprises two additional phases. The structures have the typical appearance of having been sintered in the presence of a liquid phase; both batches were, in fact, sintered at 1550°C for two hours.

Likewise, batches 511 and 518 (Figures 7 and 8) have a similar appearance. Point count analysis reveals that their compositions probably lie in the same phase triangle, i.e., MC- Cr_7C_3 - $Cr_{23}C_6$. Both exhibit reaction rims in the grain boundaries suggesting incomplete peritectic transformation during cooling. Two secondary phases are obviously present.

Figure 9 (Batch 514) shows sharp grain definition with only one second phase present. This agrees well with the analytical data, which indicates that composition lies on the tie line MC- $Cr_{23}C_6$.

(x600)

Figure 3 Batch 516. Hyperstoichiometric (U,Pu)C.
4.73 wt% C; 2,510 ppm O₂; 1,346 ppm N₂.
2:1 Acetic-Nitric Acid Etch.

(x300)

Figure 4 Batch 516. Hyperstoichiometric (U,Pu)C.
4.73 wt% C; 2,510 ppm O₂; 1,346 ppm N₂.
2:1 Acetic-Nitric Acid Etch. Area of Inclusions.

(x300)

Figure 5 Batch 502. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for two hours at 1550°C . 85% Orthophosphoric Acid Swab. Uniform distribution of second phase.

(x300)

Figure 6 Batch 503. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for two hours at 1550°C . Oxalic Acid Etch. Similar structure to batch 502, but with noticeably less second phase material.

(x600)

Figure 7 Batch 511. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for two hours at 1550°C , two hours at 1600°C . Vacuum Cathodic Etch. Reaction rims suggest incomplete peritectic transformation during cooling.

(x600)

Figure 8 Batch 518. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for two hours at 1550°C . 85% Orthophosphoric Acid Swab. Reaction similar to those observed in batch 511. Two secondary phases apparent.

(x300)

Figure 9 Batch 514. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for two hours at 1550°C . Oxalic Acid Etch. Sharp grain definition with little second phase material.

Further work on batch phase identification, through chemistry and metallographic point counting, has led to recalculation of some previously reported data.

Figure 10 shows a portion of the (U,Pu)-Cr-C ternary system. As stated in the previous quarterly[1], this has been extrapolated from the U-Cr-C diagram.[2] The diagram is intended to show tie lines for the quenched-in solid phases while taking into account the work of Anselin, et al [3] concerning the homogeneity ranges of UC and PuC.

Receipt of W and Cr analytical figures, which were previously estimated, has led to relocation of batches 511 and 514 as shown. W is considered to function essentially as Cr and thus contribute to a Cr equivalent (Cr_E). Table 6 compares the secondary phase volumes predicted from chemistry with those obtained by metallographic point count techniques. The previously reported values for batches 511 and 514 are also shown. The two secondary phase volumes calculated for batch 511 differ markedly due principally to an abnormally high W figure. Unfortunately, a complex microstructure rendered an accurate secondary volume point count impossible.

It is interesting to note that chemical analyses suggest that no free U, Pu, or Cr metal should be present in any of these five batches. The intended constitution of Cr-modified (U,Pu)C fuel is $(U,Pu)C + Cr_{23}C_6 + Cr$.

A pellet from batch 502 was sent to LASL for microprobe analysis (see Page 28 for details). A small amount of fissile material was detected as the minor secondary phase, suggesting that the final phase composition should lie just within the MC-MCrC₂-Cr₃C₂ phase field. This adjustment falls within the limits of analytical error and is, in fact, suggested by the higher secondary phase volume determined from metallographic point counting.

Metallographic data for three other Cr₂₃C₆-modified batches were in satisfactory agreement with the microprobe data. As for batch 502, however, the analytical chemical compositions were all lower in equivalent carbon than indicated by the microprobe results. One or both of the following could be responsible: (a) the pertinent phase fields should be lowered on the diagram by lowering the upper limit of the MC phase area, or (b) analytical chemistry errors tend to accumulate to give low indicated C_E .

Particle Characterization

Particle size distribution in milled (U,Pu)C powders has been investigated by means of sieves and microscopy. Both wire-mesh and micro-mesh sieves having the sizes 53, 44, 37, 30, 20, 10, and 5 μ were used. The powder feed to the 53 μ were used. The powder feed to the 53 μ sieve had previously passed through a 74 μ sieve, this being the smallest sieve size used during the routine fabrication process.

