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THE DESIGN AND FABRICATION OF
SODIUM-BONDED (U,Pu)C FUEL PINS
FOR TESTING IN EBR-II

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SECTION 1

INTRODUCTION

Studies conducted by Westinghouse Advanced Reactors Division (WARD) have demonstrated that a sodium-bonded, mixed carbide fuel pin concept could be used as the basis of LMFBR designs to yield a high efficiency, short doubling time system.^[1] A carbide-fueled core would be superior to an oxide-fueled core in terms of linear power generation and doubling time.

The higher power rating of a carbide core is mainly attributable to the high thermal conductivity of the fuel, about 6-8 times greater than that of oxide fuels. The use of sodium bond between the fuel and the clad would minimize fuel temperatures and enhance design flexibility. The design criterion of little or no fuel melting used for oxide fuels must be replaced by limits obtained by considering the heat transfer capabilities of the coolant system. Peak linear ratings of 30-40 kw/ft (cf. 15-17 kw/ft for oxide fuels) are attainable.

Prior to the deployment of a carbide-fueled core, three major developments must be solved. The first of these is swelling accommodation, which in principle can be achieved by providing a sufficiently large sodium bond volume. The second problem is that of fuel-clad interaction. Metal (U,Pu)-cladding reactions produce low-melting point eutectic alloys and higher carbides (M_2C_3 and MC_2) may cause cladding carburization and cladding embrittlement. The third problem is the behavior of the sodium bond. Fission gases released from the fuel may form bubbles in the bond impeding heat transfer causing overheating of the fuel.

The primary objectives of the experimental irradiation program described in this report are to make a preliminary evaluation of the operational problems outlined above and to provide quantitative data to guide further fuel pin and core design studies. The tests form part of the national program to study carbide fuel technology.

Some preliminary data on the effects of fast neutron exposure of vanadium alloys in sodium will also be obtained. All work on this program was sponsored under AEC Contract No. AT(30-1)-3791 and is reported in Section 10.

SECTION 2

SUMMARY OF PRIOR EXPERIMENTAL DATA

2.1 INTRODUCTION

A number of investigations have been made into the characteristics and behavior of experimental carbide fuels. A summary of relevant experimental data which were used to guide the current experiment design is presented below.

COMPATIBILITY STUDIES

Hypostoichiometric UC fuels were found to react with stainless steels at temperatures above 750°C.[2] The reaction was believed to be caused by the formation of a liquid eutectic between uranium and iron and nickel. The presence of plutonium would reduce the eutectic temperature and so enhance the reaction. If this type of reaction occurred it would lead to rapid fuel pin failure and would therefore eliminate hypostoichiometric fuels as major candidates for LMFBR use.

Second phase carbides in hyperstoichiometric fuels have a higher carbon activity than "stainless steel carbides" and may therefore carburize the cladding causing embrittlement that may reduce the endurance of the fuel pin. Carbon transfer from UC₂ to stainless steel has been found to be particularly rapid in the presence of a sodium bond.[13] Oxygen contamination of the sodium, which is almost inevitable, most likely plays an important role in the transfer process.

In mixed carbides, excess carbon forms (U,Pu)₂C₃, which is more stable than the UC₂ which forms in hyperstoichiometric UC fuel. Significant carbon transfer from the mixed sesquicarbide has been observed,[4] but to a lesser extent than from the higher carbides in hyperstoichiometric UC material. Other workers have reported no reaction between stainless steel and hyperstoichiometric (U,Pu)C fuel unless very large amounts of sesquicarbide are present[5] or if the actual carbon content, as opposed to the equivalent carbon content, exceeds 4.8 w/o.[6]

Most results indicate no reaction between stoichiometric carbides and stainless steels,[7] but recent data show that a slight reaction may occur in the presence of sodium.[8]

A reaction between fuel and cladding, usually obtained under conditions designed to favor reaction, is not adequate reason to reject a fuel pin concept. Unless the reaction seriously impairs fuel pin performance, it may be permitted provided that its existence is accounted for in fuel pin design. It was concluded that a slightly hyperstoichiometric fuel (containing up to 10 vol. % sesquicarbide) sodium bonded to stainless steel cladding represented a viable fuel pin concept suitable for further irradiation testing.

2.2 MODIFIED CARBIDE FUELS

Reactive phases in non-stoichiometric fuels may be eliminated by suitable additions. Improvements in the compatibility behavior of hypostoichiometric UC were observed after making additions of iron and Cr_{23}C_6 .^[12]

Iron combines with uranium metal to form UFe_2 , which is relatively inert towards stainless steel, while the Cr_{23}C_6 addition reacts with free uranium to form UC and chromium metal. In hyperstoichiometric fuels, the Cr_{23}C_6 modification reaction gave $(\text{U,Pu})\text{CrC}_2$ and Cr_7C_3 as the principle products (see Section 6.2).

Although iron-modification improves compatibility behavior, the fuel still contains a relatively low melting point fissile phase which may be deleterious to irradiation behavior. Preliminary results on iron-modified fuels (see Section 2.4) confirmed this hypothesis, and no samples of this type are included in the current program.

The major deficiencies of modified fuels are the dilution of fissile material, the relatively low melting points of the secondary constituents, and the uncertainties over compositional changes during prolonged irradiation. These factors make modified fuels much less attractive than slightly hyperstoichiometric fuels for long term development.

2.3 IRRADIATION BEHAVIOR

Irradiation tests have shown that helium bonded carbide fuels may be irradiated to 100,000 MWd/T^[9] using strong cladding (Nb 0.1%Zr) to restrain fuel swelling. Stainless steel-clad pins failed under similar conditions. Hypostoichiometric fuels show very high swelling rates at center temperatures over 1000°C.^[10] The poor swelling resistance is attributable to the influence of free fissile metal. Higher swelling rates are observed in sodium-bonded fuel pins than in gas-bonded pins at similar fuel temperatures.^[11] The difference is probably due to the absence of clad restraint in the former type. No clear correlation has been obtained between swelling behavior and the composition of hyperstoichiometric carbides.^[10]

The available data yields little that is immediately applicable to the current experiment. However, much of the data can be utilized by making simple adjustments to observed swelling rates to account for non-stoichiometry and clad restraint. The results of this exercise and the design lines obtained for use in this experiment are shown in Figure 1.

2.4 PRIOR WARD IRRADIATION TESTS

Nine small sodium-bonded fuel pins have been irradiated in a thermal flux to 20-50,000 MWd/Te at peak ratings of 12-30 kw/ft. Iron-modified, chromium-modified, and unmodified hypostoichiometric $(\text{U,Pu})\text{C}$ were tested. Estimates of fuel swelling from neutron radiographs showed the chromium-modified fuel to be significantly better than the other types tested^(a). The results for

(a) P. J. Levine, and others - "Irradiation Tests of Sodium-Bonded Mixed Carbide Fuel Pins." Amer. Ceramic Soc. International Symp. Washington D. C., May 1969.

Figure 1 Summary of Carbide Swelling Data

the chromium-modified fuel were largely consistent with the data presented in Figure 1.

Consideration of these results in part led to the definition of the fast flux test program which emphasizes a range of hyperstoichiometric fuels with a few additional tests on Cr_{23}C_6 -modified fuels.

SECTION 3

SELECTION OF EXPERIMENTAL PARAMETERS

To attain experimental operating conditions of direct relevance to LMFBR design, ratings of 20-30 kw/ft were established as targets. Peak cladding temperatures were to be as near to 650°C as possible. To obtain target burnups of 50-100,000 MWd/Te in reasonable times, high specific ratings (~ 200 watts.g⁻¹) were required.

The characteristics and operational limitations of the EBR-II reactor required some compromise of the experimental targets. To maximize the safety of the experiment, tests were designed for a MkA-19 encapsulated subassembly. In this vehicle, the fuel pin diameter is limited to 0.300 inches. Thus, the fissile loading and the linear rating of the fuel pin are limited for a particular core location. Using fully enriched ($\sim 93\%$ U-235) uranium in (U₈Pu₂)C, a row 3 position is required to obtain a peak rating of 30 kw/ft. The maximum capsule wall temperature, including all hot channel factors may not exceed $\sim 600^\circ\text{C}$ (1105°F). (This limit has since been removed but was used in the experiment design). Consequently, nominal cladding temperatures above 550°C are difficult to attain.

Target experimental parameters were as follows:

- Peak linear ratings: 20 and 30 kw/ft
- Burnup 25,000 and 50,000 MWd/T
- Peak cladding temperatures 500 and 550°C

It was appreciated that the requirement of a row 3 position for the higher power pins would be difficult to meet and that if it proved necessary to move to row 4 the peak attainable rating would be ~ 25 kw/ft.

Several fuel compositions were chosen for testing with the objective of studying the effect of sesquicarbide content on pin performance and the comparison of chromium-modified fuels with unmodified hyperstoichiometric fuels.

Type 316 stainless steel cladding was selected for all pins. In most cases the cladding was in the solution-treated condition, but two pins were to use 20% cold worked material.

Nominal pin design and operating parameters are shown in Table 1. The original program was curtailed and only those pins indicated in Table 1 were actually fabricated. In subsequent sections, only the details of the fabricated pins will be presented.

Small vanadium-alloy tensile specimens were chosen to utilize the spare core space below the fuel pins left because of power output restrictions on the height of the fuel column. The vanadium-alloy specimens will be immersed in sodium at $\sim 375^{\circ}\text{C}$ during the test.

Table 1. Nominal Pin Design Parameters

Capsule Number	Fuel Type	Density (%)	Cladding Type	Pin O.D. (in.)	Wall Thickness (mils)	Pellet Diam. (in.)	Active Column Height (in.)	Peak Nominal Rating (kW/ft)	Target Burnup MWD/T $\times 10^{-3}$
W2X	C	89	ST 316	0.300	12	0.244	9.50	24.5	50
W8X	C	89	ST 316	0.300	10	0.260	9.50	28.5	25
W3F	H	89	ST 316	0.300	12	0.250	9.50	30.0	50
W4F	H	94	ST 316	0.300	12	0.250	9.50	31.0	25
W5F	H	89	ST 316	0.250	10	0.220	9.50	20.4	25
W6F	H	89	ST 316	0.250	10	0.220	9.50	20.4	50
W7F	H	89	CW 316	0.300	12	0.250	9.50	30.0	25
W8F	H	94	CW 316	0.300	12	0.250	9.50	31.0	50
W10F	S	89	ST 316	0.250	10	0.220	9.50	20.4	50
W12F	S	94	ST 316	0.250	10	0.220	9.50	21.6	25

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

Type H = Hyperstoichiometric (U,Pu)C

Type S = Stoichiometric (U,Pu)C

ST 316 = solution treated Type 316 stainless steel

CW 316 = 20% cold-worked Type 316 stainless steel

SECTION 4

PIN AND CAPSULE DESIGN ANALYSIS

4.1 PIN AND CAPSULE DESIGN DESCRIPTION

Capsules were built using standard components. Internal space requirements were met by using Type II bottom end fittings and Type I top end fittings. All components were made from type 304 stainless steel.

A typical fuel pin assembly is illustrated in Figure 2. The assembly essentially consists of a stainless steel tube, end fittings, a pellet column, a hold-down mechanism, and sodium. The overall length of each pin is 28.50 inches. The radial dimensions of the pins are test parameters and vary from one pin to another. The end fittings are flanged to locate the specimen concentrically within the capsule tube. These fittings are also tapered to reduce contact areas and, hence, reduce the potential for gas entrapment during the sodium bonding process. The pins are positioned in the capsule so that the top of the fuel stack coincides with the top of the EBR-II core.

Each fuel pin contains a 9.5-inch-high fuel column comprised of 38 pellets having a nominal height of 0.250 inch each. A depleted UC (about 0.2% U-235) insulator pellet, having a height of 0.375 inch, is located at each end of the fuel column. The compositions of the insulator pellets correspond to those of the fuel pellets with which they are used. Fuel specifications are given in Table 2.

Liquid sodium is used to thermally bond the fuel pellets to the cladding. Sufficient sodium is included to ensure that the pellets remain completely covered throughout the irradiation experiment. A gas chamber above the sodium accommodates fission gases released from the fuel.

The fuel column and insulators are axially retained by a spacer rod and a light compression coil spring. The spring was designed for a maximum loading of 6 G at room temperature. Allowance was made to accommodate component thermal expansion and fuel swelling.

Temperature restrictions prevented the utilization of the full core height for fuel irradiation. It was decided to place vanadium alloy tensile specimens below the fuel pin but in the EBR-II core zone to obtain preliminary data on the effects of irradiation under sodium on mechanical properties. The specimens, based on an ORNL design have a 1.125 inch long, 0.125 inch diameter gage length. Each is contained within an enclosure similar to a miniature fuel pin. The enclosure is vented to the capsule sodium.

- 1 CHECK INDIVIDUAL COMPONENT LENGTH AGAINST DISTANCE FROM TOP OF TUBE TO TOP OF SPACER ASSEMBLY TO ENSURE NO AXIAL CLEARANCES ARE PRESENT AFTER FILLING WITH SODIUM
- 2 WELD TO BE INSPECTED PER. W PROC SPEC 292712
- 3 IDENTIFY PINS BY ELECTRIC ETCHING AT INDICATED LOCATIONS WITH 0.1 in. HIGH CHARACTERS AS SHOWN IN TABLE
- 4 ASSEMBLY TO BE CHECKED BY RADIOGRAPHY OR OTHER METHOD AGREED BY WARD INSPECT FOR VOIDS IN SODIUM METAL BOND AS DEFINED BY WARD REACTOR AND DEVELOPED ENGINEERING.
- 5 WELD BEAD MUST NOT EXTEND 0.003 in. BEYOND TUBE OD
- 6 ASSEMBLY TO BE STRAIGHT WITHIN 0.006 in. OF AXIS
- 7 USE HIGH PURITY SODIUM WITH OXYGEN AND CARBON CONTENT NOT TO EXCEED 50 ppm EACH.
- 8 BACKFILL WITH HELIUM AT ONE ATMOSPHERE AND ROOM TEMPERATURE. OXYGEN CONTENT NOT TO EXCEED 10 ppm
MOISTURE CONTENT NOT TO EXCEED 20 ppm

Figure 2 Typical Fuel Pin Assembly

Table 2. Fuel Specifications

Fuel Type $\longrightarrow$	C (Cr-modified)	H (Hyperstoichiometric)	S (Stoichiometric)
<u>Additive</u>	Cr_{23}C_6	None	None
<u>Composition (wt%):</u>			
Carbon equivalent	4.74	4.90 - 5.10	4.85
U + Pu	91.9 ± 1.0	94.6 ± 0.2	94.9 ± 0.1
Additive	1.5 - 5.5	-	-
<u>Second Phase Constituents</u>	$\text{Cr}_{23}\text{C}_6 + \text{Cr}$	$(\text{U}, \text{Pu})_2\text{C}_3$	None
<u>Theoretical Density (g/cm^3)</u>	13.16	13.63	13.63
<u>Pu in U + Pu (wt%)</u>	20 ± 0.4	20 ± 0.4	20 ± 0.4
<u>U-235 in U (wt%)</u>	93.0 ± 0.1	93.0 ± 0.1	93.0 ± 0.1
<u>Pu-239 + Pu-241 in Pu (wt%)</u>	93.5 ± 0.1	93.5 ± 0.1	93.5 ± 0.1

Table 3. Nominal Design Operating Conditions

Capsule	W2X	W8X	W2E W5F W6F W10F	W3F	W4F W8F	W12F
Grid Position	4n2	3n2	4n2	3n2	3n2	4n2
Peak Rating (kW/ft)	24.5	28.5	20.4	30.0	31.0	21.6
Average Rating (kW/ft)	22.2	25.8	18.5	27.1	28.0	19.6
Flow (GPM/capsule)	2.11	2.95	2.11	2.95	2.95	2.11
Max. Fuel Temperature ($^{\circ}\text{C}$)	1021	1070	914	1133	1104	893
Max. Clad Temperature ($^{\circ}\text{C}$)	557	543	548	558	564	552
Max. Capsule Mean Wall Temp. ($^{\circ}\text{C}$)	525	501	499	516	520	502
Max. Capsule Hot	577	546	543	566	572	527
Spot Mean Wall ($^{\circ}\text{C}$)						
Max. Capsule Heat Flux (watts cm^{-2})	269	313	224	329	341	231
Total Heat (kW)	17.6	20.4	14.6	21.5	22.2	15.1
Coolant Outlet ($^{\circ}\text{C}$)		466	672	478	481	476
Coolant ΔT ($^{\circ}\text{C}$)	122	94	101	107	110	104

The vanadium alloys being tested, VANSTAR 8 and 9, were developed by Westinghouse. Nominal compositions (w/o) are:

	<u>Cr</u>	<u>Fe</u>	<u>Nb</u>	<u>Ta</u>	<u>Zr</u>	<u>C</u>
VANSTAR-8	8.7	-	-	10.1	1.1	.05
VANSTAR-9	-	6.3	5.2	-	1.3	.05

Specimen temperatures during irradiation will be $\sim 375^{\circ}\text{C}$. Fluences of 1.6×10^{22} and 2.7×10^{22} will be received.

4.2 THERMAL DESIGN AND PERFORMANCE ANALYSIS

Peak fuel heating rates, P, were calculated from the relationship:

$$P = 9.039 \times 10^{-13} \sum_{i=1}^N w_i F_i \text{ kw/ft}$$

where:

w_i = grams of fissionable isotope i per cm of length calculated from the data presented in Tables 1 and 2

F_i = fissions per second per gm in isotope i obtained from the EBR-II Guide

N = number of fissionable isotopes. Pu-240 is treated the same as U-238.

Axial power profiles were calculated using a form factor of 1.150.

Axial temperature profiles for all components were calculated using a computer code CAPSULE. The code calculates radial temperature profiles incrementally at a series of axial points, assuming no axial heat transfer. In order to comply with the capsule temperature criterion in the most highly rated capsules, the fuel column had to be restricted to 9.5 inches, located with the top end coincident with the top of the EBR-II active core.

Peak and average power ratings and fuel pin and capsule temperatures are shown in Table 3.

Axial temperature profiles for low power (W5F, W6F and W10F) capsules and high power (W4F, W8F and W11F) capsules are shown in Figures 3 and 4.

Hot channel temperatures are based on a multiplicative combination of uncertainties in parameters. Less conservative techniques are available, but would not have significantly raised the nominal fuel design temperatures.

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Figure 3 Temperature Profiles, Low Power Capsules

Figure 4 Temperature Profiles, High Power Capsules

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Hot channel operating conditions were utilized in experimental design to ensure adequate safety margins, but nominal operating conditions will be utilized in experimental interpretation.