The 2-gram powder samples were dry sieved down to 37 μ , and an absolute alcohol carrier used for the remaining four micro-mesh sieves. A typical distribution curve is shown in Figure 11.

Table 6

Calculation of Secondary Phase Volumes from
Point Counting Techniques and Analytical Chemistry Data

Batch No.	Point Counted Secondary Phase (wt%)	Total Calculated From Analytical Chemistry Data Secondary Phase (vol%)	Predicted Secondary Phases
502	7.3	6.0	Cr_7C_3 , Cr_3C_2
503	5.6	7.4	Cr_7C_3 , Cr_3C_2
511	7.0 ^b	9.5	Cr_7C_3 only
511 ^a	7.0	4.9	Cr, Cr_{23}C_6
514	3.5	3.7	Cr_7C_3 , Cr_{23}C_6
514 ^a	3.5	3.65	Cr, Cr_{23}C_6
518	8.8	7.9	Cr_7C_3 , Cr_{23}C_6

a Previously reported figures, based on unmodified phase diagram and assumed W and Cr figures.

b Point count hindered by complex structure and therefore approximate.

Figure 11 Particle Size Distribution of (U,Pu)C Powder (-325 MESH)

3038-12

The curve represents a powder consisting of more than 50 wt % of less than 10 μ . Of this, 18.5 wt % was retained on the 5 μ sieve, i.e., between the 5 and 10 μ size, and 45.5 wt % had a particle size less than 5 μ .

The various fractions were checked optically, and little subsize retention was observed down to the 10 μ mesh sieve. Figure 12 shows particles from the 20 to 30 μ fraction. These have the rounded angular configuration expected of a milled product. However, agglomeration of the sub-10 μ particles complicated microscopic evaluation. Nevertheless, overall results imply approximate agreement with the Fisher equivalent spherical diameter findings of $\sim 3\mu$. [4]

Modifications to the Fisher Sub-Sieve-Sizer have been delayed by acquisition of accessories, such as a depth gage, which will replace the direct-reading chart. It is anticipated, however, that the apparatus will be assembled and ready for testing early in the next quarter.

Immersion Density Evaluations

Typical geometric and immersion density data for Cr₂₃C₆-modified and unmodified fuel batches are shown in Table 7. There is no apparent difference for the parameters listed between the two types of fuel. The only notable discrepancy occurs in batch 514, where there is a large difference in the geometric and immersion densities. This is presumably caused by surface microcracking, which was not detected while the geometric density of the pellet was being determined.

Thermogravimetric Studies on Binder Effects

A program was initiated to study the burnout characteristics of a variety of binders that could be used in the carbide fabrication programs. These included Carbowax 4000, Cranco, stearic acid, and Acryloid.

The program was initiated with studies on burnout characteristics of Carbowax, the principal binder used to date in the carbide fabrication programs.

Uranium carbide pellets containing one wt % binder were prepared by WARD and run by the Materials Systems Laboratory of NFD. In an atmosphere of pure, flowing argon having <10 ppm total impurities and at a heating rate of 4°C/minute, the binder came off rapidly over the range 300 to 350°C, with no further loss recorded up to 800°C. For duplicate runs, the recorded weight losses were 56 and 59 wt % of the total Carbowax added to the powder. This seems to confirm previous indications that a considerable portion of the Carbowax binder reacts with the fuel material during the burnout portion of the firing cycle. Similar runs will be undertaken for pellets containing stearic acid, Acryloid, and Cranco binders, and the effects of adding varying amounts of the binder to the carbide powders will also be studied.

Presentation and Evaluation of Results Obtained in the LASL Investigation

Results are now available on the LASL microprobe and WARD metallographic examination for the carbide pellets and are shown in Table 8.

(x75)

Figure 12

Typical (U,Pu)C particles from a 20 to 30 μ fraction
i.e., those retained on a 20 μ micromesh sieve.
Particle shape typical of a milled product.

Table 7

Typical Average Density Values for Potential EBR-II Fuel Batches

Batch	Fuel Type	Geometrical Density (% Theoretical)	Immersion Density (% Theoretical)	Open Porosity	Closed Porosity
502	C	88.8	90.3	1.9	7.8
503	C	89.0	90.4	3.3	6.3
511	C	87.9	91.7	1.2	7.1
514	C	87.5	94.1	0.8	5.1
518	C	85.8	88.9	7.7	3.4
519	U	86.0	90.6	6.2	3.2
521	U	90.5	93.6	1.2	5.2