4.3 MECHANICAL DESIGN ANALYSIS

Fuel pins were designed on the basis that at the end of life there would be a minimum fuel-clad gap of 1 mil. Data from Figure 1 were used to estimate the required gap size. In this way the only cladding stresses result from fission gas pressure and thermal gradients. Similarly, capsule wall stresses are due to internal gas pressure and thermal gradients.

Results of the stress analysis are presented in Table 4. All stresses are well below the EBR-II criteria of:

- pressure stress $< 0.25 \times$ yield stress
- maximum thermal stress $< 2.0 \times$ yield stress

Evaluation of creep in the fuel pin cladding, assuming a linear increase in fission gas pressure with time, indicated a total strain of $< 0.2\%$ at the end of life.

Fission gas release, 50%, was assumed in all pins except W2X and W8X, for which 100% release was assumed. The release was estimated from an analysis of available experimental data using a model in which gas release is a joint function of temperature and swelling behavior.^[1] The gas release was calculated for hot channel conditions. The higher gas release for the two Cr-modified fuel pins reflects the greater uncertainty in the characteristics of this material.

4.4 SAFETY ANALYSIS

It is necessary to demonstrate the safety of proposed EBR-II experiments in the event of

- a slowly increasing power transient
- loss of coolant flow followed immediately by a reactor scram
- failure of the pin or the capsule or both.

In the case of the postulated power transient, the reactor power would have to be about three times normal before fuel melting occurred. However, the reactor would scram automatically at 25% overpower.

Coolant flow loss would cause the cladding and fuel temperatures to rise to about 750 and 800°C respectively for a short period (< 1 minute) about 40 seconds after the incident. This excursion would not damage the pin or capsule.

Table 4. Cladding and Capsule Stresses

Capsule	W2X	W8X	W3F	W4F	W5F	W6F	W7F	W8F	W10F	W12F
Target B.U. (MWd/ Te x 10 ⁻³)	50	25	50	25	25	50	25	50	50	25
% Gas Release	100	100	50	50	50	50	50	50	50	50
Fission Gas Vol. (cm ³ stp)	92.2	53.6	59.9	31.6	23.2	46.4	30.0	63.2	46.6	23.3
He Volume (cm ³ stp)	12.67	13.02	12.57	12.98	8.19	8.19	12.57	12.98	8.19	8.19
Pin Gas Pressure (psia)	351	210	266	156	175	304	156	267	306	176
<u>Max. Cladding Stresses</u>										
Pressure (psi)	3464	2149	2685	1575	1305	2265	1575	2695	2290	1320
Thermal (psi)	-16,800	-15,900	-26,300	-27,100	-18,000	-18,000	-26,300	-27,100	-18,000	-18,000
<u>Max. Capsule Stresses</u>										
Pressure (psi)	686	486	540	540	1040	1040	540	540	1040	1040
Thermal (psi)	-17,450	-20,350	-27,900	-28,900	-19,400	-19,400	-27,900	-28,800	-19,400	-19,400
Capsule Wall ΔT (°F)	110	128	182	188	125	125	182	188	125	125

Sodium would be forced out of a failed capsule by internal gas pressure, but the pin-capsule bond adjacent to the fuel column would be retained. Failure of both the pin and capsule would cause a greater sodium loss and the top few fuel pellets would melt. Slumping of the liquid fuel into the bonded zone would cause it to resolidify and a reactor hazard would not be generated. The probability of a double failure is considered to be extremely small.

SECTION 5

FUEL FABRICATION

5.1 INTRODUCTION

Fuel was manufactured and pins assembled in the same facilities used to prepare test pins for the thermal flux irradiation tests. A detailed description of these facilities was presented in the appropriate design and fabrication report. (a)

Hypostoichiometric, stoichiometric, and hyperstoichiometric ($U_{0.8}, Pu_{0.2}$)C were prepared in the plutonium laboratory by arc-melting the constituent elements to form buttons with nominal carbon contents between 4.40 and 4.75 weight percent. After crushing and milling these buttons, binders were added to the resultant powders, and pellets were prepared from the material by cold pressing and sintering. In the case of the hypostoichiometric material, additions of $Cr_{23}C_6$ were made at the powder stage to modify the final pellet structure. Pressures of 20 tsi and firing temperatures up to 1550°C were used to fabricate pellets of the required densities.

5.2 FUEL MATERIALS SYNTHESIS

The synthesis of ($U_{0.8}, Pu_{0.2}$)C pellets for fast irradiation testing was a continuation of work performed by WARD on the fabrication of modified and unmodified carbide pellets for thermal flux irradiation experiments. Details of the fabrication route are given in Figure 5.

Preparation of the $Cr_{23}C_6$ -modified material was undertaken using processing parameters similar to those used in the thermal-flux test work. It was later found that these parameters required adjustment for the production of stoichiometric and hyperstoichiometric materials. These are referred to in the relevant sections below.

5.2.1 Arc Melting

Enriched uranium and plutonium metal impurity levels are given in Table 5. Both metals were cleaned prior to cutting, using a wire brush to remove any oxide from the metal surface. Carbon additions were made from 1/4 inch rods of spectrographic grade material (total impurities <6 ppm). The components were trimmed and weighed to produce final batch sizes between 50g and 60g. Charges generally consisted of single pieces of uranium and plutonium together with three or four pieces of carbon. The arc-melter was assembled, checked for leaks, and evacuated and back-filled three times with a mixture of helium and argon. The uranium and plutonium metals were melted together, and the carbon was allowed to dissolve in the molten alloy. After melting for one to three minutes, the button was allowed to cool and was then turned over and remelted. This operation was repeated six to ten times to ensure homogenization of the button. After the final cooling, the equipment was disassembled and the appearance of the button was noted and recorded prior to weighing. In many instances, buttons broke into two or more pieces and varied in color from purple to grey. Variations in texture were also observed but no correlation has been possible between the observations and other properties.

(a) P. J. Levine - Thermal Flux Irradiation Studies of (U,Pu)C Fuel
WARD-3791-16, April 1969.

Figure 5 Material Flow Diagram for Fabrication of (U,Pu)C Pellets

3471-12

Table 5. Enriched Uranium and Plutonium Metal Impurity Contents

Element	Analysis, ppm	
	Uranium Metal	Plutonium Metal
Ag	< 0.10	< 1
Al	25	< 2
Au	< 1.0	
B	< 0.20	< 1
Be	< 0.1	< 1
Bi	< 1	< 1
Ca	< 20	< 2
Cd	< 0.10	< 10
Co	< 5	
Cr	8	20
Cu	65	< 1
Fe	45	480
Mg	12	50
Mn	8	5
Mo	< 1	< 10
Na	< 40	< 1
Ni	300	20
Pb	< 1	< 1
Si	35	24
Sn	1	5
Th	< 100	
Ti	1.0	< 5
V	< 2.5	< 100
Zn	< 25	< 5
Zr	3.0	
C	33	95
N	10	
O	190	
	U-235 93.135%	Pu-238 .025%
	U-238 6.865%	Pu-239 92.574%
		Pu-240 6.464
		Pu-241 0.886
		Pu-242 0.051

Continued experience with the above operation enabled buttons to be produced with carbon contents essentially the same as the weighed-in amounts.

Three buttons were generally produced to provide sufficient material, ~180g, for fabrication of a complete pin of fuel and all characterization and archive samples.

5.2.2 Powder Preparation

The preparation of powder from buttons was performed in two stages:

1. Initial crushing was accomplished in a hand percussion mortar. The button was pulverized so that all particles would pass a 20# screen (< 841 μ). Samples taken at this stage were believed to be representative of the button composition.
2. Fine milling was performed on a Pica blender mill. The 180_g charge of -20# material was loaded into three tungsten carbide vials and two tungsten carbide balls were added to each vial. The closed vials were positioned in the mill and argon, cooled by passage through a liquid N₂ loop, and purged through the equipment at 16 cfh during milling and cooling. Milling times of 2-1/2 hours for the hypostoichiometric Cr₂₃C₆-modified material and 6 hours for the stoichiometric and hyperstoichiometric materials were used. Table 6 details all processing parameters used for each batch. Following milling, the equipment was cooled to room temperature prior to dismantling and sampling.

Continued improvements in the box atmospheres resulted in impurity levels typically 80-100 ppm oxygen and less than 1 ppm moisture, but even at these levels care was essential to prevent excessive reaction with the atmosphere.

In early work the -200# or -325# fraction of powder had been used for pellet fabrication, involving lengthy powder exposure to the box atmosphere. With later batches it was determined that the longer milling times produced more uniform powder and a coarse screen was sufficient to remove any large particles. Only the modified batches were prepared from the -200# fraction while stoichiometric and hyperstoichiometric batches were screened through the coarse 35# screen.

A number of binder materials were investigated. Initial investigations indicated that Carbowax 4000 dissolved in 200-proof ethanol and added at the rate of 0.5 w/o of the powder weight was suitable as a binder. High oxygen contents, in excess of 0.3 w/o, had been noted in pellets prepared from such powder, but this had been attributed to the relatively impure argon atmosphere. Atmosphere improvements indicated that approximately half of this contamination was derived from the binder. This conclusion was subsequently verified through a T.G.A. investigation using Carbowax-bonded UC pellets.

To decrease the pellet oxygen content a number of alternate binders were examined. A 0.75 w/o addition of a mixture of 2 parts acryloid: 1 part stearic acid was finally selected. Two hundred proof ethanol was used as

Table 6. Fuel Processing Details

Material Type	Capsule/ Batch No.	No. of Pellets	Additions (%)	Mill Time (Hrs)	Sieve Size (Mesh)	Pressure (tsi)	Fire Temp. (°C)	Fire Time (Hrs)	% TD (Green)	% TD (Fired)	Wt. Loss (%)	Length Shrinkage (%)	Diameter Shrinkage (%)
Hypostoichiometric (U,Pu)C	W8X/502	38	2.00 Cr ₂₃ C ₆	2.5	200	20	1550	2	72.0	88.0	0.52	5.78	7.27
	W2X/503	38	2.50 Cr ₂₃ C ₆	2.5	200	20	1550	2	71.0	88.0	0.47	5.56	7.20
Hyperstoichiometric (U,Pu)C	W5F/532	38		6	35	20*	1550	2	75.2	88.9	0.78	5.13	5.94
	W6F/533	31		6	35	20	1550	2	75.3	87.8	0.77	4.18	5.64
	+ 532	7		6	35	20	1550	2	75.2	88.9	0.78	5.13	5.94
Stoichiometric (U,Pu)C	W10F/534	38		6	35	20	1475	0	73.9	87.0	0.90	4.69	6.02
Hyperstoichiometric (U,Pu)C	W4F/535	28		6	35	20	1550	2	73.0	92.5	0.93	6.95	8.06
	+ 536	10		6	35	20	1550	2+2	72.0	92.5	0.86	7.18	8.16
"	W7F/538	38		6	35	20	1550	2	75.5	88.5	0.76	4.38	5.73
"	W3F/539	38		6	35	20	1550	2	72.7	90.2	0.72	5.88	7.11
"	-/548	38		6	35	20	1550	1	73.8	94.5	1.15	8.53	7.52
"	W8F/549	38		6	35	20	1550	2	73.8	91.5	0.91	7.72	6.04
Hypostoichiometric	W8X/175	2	4.64 Cr ₂₃ C ₆	2.5	200	20	1550	2	-	88.5	-	-	-
UC Insulators	W2X/175	2	4.64 Cr ₂₃ C ₆	2.5	200	20	1550	2	-	88.5	-	-	-
Stoichiometric UC		2/Pin			325	20	1550	8	-	88.0	-	-	-

Note: Batches 502 and 503 were prepared using 0.5 w/o Carbowax 4000; all other batches were prepared using 0.75 w/o binder, 2 parts Acryloid B-67 to 1 part stearic acid.

* Preslug at 55 tsi used prior to pressing

solvent and the carbide powder was screened directly into the solution to minimize exposure to the atmosphere.

It was noted that powder feed containing 60% of +140# agglomerates pressed easily to give defect-free pellets. Such material was produced by partially drying the mixture of carbide powder, binder, and solvent; and then, while still moist, passing the mixture through a 20# screen. Further drying produced granules that were finally passed through a 35# screen before checking size distribution prior to pressing.

This technique produced material with bulk and tap densities greater than 3.6g/cc and 6.8g/cc respectively and that pressed into sound pellets. Material having less coarse material and lower bulk and tap densities (Figure 6) indicated that pellet defects were often obtained, and such powder was not generally used for fuel pellet production.

5.3 PELLET MANUFACTURE

5.3.1 Pelletizing Procedures

Initial experience with the pressing of (U,Pu)C indicated that pressures of 30 tsi and above were required to produce sintered pellets of acceptable density. It was later determined that lower pressures could be employed without loss of density provided the initial powder was sufficiently fine. Figure 7 shows the effect of pressing load on density taken from some recent pressing studies using fine, 6-hour milled powder. There is very little fired density increase at pressures above 20 tsi, and minimizing the pressure reduces the possibility of pressure lamination defects in the final pellet. This pressure was used throughout as the final pelletizing pressure, although some work was undertaken using a preslugging and crushing operation prior to final pressing (see Table 6).

Four test pellets were pressed from each batch of powder prior to pressing the main batch of material; two were prepared from preslugged material, the other two from unslugged carbide. These pellets were fired at 1550°C for two hours. The processing conditions for the main batch were then established. The final processing conditions are detailed in Table 6.

Pellets were dry-pressed on a Carver hand-press using tungsten carbide lined dies and hardened steel punches lubricated with stearic acid. The diameters of the dies were calculated from firing shrinkage data so that minimal diametral grinding would be required. Green densities ranged from 71 to 75% T.D. with a range within any one batch not exceeding 1% T.D. Although manual pressing was used, every effort was made to approximate to double end pressing using the floating die principle. Also, care was taken to release entrapped gas by pressing slowly to approximately 10 tsi, holding at this pressure for a few seconds and then continuing to maximum pressure. Using these procedures, defect-free pellets with L/D ratios > 1 could be routinely produced.

5.3.2 Pellet Sintering

Pressed pellets were debonded and sintered in a graphite-tube resistance

Figure 6 Plot of Coarse vs Medium vs Fine Material for Various (U,Pu)C Batches

Figure 7 Density vs Pressing Pressure (Original Powder Milled for 6 Hrs)

furnace. For the Cr_{23}C_6 -modified material, an argon purge rate of 2 cfh was used. In stoichiometric and hyperstoichiometric materials, however, a layer of dicarbide needles was noted on the surface of many pellets when fired at these low flow rates, and the rate was increased to 10 cfh. It was assumed that the low flow rate was not capable of removing all binder decomposition products, and the firing cycle was increased to permit a longer burnout period. The two basic cycles used during the work were:

1. Cr_{23}C_6 -modified material--3 hours to 700°C, 1-1/3 hours to 1550°C, 2 hours at 1550°C, 1-1/2 hours to room temperature
2. Stoichiometric and hyperstoichiometric material--7 hours to 500°C, 1-1/3 hours to 1550°C, 2 hours at 1550°C, 2 hours to room temperature.

A maximum sintering temperature of 1550°C was found to be adequate for all batches processed. In certain instances, increases in fired density were obtained by refiring pellets on a standard firing cycle (batch 536) and in other cases where it was necessary to lower the final density, the firing time and/or temperature were decreased (batches 534, 548 and 549).

All sintered pellets were visually checked for obvious defects prior to complete mensuration and density calculations. Suitable batches of material were stored pending the initial characterization results (carbon, oxygen, nitrogen, and metallography), while oversize pellets were dry ground on a centerless grinder. The edges of all pellets were then radiused to specification using dry emery cloth.

5.3.3 UC Insulator Pellet Fabrication

Although carbide fuel pellets were synthesized from arc-melted material, certain of the insulator pellets were fabricated from carbothermically reduced oxide. Depleted UO_2 and graphite powder were wet blended in an oxylene medium and were then dried and pressed into 1 inch diameter by 1 inch long pellets. These pellets were reacted under vacuum for periods in excess of seven hours at 1550°C, and after cooling, the porous cake was comminuted with a pestle and mortar. Fine grinding of the material was accomplished in a tungsten carbide lined mill, and a Carbowax 6000 binder was added in carbon tetrachloride solution. After drying, press charges were weighed out and the material was cold pressed and sintered. In order to attain density, two pressures were used with each batch, 20 tsi and 30 tsi. Sintering was undertaken at 1550°C for eight hours.

SECTION 6

FUEL CHARACTERIZATION

6.1 CHARACTERIZATION TECHNIQUES

6.1.1 Powder Particle Size and Shape

Two methods of sizing were employed utilizing either a Fisher sub-sieve sizer or micro-mesh sieves, while particle shape was checked microscopically. The majority of particle size determinations were made using the Fisher, to which two modifications were made for glove box operation. First, the water in the pressure control tube and manometer was replaced with a silicone oil having a density and viscosity close to that of water. The particle size of ZrO_2 was then checked using the Fisher in a high-purity argon atmosphere and no difference was observed from similar tests performed in the open laboratory.

Second, the performance of the instrument with small samples was determined. With mixed (U,Pu)C powder samples, the standard sample size is 13.6 g. Because of the risk of oxidation, samples may not be returned to the main batch.

The instrument was recalibrated for half and quarter size samples. Curves relating compact porosity, manometer height, and mean particle size were constructed for the new sample sizes. The standard Fisher chart was replaced with a depth gage yielding greater accuracy in the measurement of both porosity and manometer fluid level. The use of the depth gage was essential to yield accurate results, as many powder batches gave compacted porosities < 40% (the lower limit of the standard Fisher chart).

Preliminary results indicated that half-sized samples gave consistent results, but the quarter-sized samples gave results that were sensitive to the sampling technique. Indicated particle size increased with decreasing sample size, thus requiring a calibration curve.

A limited amount of micro-mesh sieving was performed on the milled powder. Micro-mesh sieve sizes of 30, 20, 10, and 5 μ were used with 200-proof absolute alcohol as the carrier. 53 μ and 44 μ wire mesh sieves were added to the stack of micro-mesh sieves to extend the range surveyed. With a 5-gram sample of powder tested, 98% recovery was required for a particular test to be accepted. Dry sieving gave only a 70% recovery.

To check particle shape, a small quantity of powder (~0.2 gram) from one of the sub-micron sieves was placed on a thin glass slide with a stainless spatula and mixed with epoxy resin. The powder and epoxy were spread evenly over the slide to form as thin a layer as possible. The sample was examined on the microscope using transmitted light.

6.1.2 Chemical Analyses

6.1.2.1 Carbon - Carbon analysis was made using a combustion-gravimetric technique where the sample is ignited in a stream of oxygen at 900-950°C.