Notes: C = Cr_{23}C_6 -modified (U,Pu)C

U = unmodified (U,Pu)C

Table 8

Details of WARD Carbide Pellets Forwarded to LASL for Microprobe Examination

WARD Batch No.	Material	Irradiation Type	Type	Modifier	
				Wt% Added (Cr ₂₃ C ₆ or Fe)	wt% Analyzed (Cr or Fe)
72	(U,Pu)C	GETR	Cr ₂₃ C ₆	5.01	4.18
145	(U,Pu)C	GETR	Cr ₂₃ C ₆	4.00	1.92
147	(U,Pu)C	GETR	Cr ₂₃ C ₆	3.00	2.85
502	(U,Pu)C	EBR-II	Cr ₂₃ C ₆	2.00	2.71
148	(U,Pu)C	GETR	Fe	2.50	2.70
152	UC	GETR	Fe	2.71	2.17
154	(U,Pu)C	GETR	Fe	1.51	1.48
168B	(U,Pu)C	--	Fe	3.14	--

Details of the extra phases found by LASL in Cr_{23}C_6 -modified (U,Pu)C samples are shown in Table 9. With this type material, WARD has originally specified that the only secondary phases should be $\text{Cr}_{23}\text{C}_6 + \text{Cr}$. With Fe-modified (U,Pu)C the secondary phases should be (U,Pu) $\text{Fe}_2 + \text{Fe}$. A summary of the examination of the four Cr_{23}C_6 -modified (U,Pu)C pellets is given in Table 10.

There was a variation in the constitution of the secondary phases in the Cr_{23}C_6 -modified material, as shown in Table 10. However, three out of four modified carbides had uniform concentrations of U and Pu within the (U,Pu)C phase. LASL also observed that no two pellets had identical microstructural appearances; this concurred with WARD's metallographic observations.

The microprobe data appear consistent with the phase relations shown in the proposed phase diagram of Figure 10. Phase A (Table 9) logically corresponds to the MCrC_2 phase shown in the diagram, while phase B is more than likely Cr_3C_2 . If we assume the diagram is correct, it is impossible under equilibrium conditions for MCrC_2 and Cr_{23}C_6 to exist together. The only chromium carbide that can be in equilibrium with MCrC_2 is Cr_3C_2 . Compositions 72 and 502 are thus indicated as falling within the MC- MCrC_2 - Cr_3C_2 compositional triangle, whereas compositions 145 and 147 fall approximately on the MC- MCrC_2 tie line.

The occurrence of an oxide third phase in composition 147 indicates that the solubility of oxygen in the MC phase is lower than anticipated at this point on the diagram. This batch had a higher than normal oxygen content because the material was recycled. The intensity data show that the oxide phase is richer in Pu than the matrix MC phase, in contrast with the MCrC_2 phase which is richer in uranium.

The Fe-modified pellets indicated widely varying microstructural features, as with the Cr_{23}C_6 -stabilized material. In each of the three (U,Pu)C pellets, U and Pu concentrations were very variable within a given pellet. The microstructure of batch 168B, which was not a potential fuel, has previously been discussed.[5]

The major "impurity" phase in the Fe-modified samples contained more Fe and less Pu and U than the fuel matrix phase, while the minor "impurity" phase was rich in Pu and contained less Fe, U, and C. The one pellet of Fe-modified UC (batch 152) indicated eutectic formation in the second phase as one would expect from the work of Briggs, et al.[2]

The following comments summarize the remainder of the LASL findings.

- a. In all Cr_{23}C_6 -modified samples, amounts of Cr, usually estimated to be less than 0.5 wt % were found in the (U,Pu)C matrix.
- b. With all Fe-modified samples, Fe was found to be heterogeneously distributed throughout the matrix in estimated amounts ranging from "trace" to 5%.
- c. With batch 72, the microhardness of the (U,Pu)C matrix ranged from 1,332 to 1,478 kg/mm^2 , i.e., approximately twice that of unmodified (U,Pu)C.

Table 9

Secondary Phases Found in Cr_{23}C_6 -Modified (U,Pu)C Samples

Phase	X-Ray Intensity (Electron Microprobe)			
	Pu ^a	U ^a	Cr ^b	C
A	40%	75%	5-15%	Present
B	1%	1%	90%	Present
C ^c	130%	85%	(Oxygen)	--

a Referred to intensity from (U,Pu)C

b Referred to intensity from Cr metal

c LASL considers phase C is probably an oxide.