CO₂ is absorbed in "Ascarite" (sodium hydroxide-impregnated asbestos), which is weighed to determine carbon content from the weight gain. Micro-weighed samples of high-purity graphite are used as recovery standards, for which the coefficient of variation at the 95% (2 σ) confidence limit is $\pm 2.4\%$. The corresponding coefficient for duplicate sample analyses is similar. With an average carbon content of 4.65% w/o in the duplicates, the total error (analytical plus sampling) for carbon analysis is ± 0.11 w/o.

6.1.2.2 Oxygen and Nitrogen - The oxygen and nitrogen analyses are made on a single sample using inert gas fusion and gas chromatography. A commercially available unit (Nitrox-6, Leco) was modified for glove box operation. The sample is introduced to the induction furnace, which contains a Pb-10% Sn bath in a graphite crucible. The oxygen, as CO, and elemental nitrogen are liberated at 2000-2100°C. The O₂-N₂ concentrations are calculated by comparison of the liberated gases with standards (potassium acid phthalate for oxygen and potassium thiocyanate for nitrogen). Using NBS recovery standards of U₃O₈ and UN_x, the respective coefficients of variation, at 2 σ for the oxygen and nitrogen analyses are $\pm 10\%$ and $\pm 22\%$, respectively.

6.1.2.3 Pu/U Ratio - The Pu/U ratio was determined by X-ray emission spectrometry. The (U,Pu)C sample is oxidized and fused with 95% potassium pyrosulfate-5% borax at 750-850°C. A ratio of the L α peaks of U and Pu is compared to the corresponding ratio from a standard (PuO₂ and U₃O₈) analyzed concurrently. The relative variation for the Pu/U ratio at the 95% confidence limit is $\pm 1.0\%$.

6.1.2.4 Plutonium - The plutonium analysis is performed on the same sample used for the Pu/U ratio. Yttrium is added as an internal standard, resulting in a Pu/Y ratio from which Pu concentration is determined. The relative variation at 2 σ for the Pu analysis is $\pm 2.0\%$.

6.1.2.5 Chromium and Tungsten - The tungsten and chromium analyses are determined by X-ray emission spectrometry on the Pu/U ratio samples. The concentrations result from direct counting of the W and Cr lines and subsequent comparisons to similarly-prepared standards.

6.1.2.6 Total Metallic Impurities - The total of metallic impurities is determined by emission spectroscopy using the carrier-distillation technique. A silver chloride-barium fluoride carrier was used.

6.1.3 Metallographic Evaluation

Routine metallographic procedures and standard equipment were used in the box line with no major problems encountered. Samples were evaluated microscopically for capping, laminations, and other pressing defects. Microscopic evaluation included determination of the nature, size, and distribution of porosity; grain size, and the nature, volume, and location of secondary phases.

Samples were singly hot mounted. All water was removed from the various liquids used in the grinding and polishing operations to prevent sample oxidation. The water and oxygen contents in the argon circulating through the box line were maintained at less than 1 ppm and 100 ppm, respectively.

Three types of etching were employed--aqueous acidic, electrolytic, and vacuum cathodic, to provide delineation of grain boundaries, secondary phases, and inclusions. A linear intercept method was used to determine the volume of secondary phases as well as pore and grain size. Specific etching techniques were developed for each type of fuel.

6.1.3.1 Cr₂₃C₆-Modified (U,Pu)C Fuel - A 2:1 acetic:nitric acid electrolytic etch gave the most consistent results, producing good grain boundary delineation and phase contrast. The various chromium carbide phases exhibited a lighter etched color than the matrix. Other etches used include 10% oxalic acid in methanol, and 5% nitric acid in methanol (nital), both electrolytic. Five v.o.c. was used for durations between 5-30 seconds for electrolytic etching.

6.1.3.2 Stoichiometric (U,Pu)C - Water-containing swab etches produced the most consistent results for the unstabilized fuels, 1:1:1 nitric:acetic:water being the principal etch employed for stoichiometric (U,Pu)C. Typical results showed a grey etched matrix with (U,Pu)₂C₃ or free metal as the lighter etched phase. Five percent bromine in methanol as a swab etch was used to identify free metal, which is preferentially attacked. The deleterious effect of bromine vapor on the argon purification system was a major disadvantage of this technique.

A 1:39:60 hydrofluoric:nitric:water swab etch proved to be superior to the bromine-methanol etch in differentiating between hypo- and hyperstoichiometric carbides. This etch only attacks metal-rich areas, turning them black (Figures 8 and 9), but does not attack (U,Pu)₂C₃. This etch was compatible with good atmosphere control and produced no significant damage to microscope objectives.

6.1.3.3 Hyperstoichiometric (U,Pu)C Fuel - The two basic etches for stoichiometric fuel (i.e. 1:1:1 nitric:acetic:water and 1:39:60 hydrofluoric:nitric:water) were also found to give the most satisfactory results for the hyperstoichiometric fuel. (U,Pu)₂C₃ etched to a light color with both etches and acicular dicarbide-type phases a dark color.

The 1:39:60 hydrofluoric:nitric:water swab etch was found to be more reliable in acicular, or needle-type, phase delineation, although the nitric:acetic:water etch generally produced a more balanced grain boundary delineation with secondary phase contrast, when presence of needle-type phase was not suspected.

6.1.4 Density Determinations

Two types of density determinations were made on the pellets. Geometric densities were determined on all green and fired pellets while duplicate immersion-type determinations were undertaken on 10% of the fired pellets. Dried xylene was the immersant used for all immersion-type determinations and was calibrated with stainless steel standards. The maximum deviation between duplicates on a particular pellet was $\pm 0.3\%$ for theoretical density and $\pm 0.1\%$ for open and closed porosity when the theoretical density was greater than 88%. Greater deviations were observed in the porosity values at lower densities, but in every case these were $< 1\%$.

Figure 8 Section of Hypostoichiometric Pellet Showing White Metal-Rich Areas and Rounded (U,Pu)C Matrix Grains. Swab Etched in 1:1:1 Nitric Acid:Acetic Acid:Water. (Mag. X740)

Figure 9 Same Section as Shown in Figure VII-6-2 after 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. Etch. Note Darkened Metal-Rich Areas. (Mag. X740)

6.1.5 LASL Pellet Characterization

Representative pellets were sent to LASL for study by metallography, X-ray diffractometry, electron-probe micro-analysis, and differential thermal analysis. A total of 39 pellets (~ 4% of the total made) were examined.

6.2 FUEL CHARACTERIZATION RESULTS

6.2.1 Powder Particle Size and Shape

A typical particle size distribution curve is shown in Figure 10. The curve represents a powder consisting of more than 50 w/o less than 10μ in size. Of this, 18.5 w/o was retained on the 5μ sieve (i.e., the particle size was between 5 and 10μ), and 45 w/o had a particle size less than 5μ .

The various fractions were checked optically, and little subsize retention was observed down to the 10μ sieve. Figure 11 shows particles from the 20 to 30μ fraction. These have the rounded angular configuration expected of a milled product. However, agglomeration of the sub- 10μ particles complicated the evaluation. Furthermore, with some powders, blockage of the 10 and 5μ sieves occurred necessitating prolonged ultrasonic cleaning to clear these. Nevertheless, overall results imply approximate agreement with the Fisher equivalent spherical diameter findings of 2- 3μ .

6.2.2 Chemical Analysis

Results of chemical analyses are presented in Table 7. There are fluctuations in oxygen and nitrogen contents from batch to batch which cannot be correlated with fabrication variables. It is thought that the relatively low oxygen content of the stoichiometric and hyperstoichiometric batches was caused by the change from Carbowax, which was the binder used in the fabrication of the two Cr_{23}C_6 -modified batches (502 and 503), to an Acryloid/stearic acid binder system.

The tungsten content of most batches is considerably higher than expected, but is not expected to have deleterious effects during testing. Tungsten contamination can occur during arc melting (a W electrode is used) and milling (a WC liner and balls are employed).

Figure 12 shows a portion of the (U,Pu)-Cr-C ternary system, typical of temperatures below 1300°C , on which has been located the analytical compositions of the two Cr_{23}C_6 -modified batches 502 and 503. The diagram is based on the U-Cr-C diagram, proposed by Briggs, et al., [12] while taking into account the work of Anselin, et al., [13] concerning the homogeneity ranges of UC and PuC. Tungsten has been assumed to function essentially as chromium and thus contribute to a Cr equivalent (Cr_E). The chemical analysis suggests that no free U, Pu, or Cr metal should be present in the material. The intended composition of such fuel is $(\text{U,Pu})\text{C} + \text{Cr}_{23}\text{C}_6 + \text{Cr}$.

6.2.3 Metallographic Observations

Metallographic observations on all fuel types are summarized in Table 8.