- With other samples there was a far wider range in microhardness in the (U,Pu)C matrix. However, it is known that small amounts (0.9 wt %) of W increases the hardness of UC^[6] (possibly for Cr additions also). Unfortunately, the only accurate figure for W available to date and relevant to the LASL evaluation, is for batch 502, which contained 0.29 wt %. However, in Table 5, Page 18, it will be seen that there is a considerable variation in W and Cr content from one batch to another. It is hoped to have accurate W figures for other relevant batches shortly. The microhardness of the secondary phases in Cr₂₃C₆-modified material ranged from 1,378 to 3,386 kg/mm². Examination of Table 10 suggests that the higher values are associated with Phase B (Cr₃C₂) and the lower values with Phase A (M₂Cr₂C₂).
- d. With Fe-stabilized material, the hardness of the matrix ranged from 773 to 1,505 kg/mm², indicating the possible presence of a metallic phase.
 - e. There is no evidence in the fabrication histories to indicate why batch 147 and all the Fe-modified batches should have heterogeneous U and Pu distributions in the matrix.
 - f. The particles of secondary phase in the Cr₂₃C₆-modified (U,Pu)C samples always had a larger grain size than the matrix.
 - g. When W was detected in the Cr₂₃C₆-modified samples, it was always in the presence of Cr, indicating that it could possibly substitute for the latter. It should be observed that in the point count analysis, it has been assumed that W acts like Cr.

The authors would like to thank Dr. R. D. Baker, Dr. J. A. Leary, Mr. C. Metz, and all other LASL staff concerned with the evaluation of WARD carbide samples at LASL. In addition, we wish to compliment them on the excellence and comprehensive documentation of their findings.

Related Work Not Charged to the Contract

Differential thermal analysis (DTA), thermogravimetric analysis (TGA), and thermal dilatometric (T.D.) equipment has now been received from the vendors. The furnaces and supports have been installed. Required maintenance work on the complete installation is expected to start shortly, and it is hoped to have the equipment working with non-active samples during the next period.

Table 10

Summary of Examination of (U,Pu)C- Modified with Cr_{23}C_6
(Data Provided by LASL)

1. WARD Batch No.	72	145	147	502
2. Cr_{23}C_6 added, wt%	5.01	1.92	2.85	1.96
3. Matrix grain size	25	10	10	25
4. Phases: Matrix	Homog.	Homog.	Heterog.	Homog.
Major 2nd	A	A	A	B
Minor 3rd	B	(none)	C	A
5. "Halo" around pores	No	Yes	Yes	No
6. Hardness kg/mm^2				
Matrix	1332-1478	782-1378	1532-1854	627-1288
2nd Phase	2098-3386	1378-1714	1714-2012	1478-3047
7. Presence of W	No	in A	in A little in C	in A

Notes: The (U,Pu)C phase contained some Cr
The W generally was present in large grains

CDA-300 - FUEL ELEMENT FABRICATION DEVELOPMENT

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OBJECTIVES

The principal objectives are to fabricate and characterize the required fuel pin and capsule assemblies for the various irradiation test programs using current state-of-the-art techniques, and to continue development of procedures for producing and evaluating sodium-bonded, uranium-plutonium carbide elements required in future irradiation programs.

The objective of the development work is to improve reproducibility and understanding of sodium bonding procedures such as impurity control of sodium and fuel, efficiency of bonding, and destructive and non-destructive tests.

PRIOR WORK

Pins simulating actual irradiation test pins have been fabricated and evaluated. Development work has advanced in the areas of sodium handling, loading and bonding, end plug closures, characterization tests such as eddy-current trace analysis, radiographic interpretation of internal component positioning, and correlation with destructive examination. Equipment that is adequate for the test programs has been installed, and characteristics and limitations of the equipment have been explored.

Ten irradiation test pins were fabricated, nine of which were inserted in the GETR reactor. All information, procedures, and test data have been documented.

CURRENT PROGRESS

EBR-II Irradiation Test Fabrication

Two irradiation test pins (W2X, W8X) were fabricated and encapsulated. A flow diagram showing all operations involved in manufacture and characterization of pins, tensile specimens, and capsules is presented in Figure 13. The capsules were shipped to EBR-II on September 20. Data sheets have been compiled for all operations. Two control pins were processed along with the fuel pins. These pins contained steel pellets, but all other materials and processing steps were identical to the fuel pins. The two pins were then analyzed for oxygen in the sodium bond, showing 308 and 394 ppm oxygen.

Figure 13 Flow Diagrams showing Operations Involved in Manufacture and Characterization of Pins, Tensile Specimens, and Capsules.