Figure 10 Particle Size Distribution of a Milled (U,Pu)C Powder

Figure II Typical (U,Pu)C Particles From a 20 to 30 μ Fraction, i.e., Those Retained on a 20 μ Sieve. Particle Shape Typical of a Milled Product. (Mag. X90)

Table 7. Fuel and Insulator Pellet Chemical Analyses

Pin # Batch #	Pu/U Ratio	Pu (%)	U (%)	Cr (%)	Total U + Pu (%)	C (w/o)	O (ppm)	N (ppm)	Total Metallic Impurities (ppm)	Tungsten (ppm)	Carbon Equivalent
W8X, 502	0.2372	17.61	75.0	2.71	92.61	4.72	3017	281	3698	2900	4.97
W2X, 503	0.2494	18.18	74.24	3.30	92.42	4.59	4662	969	4571	3700	5.02
W5F, 532	0.2347	18.00	76.69	--	94.69	4.64	1470	1600	3450	1780	4.89
W6F, 533 +532	0.2359	18.06	76.54	--	94.60	4.72	1320	1700	3410	2220	4.96
W10F, 534	0.2547	18.36	76.57	--	94.93	4.47	2020	2100	1930	1110	4.80
W4F, 535 +536	0.2483 0.2441	18.87 18.57	76.00 76.06	-- --	94.87 94.63	4.68 4.86	1620 2370	1400 800	1530 1840	910 960	4.92 5.11
W7F, 538	0.2402	18.36	76.46	--	94.82	4.71	1380	500	2820	1920	4.86
W3F, 539	0.2494	18.94	75.94	--	94.88	4.71	1470	600	1930	1170	4.87
W12F, 548	0.2407	18.32	76.09	--	94.41	4.86	1501	2186	3580	2690	5.16
W8F, 549	0.2400	18.38	76.56	--	94.94	4.63	1172	1906	1230	800	4.88
W8X, W2X Insulators	-- --	-- --	88.92 91.45	5.89 3.20	-- --	4.56 4.69	2837 3548	1481 1012	-- --	2000 2100	4.90 5.05
Unmodified Insulators	--	--	95.00	--	--	4.38	5179	103	--	--	4.78

Figure 12 Tentative Ternary Phase Diagram of $(U_{0.8}Pu_{0.2})$ -Cr-C Showing Tie Lines for the Solid State. Batch Compositions Determined from Analytical Chemistry are Located on the Diagram.

Table 8. Metallographic Observations on Fuel Pellets

Pin #, Batch #	Avg. Grain Size (Metallography) (μ)	Avg. Pore Size (μ)	Porosity Type(5)	Imm. Density (% of Theoretical)	Open Porosity (%)	Closed Porosity (%)	Secondary Phases (v/o)	(U,Pu) ₂ C ₃ (v/o) (1)	(U,Pu) ₂ C ₃ (v/o) (2)	(U,Pu) ₂ C ₃ (v/o) (3)
W8X,502	28	20	R,A	88.9	1.8	9.3	7.0	--	--	--
W2X,503	25	20	R,A	90.1	0.6	9.3	5.6	--	--	--
W5F,532	5	4	R,S	89.3	5.2	5.5	--	9.6	3.0	7.0
W6F,533	5	8	A,S,I	88.8	7.6	3.6	--	9.0	5.8	9.8
W10F,534	5	10	A,S,I	88.1	8.3	3.6	--	0.0	0.0	3.4
W4F,535	7	15	A,S	92.9	2.4	4.7	--	$\sim 7.0^{(4)}$	4.0	8.0
+536	10	5	A,S,I	93.7	3.3	3.0	--	$\sim 10.0^{(4)}$	13.0	17.0
W7F,538	10	10	R,A,S	89.3	6.4	4.3	--	$\sim 5.0^{(4)}$	1.0	5.0
W3F,539	10	5	A,S	91.0	4.3	4.7	--	$\sim 15.0^{(4)}$	1.5	5.5
W12F,548	5	5	R,S	95.3	0.4	4.3	--	$\sim 24.0^{(4)}$	15.25	19.25
W8F,549	5	10	A,S,I	92.7	1.5	5.8	--	$\sim 3.0^{(4)}$	2.0	6.0

(1) Based on metallographic observations

(2) Based on analytical chemistry data using a theoretical C_E value of 4.835 w/o

(3) Based on a practical stoichiometric C_E value of 4.75 w/o from data published by LASL (LASL Quarterly Report for the Period Ending December 31, 1968, LA 4114-MS)

(4) Impossible to obtain an accurate value by point counting because of the structure type.

(5) R - rounded
A - angular
S - stringered
I - irregular

6.2.3.1 Cr₂₃C₆-Modified Fuel - Typical porosity distributions in Cr₂₃C₆-modified and unmodified fuel pellets are shown in Figures 13 and 14. When the Carbowax binder was used in the Cr₂₃C₆-modified material, the pores were invariably large and round. An Acryloid/stearic acid binder used in the fabrication of the unmodified fuel batches gave a much finer porosity, which occurred in regular networks and often appeared interconnected. The Cr₂₃C₆-modified pellets were sintered in the presence of a liquid phase and this probably affected the porosity structure and caused the average grain size of the Cr₂₃C₆-modified (U,Pu)C to be significantly greater than that for the unmodified fuel. Typical figures for the modified and unmodified are 25-30 μ and 5-10 μ , respectively.

The microstructures of batches 502 and 503 (Figures 15 and 16) have a very similar appearance. This observation is consistent with the point count analyses which indicate the compositions of both lie in the M-Cr₃C₂-Cr₇C₃ phase triangle. Both exhibit a uniform grain size and porosity distribution, with two minor phases surrounding the (U,Pu)C matrix grains.

6.2.3.2 Hyperstoichiometric Fuels - The porosity and phase distributions observed in the hyperstoichiometric samples were generally similar. Examination of the as-polished surfaces indicated an open type of porosity with some interconnection that is consistent with the immersion density data. Figure 14 illustrates this point. The structures consisted of angular (U,Pu)C grains with varying amounts of (U,Pu)₂C₃, which occurred as islands. This is illustrated in Figures 17 and 18, which also show the effect of the different etches used. In the case of the one stoichiometric batch (534), the matrix grains were also angular, (Figure 19).

A needle-like type phase was found to a limited extent within the matrix grains of all samples. The great majority of needles were fine and responded to polarized light except in the case of batch 549. In all cases the amount of this phase was less than 1 v/o and for batches 535 and 539 was so small that it was impossible to point count. A similar needle phase has been observed in button material (Figure 20), and the sintering temperature is probably too low to remove it from the structure. These small amounts of the needle-phase were considered acceptable in the material to be used in irradiation studies.

The structure observed in batch 548 was very homogeneous, consisting of (U,Pu)C matrix grains interspersed with islands of (U,Pu)₂C₃ (Figure 21), but no needle-type phase was observed. Point counting indicated that there was 24 v/o (U,Pu)₂C₃, equivalent to C_E of 5.34 w/o, assuming stoichiometric C_E in (U,Pu)C is 4.835 w/o.

The initial examination of batch 549 indicated that it contained approximately 3 v/o (U,Pu)₂C₃ and 3 v/o of a needle-type phase. The pellets of this batch were refired at 1500°C for two hours in an attempt to reduce the quantity of the needle-type phase. A skin, 0.006 to 0.008-inch deep, was observed in this refired material; and this contained 3-5 v/o of the needle-type phase, which did not respond to polarized light (Figure 22). However, the needle-type phase had been completely removed from the interior regions of the pellets.

Figure 13 Representative Porosity Distribution in Typical Sintered Cr_{23}C_6 -Modified Fuel Pellet. (Mag. X75)

Figure 14 Representative Porosity Distribution in Typical Sintered Hyperstoichiometric Fuel Pellet. (Mag. X90)

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Figure 15 Batch 502. Cr_{23}C_6 -Modified (U,Pu)C. Sintered for Two Hours at 1550°C . Uniform Distribution of Second Phase. Swab Etched 85% Orthophosphoric Acid. (Mag. X185)

Figure 16 Batch 503. Cr_{23}C_6 -Modified (U,Pu)C Sintered for Two Hours at 1550°C . Similar Structure to Batch 502, but With Less Second Phase Material. Electrolytic Oxalic Acid Etch. (Mag. X185)

Figure 17 As-Sintered Pellet from Batch 538 Showing $(U,Pu)_2C_3$ Distributed Throughout the Matrix. Occasional Fine Needles can Also be Observed. Swab Etched in 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. (Mag. X740)

Figure 18 Central Area of an As-Sintered Pellet from Batch 536 Showing Distribution of $(U,Pu)_2C_3$ in the Matrix. Note That There is Very Little Grain Boundary Resolution. Swab Etched in 1:1:1 Nitric Acid:Acetic Acid:Water. (Mag. X740)

Figure 19 As-Sintered Pellet From Batch 534 Showing Angular Matrix Grains. No Secondary Phases are Present. Swab Etched in 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. (Mag. X740)

Figure 20 Segment of an Arc-Melted Button Showing an Area Containing a Concentration of the Needle-Type Phase in Large Matrix Grains. Swab Etched in 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. (Mag. X370)

3471-20.

Figure 21 As Sintered Pellet from Batch 548 Showing $(U,Pu)_2C_3$ Islands Dispersed in $(U,Pu)C$ Matrix. Swab Etched in 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. (Mag. X740)

Figure 22 Pellet from Batch 549, Refired for Two Hours at $1500^{\circ}C$. Peripheral Area Exhibiting Needle-Type Phase Which was Unresponsive to Polarized Light. Swab Etched in 1:39:60 Hydrofluoric Acid:Nitric Acid:Water. (X740)

3471-21

It has proved difficult to obtain accurate figures for the volume fraction of $(U,Pu)_2C_3$ by point counting the samples from batches 535 to 548 because of the texture and phase distribution. However, the approximate figures that were obtained for $(U,Pu)_2C_3$ content in these batches were reasonably consistent with those anticipated from analytical chemistry.

In the majority of cases, the v/o $(U,Pu)_2C_3$ obtained by point counting is much closer to that calculated using the value for C_E in the $(U,Pu)C$ phase obtained from analytical chemistry data published by LASL[14] than that calculated assuming a theoretical 1:1 stoichiometry (see Table 8).