Radiography indicated that all welds were satisfactory, and in addition, revealed an accumulation of fuel powder and small chips around the bottom insulator pellet. However, in no case did this material extend more than half the length of the insulator; this was judged to be an acceptable condition.

Leak testing, eddy current testing of the sodium bond, and dye penetrant testing did not reveal any imperfections. Pellets were sodium bonded in pins in the vibratory furnace that was described in the quarterly reports covering the last half of calendar year 1967 (WCAP-3791-10 to WARD-3791-12). The pins and tensile specimen containers were then bonded in capsules by the new impact bonding furnace. To determine that impact bonding did not harm fuel pellets, three pins containing UC pellets were impact bonded and examined radiographically. No chipping or cracking of pellets was detected. Also, radiographs of capsules W2X and W8X before and after impact bonding showed no change in the pellet silhouettes or quantity of fuel debris.

Profilometer diameter measurements made on the two fuel pins are presented in Figure 14. The equipment, designed and constructed by Nuclear Fuel Division, consists of an LVDT transducer actuated by a ball-point probe riding on the tube surface. The amplified signal is displayed on a strip-chart recorder. Repetitive traces within $\pm 5 \times 10^{-5}$ inches were achieved. Six axial traces were made on each fuel pin at 30° increments of rotation. Diameter variations are less than ± 0.0002 inch from a mean diameter. A very slight tube reduction (roughly 0.0002 inch) seems apparent on the traces in Figure 14 at the top level of sodium in the tube. Traces of future pins will indicate if this is a consistent occurrence.

Two additional EBR-II irradiation pins have been fabricated, and characterization tests are nearing completion. One of these pins contains 0.220-inch-diameter fuel in a 0.230-inch I.D., 0.250-inch O.D. tube; this is the first carbide fuel material of this diameter that has been made available for pin fabrication. Two experimental pins were fabricated with rejected UC insulator pellets of 0.220-inch diameter. Several pins containing steel pellets and with the above dimensions are in various stages of fabrication and characterization with the main purpose of evaluating the 5-mil radial sodium bond. All other tube-pellet combinations previously fabricated have had at least twice this radial clearance.

New Equipment and Facilities

The large impact bonding furnace was completed and was used for bonding of irradiation test pins as described above. Accelerometer readings measured on the specimen magazine indicate a ten-fold increase in acceleration over the electromechanical vibrator formerly used. With this equipment, it is possible to bond six EBR-II length capsules during one run. The Erik model eddy current tester from Magnetics Analysis has been evaluated. The two features desired, low-frequency generated signal and phase discrimination of output signal, were found to be ineffectual for our application of this equipment. The probe supplied was not suited for low-frequency use, and the

Figure 14a Pre-Irradiation Outside Diameter
 Profilometer Measurements of EBR-II Fuel Pin 5
 (Capsule W2X) 9/5/68

3038-9

Figure 14b Pre-Irradiation Outside Diameter
 Profilometer Measurements of EBR-II Fuel Pin 1
 (Capsule W8X) 9/5/68

phase response of defect signals was not sufficiently sharp. The tester has been returned, at least temporarily, to the supplier. Investigation of testers more sophisticated than the presently-used Helitester will be continued.

The dark room has been completed and is in use. Cheswick now has complete facilities for radiography of fuel pins, as well as most of the other characterization tests.

Reclamation of Fuel Pellets from Sodium

An evaporation has been made of the facilities necessary for, and available for, the reclamation of fuel pellets that have been heated in molten sodium. Vacuum distillation removal of sodium has been identified by LASL as the only method that has no deleterious effect on pellets. Until such equipment is installed in the Cheswick box line, an alternative method was developed as follows: two pins containing UC pellets had their cladding mechanically peeled away in a spiral ribbon within a glove box. The resulting "stack" of pellets was scraped as a unit in order to remove enough adhering sodium so that the stack could be inserted into a new tube. After completing fabrication and bonding, a radiographic study showed that no pellet chipping had occurred. A disadvantage of this procedure is that some increase can be expected in oxygen contamination of the surface of the sodium-covered pellet stack during handling in the ambient glove box atmosphere. But, in cases where only transference of the contents from a damaged tube to a new tube is desired, the method is ideal, provided special atmosphere control is employed in a glove box to protect the sodium.

Oxygen Contamination of Sodium

Simulated fuel pins have been analyzed in the continuing program on decreasing the oxygen contamination levels in sodium. Four pins were fabricated in an identical manner. Two pins were bonded at 1000°F while the other two were not. The bonded pins had 365 and 367 ppm oxygen in the sodium, while the unbonded pins had 211 and 248 ppm oxygen. These are only the initial results of a parametric program in which two levels of oxygen and three temperatures of bonding are studied. For this reason, discussion of the results will be deferred until the next quarter when completion of the entire study is expected.

CDA-400 - THERMAL FLUX IRRADIATION STUDIES

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OBJECTIVE

The objective of this task is to evaluate the in-pile performance of sodium-bonded (U,Pu)C fuel elements. In-pile tests will be performed in currently available thermal flux facilities. The post-irradiation test results are intended to provide:

- a. preliminary performance test of the sodium-bond concept in carbide fuel pins;
- b. a comparison of fuel performance characteristics, such as fission gas release rates and fuel swelling rates, for various modifications of (U,Pu)C fuel;
- c. a comparison of fuel-cladding compatibility at fuel surface temperatures in the range of expected large plant operating conditions;
- d. data to supplement the fast flux test results; and
- e. early performance information for re-assessing the direction of future fast flux tests.

PRIOR WORK

Nine (U,Pu)C fuel pins of varying compositions were fabricated and assembled into three instrumented, double-containment capsules. These capsules were inserted into the reactor in March, 1968, where they have been operating continuously. Methods and facilities for post-irradiation shipping and examination have been investigated in preparation for making detailed arrangements for the program. Preliminary design work on a second phase of this task was initiated, but additional effort was postponed.

CURRENT PROGRESS

In-Pile Status

The nine fuel pins presently in GETR continued to perform satisfactorily throughout the quarter. As indicated in the last quarterly report, capsule W2G was moved from pool position X3 to W5 at the start of cycle 98 to achieve more consistent operating conditions and maintain V-RAFT control throughout the cycle. The change of position was successful, as illustrated in Table 11, by the increased average power ratings of W2G fuel pins in cycle 98. However, the W1G pins started to lose power toward the end of the cycle, resulting in a reduced average power rating shown in the same table. This loss was expected in the latter cycles of irradiation as significant burnups were experienced. [1]

Table 11

Summary of Average Power and Integrated Fluxes in Fuel Pins

Fuel Pin	Fuel Type ^a	Design Peak Power Rating (kW/ft)	Average Power (kW/ft)					Integrated Fluxes (n/cm ²) Thru Cycle 98			
			Total	Average	Power (kW/ft)			Fuel	Cladding		
			Cycle 94	Cycle 95	Cycle 96	Cycle 97	Cycle 98		Thermal x10 ²⁰	Thermal x10 ²⁰	Fast x10 ²⁰
W1GL	U	12.2	7.4	8.7	9.0	9.4	8.9	1.1	2.0	3.6	1.1
W1GM	C	23.4	16.2	17.6	18.1	19.2	17.8	2.4	4.2	7.3	2.1
W1GH	C	28.1	23.4	25.2	25.5	28.7	25.9	3.7	6.3	10.8	3.2
W2GL	F	12.2	5.4	6.2	6.3	6.5	7.3	0.8	1.5	2.8	0.8
W2GM	C	23.4	12.8	13.7	14.3	14.8	16.5	1.9	3.4	6.2	1.7
W2GH	U	28.1	21.1	21.8	22.3	25.6	27.1	3.2	5.6	10.2	2.8
W3GL	C	12.2	--	4.4	5.5	5.4	6.5	0.6	1.1	1.7	0.5
W3GM	C	23.4	--	12.5	12.8	12.5	15.0	1.5	2.7	4.1	1.2
W3GH	F	28.1	--	20.6	20.4	22.6	25.9	2.6	4.6	7.1	2.0

^a U - Unmodified, C - Cr₂₃C₆ modified, F - Fe modified

^b Thermal - E<0.17 ev, Fast - E>0.18 Mev, Epithermal - 0.17 ev ≤ E ≤ 0.18 Mev

The status of the fuel pins is summarized in Tables 11 and 12. The capsules have completed 5 (W3G) and 5-1/2 (W1G and W2G) cycles of irradiation at the end of cycle 99 (September 15). The calculated data reported from the test site indicate W1GH has an average accumulated burnup of 45,472 MWD/T (more than 90% of the planned burnup). In view of this, capsule W1G, and probably W2G, will be withdrawn from the reactor at the end of the present cycle 100 about October 20. The fact that these capsules will have achieved 50,000 MWD/T burnup in 6-1/2 cycles instead of the planned 7 or 8 cycles [7] is attributed to the high operating efficiency of GETR at a consistent 50-MW operating power.