The needle-type phase referred to above always occurred within $(U,Pu)C$ matrix grains and consisted of two types - fine and coarse. The black, fine needles, which respond to polarized light, are considered to be $(U,Pu)C_2$. At present, it is not possible to assign a definite composition to the coarser structure, which gives a grey color on etching and shows little response to polarized light. However, reassessment of all available data indicates that an oxygen alteration product of $(U,Pu)C_2$ results from incursion of oxygen and nitrogen gaseous impurities during the sintering stage of the fabrication process.

Two reasons for this conclusion are:

1. The observed depth of rod formation into the fuel, under different firing conditions, is in reasonable agreement with the calculated penetration by diffusion of the two gaseous impurities.
2. Henney[15] observed that UC_2 has an affinity for oxygen, which gives rise to a UC_xO_y solution. The stability of this phase is greater than that of sesquicarbide, especially if the monocarbide matrix is even slightly substituted with nitrogen. When nitrogen is the predominant impurity, the equilibrium phases are a mixture of UC_2 and a precipitate of a cubic $UC_{(1-x)}N_x$ solid solution. Furthermore, sesquicarbide has a very rigid lattice, which does not allow oxygen or nitrogen substitution.

The following observations are also significant:

- The coarse needles give only a very slight response to the conventional nitric:acetic:water etch. A 1:39:60 $HF:HNO_3:H_2O$ swab etch has proved much more effective in revealing these structures.
- In sintered fuel pellets where there is less than 1 v/o needle phase, the needles only occur in the larger grains. This result is similar to that described in Harrison's work,[16] where local CO pressure within the larger grains during sintering has been observed to promote growth and retention of dicarbide material.
- If the coarse needles were $(U,Pu)O_2$, it is felt that their lower reflectivity would render them visible in the as-polished sample, and this is not the case. On etching, however, the coarse needles have the characteristic grey coloration of oxides.

6.2.4 Density Determinations

Representative geometric and immersion density values of the various batches are listed in Table 6. The geometric density was always lower than that obtained by the immersion technique. The open porosity values of unmodified fuel batches are generally higher than those observed in samples of Cr_{23}C_6 -modified fuel batches having comparable immersion densities. This is consistent with the observed pore structure, which also indicates a higher volume percent of open porosity with a degree of interconnection (see metallography section). In turn, this can be related to a change in binder type during fabrication from Carbowax to a 2:1 Acryloid:stearic acid mixture and/or sintering in the presence of a liquid metal phase in Cr_{23}C_6 -modified (U,Pu)C. With reference to the metallographic evaluation, a minimum of two pellets from each batch was examined, immersion densities having previously been determined on the same pellets.

6.2.5 LASL Characterization Results

The volume fraction of secondary phases in Cr_{23}C_6 -modified (U,Pu)C pellets was too small to permit identification by X-ray diffractometry. However, deduction of the nature of secondary phases present was possible from the results of the electron microprobe examination. Details of these phases are given in Table 9. Phase A, which is found in both batches, is presumably (U,Pu) CrC_2 so that if the (U,Pu)-Cr-C diagram (Figure 12) is correct, the other secondary phase should be Cr_3C_2 . However, assessment of the accumulated microprobe and microhardness data suggests that it is Cr_7C_3 rather than Cr_3C_2 . The choice of Cr_7C_3 over Cr_3C_2 is also in better agreement with the location of the analytical compositions in Figure 12. Thus, there are strong indications that the (U,Pu)- Cr_3C_2 tie line does not exist in the (U,Pu)-Cr-C system. Details of the secondary phase volumes obtained by point counting and from analytical chemistry data for these two batches are given in Table 10. The Cr-based phases [Cr_7C_3 , Cr_{23}C_6 , (U,Pu) CrC_2] all appeared white as polished or etched. Microhardness values were 980-1170 kg/mm^2 for Cr_7C_3 and 1380 kg/mm^2 for Cr_{23}C_6 .

Small amounts of tungsten and chromium (<0.5 w/o) were found in the (U,Pu)C matrix of these fuels. The matrices were homogeneous in that relative standard deviations in U and Pu microprobe X-ray intensities were only ± 1 -2%.

Although the composition of fuel in batches 502 and 503 was not in the "preferred" (U,Pu)C- Cr_{23}C_6 -Cr phase triangle, indications are that this composition is more refractory than those containing the "preferred" phases. DTA tests made on batch 511, which contains the same phases as batch 502 and 503, showed a thermal arrest on heating and cooling at 1385°C. This apparent solidus temperature, even though somewhat lowered by W impurity, is significantly higher than the solidus temperature of approximately 1280°C estimated for the (U,Pu)C- Cr_{23}C_6 -Cr phase area.

The presence of Cr_7C_3 or Cr_3C_2 may affect the compatibility of the fuel with stainless steel in a sodium environment. Mol^[17] reports that with Cr_{23}C_6 -modified UC in a stainless container with a sodium environment,

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

(a) Referred to intensity from (U,Pu)C

(b) Referred to intensity from Cr metal

Table 10. Details of Secondary Phases Occurring in the Cr_{23}C_6 -Modified Batches 502 and 503

Batch No.	Point Counted Secondary Phase	Total Calculated From Analytical Chemistry Data - Secondary Phase	Major Secondary Phase	Minor Secondary Phase
502	7.3	6.0	Cr_7C_3	(U,Pu)CrC ₂
503	5.6	7.4	(U,Pu)CrC ₂	Cr_7C_3

unless the composition lies in the UC-Cr₂₃C₆-Cr phase field, gross carburization of the steel occurs. Tests were run at 800°C for 2000 hours. The fuel was fabricated by arc casting UC and chromium.

The work of Jacoby, et al, [18] at ANL discounts this. Compatibility tests were run at 800°C for 1000 hours of Cr₂₃C₆-modified (U,Pu)C against stainless steel in a sodium environment and no reaction was observed. Microprobe examination of this fuel revealed no free chromium metal or fissile secondary phase, indicating the composition lay in the large (U,Pu)C-Cr₃C₂-Cr₂₃C₆ phase field. The fuel for these experiments was prepared by the same route as the irradiation test samples.

6.3 PELLET QUALITY CONTROL

The dimensions of all pellets (Table 11) were checked two or more times. The initial measuring was performed immediately after the pellets were removed from the sintering furnace. Only pellets with acceptable density and dimensional values were reserved for loading. A second check was made on these pellets immediately prior to loading. Micrometer readings of the pellet diameters were taken at three places and a mean value was calculated. A single reading of the length was taken. Dimensions and weights were recorded to the fourth decimal place and the pellet densities were recalculated.

Each pellet was inspected at 20X magnification for signs of cracks, fissures or other surface irregularities and defects were compared to an agreed standard.

After the pellets had been individually measured, they were assembled into a stack and the overall length was compared to the summation of the individual lengths. Nominal 0.375 inch UC insulator pellets were added to each end of the stack and the total length rechecked.

Table 11. Fuel Pellet Dimensions

No.	Batch No.	Type *	Total Wt. (g)	Avg. Dia. (inch)	Max. Dia. (inch)	Min. Dia. (inch)	Avg. Height (inch)	Total Fuel Stack (inch)	Density (g/cc)
W8X	502	Cr	97.83	0.2608	0.2620	0.2600	0.2498	9.493	11.79
W2X	503	Cr	86.95	0.2451	0.2460	0.2450	0.2487	9.491	11.83
W3F	539	H	95.31	0.2511	0.2515	0.2510	0.2500	9.503	12.34
W4F	535/536	H	95.71	0.2493	0.2495	0.2490	0.2495	9.481	12.65
W5F	532	H	71.78	0.2200	0.2200	0.2200	0.2501	9.504	12.13
W6F	532/533	H	70.96	0.2195	0.2200	0.2195	0.2501	9.506	12.03
W7F	538	H	92.60	0.2493	0.2500	0.2475	0.2501	9.507	12.11
W8F	549	H	96.44	0.2508	0.2510	0.2500	0.2500	9.500	12.54
W10F	534	S	70.24	0.2194	0.2195	0.2190	0.2502	9.5085	11.93
W12F	548	H	75.42	0.2183	0.2190	0.2180	0.2502	9.509	12.92
W14F	537	H	91.49	0.2510	0.2510	0.2510	0.2493	9.477	12.24

*
 S - Stoichiometric
 H - Hyperstoichiometric
 Cr - Chromium modified

SECTION 7

FUEL PIN FABRICATION

7.1 INTRODUCTION

The sequence of activities used in the fabrication and assembly of fuel pins is summarized in Figure 23. The major operations in the assembly include: welding in the bottom end plug; loading sodium; loading pellets; initial sodium melting; loading the hold down components; welding in the top end plug; sodium bonding; and final certification and characterization. The two most critical development areas were the welding and sodium bonding procedures.

7.2 ASSEMBLY PROCEDURES

The bottom end plug was welded into the cladding tube and the weld inspected by helium leak testing, radiography, and dye penetrant testing. A charge of solid sodium was loaded into the tube and then melted to form a solid plug in the tube. Sodium loading was performed in an argon atmosphere in the sodium handling glove box.

The sodium-loaded tubes, capped with a short sealed section of plastic tubing, were transferred into the plutonium laboratory for pellet loading. The capped tube was inserted into the pellet loading glove box through an "udder bag." A guide tube was inserted inside the clad tube for loading the insulator and fuel pellets and the spacer rod. The guide tube minimized contamination of the open end. A resistance heating furnace was raised to surround the bottom portion of the fuel pin and the sodium melted allowing the pellets to sink to the bottom of the pin. The udder bag tubing was sealed and the fuel pin removed by cutting through the seal.