Interim Examination

At the end of cycle 98, the capsules were removed from the reactor to take neutron radiographs and examine the condition of the fuels at an intermediate exposure. The capsules were maintained in a vertical position while radiographic exposures were made of the "H" and "M" pins in each of two directions, 90° apart for each capsule. The "L" pins were not included because a single exposure could only accommodate a 15-inch length, and the operating power levels and burnups of these pins were of lesser interest. Along with the X-radiograph [Figure 15 (a)] a reproduction of the W1GH neutron radiograph is shown in Figure 15 (b). Figures 15 (c) and 15 (d) are enlarged (~3X) reproductions of the fuel columns in W1GH and W3GH, respectively.

As indicated by the figures, the fuel pellets were transversely and longitudinally cracked, with the fractures in the "M" pins appearing more separated than those in the high-power pins. Measurements of length and diameter were made on apparently uncracked individual pellets. The measurements unaffected by fractures were used to calculate the preliminary swelling rates shown in Table 13. These data support the expectation that Cr₂₃C₆-modified fuel would have the lower rate of the three fuels examined. The results, adjusted by +80°C to account for thermal flux depression effects, are shown in Figure 16 for comparison with a previously established curve. The swelling rate for the hottest Cr₂₃C₆-modified fuel pin (1H) is somewhat higher than expected. This may be due, in part, to the large experimental errors. Also, although the swelling measurements were based on apparently solid pellets, undetected microcracks may have been present, resulting in higher measured values.

Within the limits of detection, no incompatibility between fuel and cladding was observed. Analysis of the results is continuing and additional neutron-radiographs are planned upon removal of the capsules.

Figure 15 shows some high density longitudinal indications in the 2nd, 3rd, and 4th pellets from the bottom. These indications are fairly symmetric with corresponding marks in another view oriented 90° to this one, which indicates a concentric ring of high density material. Although not well characterized, it appears that some segregation of fissile material may have occurred, possibly due to thermal gradients in the fuel. This was only observed in the Fe-modified (W3GH) and unmodified (W2GH) pins; it was not seen in the C₂₃C₆-modified fuel. Complete characterization of these indications and better defined swelling rates will be determined during the planned post-irradiation examination.

Table 12

Summary of Burnup Accumulation in Fuel Pins

Fuel Pin	Fuel Type ^a	GETR Pool Position	Nominal Design Power Rating kW/ft	Average Burnup in GETR, MWD/T								% of Goal Achieved	Anticipated Withdrawal	
				Cycle 94	Cycle 95	Cycle 96	Cycle 97	Cycle 98	Cycle 99	Accum.	Goal		End of Cycle	Date
W1GL	U	X2	10	1174	2601	2664	5041	2365	3131	14,967	20,000	74.9	100	10/20/68
W1GM	C	X2	20	2666	5462	5499	6426	4883	6370	31,306	35,000	89.4	100	10/20/68
W1GH	C	X2	30	3863	7860	7786	9642	7169	9152	45,472	50,000	90.9	100	10/20/68
W2GL	F	W5	10	889	1906	1896	2155	1999	2411	11,256	20,000	56.3	100	10/20/68
W2GM	C	W5	20	2049	4152	4265	4832	4440	5234	24,972	35,000	71.3	100	10/20/68
W2GH	U	W5	30	3361	6544	6567	8291	7235	8513	40,511	50,000	81.0	100	10/20/68
W3GL	C	X11	10	--	1350	1659	1780	1755	2596	9,140	20,000	45.7	101	11/24/68
W3GM	C	X11	20	--	3915	3912	4202	4170	6159	22,358	35,000	63.9	101	11/24/68
W3GH	F	X11	30	--	6317	6150	7493	7058	10,171	37,189	50,000	74.4	101	11/24/68

^a U = UnmodifiedC = Cr₂₃C₆-modified

F = Fe-modified

FIGURE 15 (a) X-RADIOGRAPH OF WIGH-PRE-IRRADIATION
 (b) NEUTRON RADIOGRAPH OF WIGH-POST-IRRADIATION
 (c) NEUTRON RADIOGRAPH OF FUEL COLUMN IN WIGH-POST-IRRADIATION
 (d) NEUTRON RADIOGRAPH OF FUEL COLUMN IN W3GH-POST-IRRADIATION

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Table 13
Preliminary Swelling Rates
of (U,Pu)C Fuels

Fuel Type ^a	C	C	U	F
Nominal Power Rating (kW/ft)	20	30	30	30
Swelling Rate (% $\Delta V/V/10^4$ MWD/T)				
Minimum	0	2.3	4.7	5.5
Maximum	4.1	5.4	10.0	15.1
Mean	1.9	3.5	7.0	8.2

^a C = Cr₂₃C₆-modified

U = Hypo unmodified

F = Fe-modified

Figure 16 Adjusted Carbide Fuel Swelling

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Fuel Pin Shipment

A cost analysis was performed on the alternative methods of making the post-irradiation shipment of the three radioactive capsules from GETR to LASL. The result indicate rental of a cask from and shipping arrangements made by the reactor site is the more economical approach. Therefore, negotiations to accomplish this are being pursued.

Post-Irradiation Examination

In accordance with AEC directions, the post-irradiation examination will be conducted in the LASL hot cell facilities. Initial contact has been made with this laboratory, and arrangements are presently being made for a meeting between WARD and LASL to establish their capabilities in accomplishing a proposed examination, which could begin in March of 1969.

General Task Activities

Due to reduced funding this fiscal year, Phase II of this task was postponed in favor of the fast flux experiments. As a result, the Fiscal Year 1969 work program was revised.

The first complete draft of the design and fabrication report for the nine fuel pins in GETR was completed. It was distributed within WARD for comments and will be published in the next quarter.

CDAA-500 - FAST FLUX IRRADIATION STUDIES

A. Biancheria, B. Harbourne, A. Lipps

OBJECTIVE

The objective of this task is to evaluate the performance of sodium-bonded (U,Pu)C fuel pins in a fast flux. Various fuel types will be tested to study the effects of composition variations with the objective of defining a satisfactory carbide fuel for LMFBF application.

PRIOR WORK

The program of testing under this task was revised to emphasize basic fuel technology development. The new Phase I program contains 16 pins operating at nominal ratings of 20 and 30 kW/ft. Three fuel types--stoichiometric (type S), hyperstoichiometric (type H) and Cr-modified (type C) fuel--in approximately equal numbers will be tested. Much of the detailed pin and capsule design was completed.

CURRENT PROGRESS

Fuel Pin Design

The thermal, nuclear, and mechanical design of the Phase I capsules has been completed. The revised design and hazards report was completed and is in reproduction. Detailed engineering drawings have been completed for all except the high-burnup pins. The 16 Phase I fuel pins have been designed to operate at nominal ratings of 20 and 30 kW/ft to burnups of 20 to 30,000, 40 to 60,000 and 80 to 100,000 MWD/T. The design criterion of no fuel-cladding mechanical interaction has been applied to all pins. The preliminary results of the interim examination of GETR test pins (CDAA-400) suggest that this condition will be met in the low and medium burnup pins with perhaps some restraint above 90,000 MWD/T.

AEC Negotiations

Approval-in-principle was granted to the Phase I program during August. The approval for the high rating pins was on a "space-available" basis. Close liaison has been maintained with the AEC on selection of fuel types to be irradiated.

Fuel Pin Fabrication

Two fuel pins, W2X and W8X, were fabricated, encapsulated and shipped to Idaho. The fabrication and inspection data package was also prepared and sent to the EBR-II site.

Nominal operating conditions for these pins are 50,000 MWD/T at 25 kW/ft for W2X and 25,000 MWD/T at 30 kW/ft for W8X. The burnup targets are currently being examined and may be revised.

Chemical analyses of the fuel gave the following (in at.%):

W2X: 44.0 U,Pu, 48.3 C_E*, 7.3 Cr

W8X: 45.2 U,Pu, 48.2 C_E*, 6.1 Cr

*C_E, carbon equivalent is defined by:

$$\text{at.\% C}_E = (\text{at.\% C}) + (\text{at.\% O}) + (\text{at.\% N})$$

From the equilibrium diagram, both fuels should contain (U,Pu)C and chromium carbides. The non-fissile phases represent 5 to 7 vol% of the fuel.

This fuel does not conform to the original specification for Cr-modified (U,Pu)C which should contain Cr₂₃C₆ and Cr as second phases. In the absence of Cr metal, the temperature-limiting (U,Pu)C-Cr-Cr₂₃C₆ pseudo-ternary eutectic cannot form and the minimum melting point in the system is raised to about 1500 to 1600°C. Under the test conditions, this fuel composition is expected to give satisfactory performance.

All the interpretation of the structures of these fuels is based on the U - Cr - C system. Work is continuing by analysis and metallography at WARD and by D.T.A. and electron microprobe analysis at LASL to elucidate further details of the (U,Pu)-Cr-C system.

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