The pins were transferred to the decontamination box where the plastic tube was removed and the open end of the fuel pin tube was cleaned until the alpha count of a swab was less than 100 dpm. The cleaned end was sealed with a clean plastic cap and transferred into the welding glove box.

The welding box was evacuated to less than 0.02 micron Hg and back-filled with helium twice, before and after removing the plastic cap from the fuel pins. The helical spring and top end plug were inserted in the open tube and the final closure weld was made to complete the assembly of the fuel pin.

After removal of the pin from the weld box, the pins were heat treated at 1000°F for ~1 hour to ensure a void free sodium bond and complete wetting of the internal components. Details of the sodium bonding cycle are given in Section 7.5.

Figure 23 Flow Diagram for Fuel Pin Fabrication

7.3 FUEL PIN INSPECTION

7.3.1 Leak Testing

Two types of leak test were performed on the completed pins. The first was a bubble test in which the pins were externally pressurized with gas at 150 psi and then submerged in a transparent filled container. Small leaks could be detected by observing the source of streaming bubbles. If no leak was detected, the more sensitive standard helium leak test was performed. Although the specification acceptance limit was a maximum leak rate of 1×10^{-8} cc/sec., no leaks were detected with the equipment set at a sensitivity of 3×10^{-9} cc/sec.

7.3.2 Radiography

End plug welds were radiographed with correction blocks to attain a sensitivity of 2-2T. Acceptance was based on the absence of detectable (>1 mil) defects and a minimum penetration equivalent to 100% of the cladding wall thickness.

Uncorrected full length radiographs were made to check the location of the fuel and internal components and to ensure that no fuel damage had occurred during the bonding operation.

7.3.3 Eddy Current Testing

The continuity of the sodium bond was checked by eddy current testing. Heat transfer analyses established the following criteria.

- Maximum single defect frontal area must not exceed 0.004 in.^2 (equivalent to a diameter of 0.07 in.)
- Individual defects must be greater than 0.04 in. apart
- The sum of the frontal area of all defects must be less than 0.01 in.^2

Typical examples of results attainable by the eddy current test are shown for pin number W3F in Figure 24. Figure 24(a) is a trace of a pin in an unacceptable condition showing numerous defects in the sodium bond. Figure 24(b) shows a reduction in size and number of defects after centrifugal bonding. Figure 24(c) is an acceptable trace of the same pin after subsequent vibratory rebonding treatment eliminated the defects. The low amplitude, long pitch indications are caused by eccentric positioning of the fuel pellets within the pin.

Defects as small as 0.03 in. frontal diameter can be detected based on the experience of comparing eddy current traces with radiographs and destructive examinations of development pins.

7.3.4 Sodium Purity Analysis

Two simulated fuel pins containing 3g of reactor grade sodium and ten 1 inch long 316 stainless steel pellets were simultaneously processed with each group of (U,Pu)C bearing pins. The processing operations for the dummy sodium

Figure 24 Typical Eddy-Current Traces, Pin W3F

purity samples were identical to those experienced by fuel pins. After welding and bonding of the sodium purity control samples, oxygen-in-sodium analyses were performed on the representative control samples by the mercury amalgamation method. The results of these analyses are presented in Table 12.

These values are the summation of all of the oxygen contributing operations, namely, initial loading and melt down, fuel bonding, fuel contribution in sodium melt-down and decontamination, and fuel pin bonding.

7.3.5 Profilometer Measurements

Diameter measurements were performed on each fuel pin after sodium bonding and immediately prior to encapsulation. A LVDT mechanically-actuated transducer was used.

A slight reduction in tube diameter was apparent in all pins above the sodium level in the plenum region. Figure 25 is a typical representation of six traces of diameter measurements at 30° rotational increments for fuel pin W2X. Table 13 summarizes the average of the six measurements in the sodium bond region and the plenum region of eight fuel pins.

Fuel pin dimensional measurements are summarized in Table 14.

7.4 FIDUCIAL MARKING OF CLADDING

To obtain post-irradiation axial clad strain data, fiducial marks were applied to the outside diameters of all fuel pins except W2X and W8X. By making accurate linear measurements between adjacent impressions before and after irradiation exposure, direct measurements of local axial clad strain can be made.

A Tukon microhardness tester with a Knoop indenter was used to make controlled depth impressions on the final fabricated fuel pins. Room temperature burst test results on solution treated and cold worked tubing specimens containing impressions approximately .0005-inch deep x .0021-inch wide indicated that the impressions had no affect on failure initiation, crack propagation, or ultimate burst pressure. Final impressions on "as fabricated" fuel pins were controlled to .0014 - .002 inches wide resulting in impression depths of .0003 - .0005 inches.

Impressions were placed on the fuel pins along a line parallel to the pin axis beginning 1.100 in. from the bottom end plug and continuing in .500 in. increments for 10 in. (along the projected fuel stack region); the final three impressions were separated by 1.000 inch. Measurements between the Knoop impression diagonals were made using a bench microscope and verified with the hardness testing machine.

7.5 SODIUM BONDING

To obtain wetting between the sodium and contacting surfaces, the pins and capsules were subjected to an elevated temperature bonding treatment. Mechanical energy, imparted to the pin or capsule while at the bonding temperature, was also necessary to eliminate unwetted areas and gas bubbles.

Table 12. Oxygen Contamination in Sodium Bond of Fuel Pins

Capsule No.	Fuel Pin No.	Analyses of Representative Control Samples	
		O ₂ in Na (ppm)	Avg. O ₂ (ppm)
W2X	5	[308]	[351]
W8X	1	[394]	[351]
W3F	9	[208]	[202]
W4F	10	[196]	[202]
W5F	15	[129]	[125]
W6F	16	[121]	[125]
W7F	27	203 253	228
W8F	30	61 66	64
W10F	18	183 174	179
W12F	32	80 75	78
W14F	8	182 170	176

"As received" Sodium analysis (ppm)

Al	2	Mn, Cr, Ni, V, Be, Ag, Sr, Li	< 1 each
Fe	3	B, Co, Sn, Pb, Y, Ti, Mo, Bi	< 5 each
Mg	1	Zr, Rb, O	<10 each
Cu	2		
Si	15		
Ca	5		
K	40		
C	1.7

Figure 25 Pre-Irradiation Outside Diameter Profilometer Measurements of Fuel Pin W2X
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Table 13. Transducer Profilometer Diametrical Measurements

No.	Avg. Dia. of Fuel Pin in Na Bond Region (in.)	Avg. Dia. of Fuel Pin in Plenum Region (in.)	Avg. Difference Between Regions (in.)
W2X	0.30045	0.30026	0.0002
W8X	0.30040	0.30010	0.0003
W3F	0.30035	0.29994	0.0004
W4F	0.30020	0.29980	0.0004
W5F	0.25003	0.24960	0.0004
W6F	0.25106	0.24980	0.0004
W7F	0.30030	0.30000	0.0003
W8F	0.30041	0.29999	0.0004

Table 14. Principle Fuel Pin Dimensions

No.	Tube Wall Thickness (Inch)	Tube O.D. (Inch)	Pin Length (Inch)	Pin Weight (g)	Na Weight (g)	Pellet Stack Length (Inch)
W3F	0.0122	0.3004	28.485	178.46	2.90	10.254
W4F	0.0123	0.3002	28.498	178.64	2.49	10.233
W5F	0.0102	0.2500	28.495	135.17	1.48	10.245
W6F	0.0102	0.2511	28.492	134.44	1.48	10.248
W7F	0.0121	0.3003	28.500	175.38	2.89	10.257
W8F	0.0121	0.3004	28.475	178.87	2.49	10.246
W10F	0.0101	0.2509	28.499	134.03	1.46	10.251
W12F	0.0101	0.2509	28.487	139.17	1.48	10.258
W14F	0.0123	0.3010	28.485	174.71	2.89	10.247
W8X	0.0101	0.3004	28.487	175.31	2.43	10.254
W2X	0.0122	0.3005	28.486	169.99	3.06	10.245

Two types of mechanical energy were used in the pin bonding cycles, namely, vibrational and/or centrifugal. Capsule bonding necessitated the use of vibrational and/or impacting energy.

The vibrational and impact bonding was performed in a 4-inch ID by 48-inch long tube furnace. Temperature uniformity as well as unidirectional cooling was obtained by dividing the furnace into four zones along its length and providing each with its own temperature controller. The muffle could accept up to six capsules or pins per bonding cycle.

Tables 15 and 16 show the bonding history for 11 of the fuel pins and capsules fabricated. The most successful pin bonding treatment consisted of heating the pin to 1000°F, vibrating at temperature for one hour, and cooling unidirectionally to room temperature. Accelerometer readings showed the intensity of vibrations to be approximately 5.7g. The impactor yielded values of about 12.4g.

When persistent defects were encountered, it was necessary to utilize the centrifugal method followed by a vibrational cycle to obtain desired results. The vibrational treatment was necessary to eliminate shrinkage voids formed by non-directional solidification of the sodium during the centrifugal cycle.

The centrifugal bonding device consisted of a section of pipe wound with a heating tape and attached to the hub of a shaft driven at 400 rpm. It had a maximum temperature capability of 650°F; however, power was not applied to the heating tapes after spinning was initiated. It was not possible to obtain a unidirectional cooling pattern using this method, so a subsequent heat treatment in the tube furnace was required.

The most successful bonding treatment for the capsules consisted of heating the capsule to 1000°F, impacting every 2.3 seconds for a total of 38 minutes for 1000 impacts, and then unidirectionally cooling to room temperature.

Table 15. Sodium Bonding History of EBR-II Fuel Pins

No.	Sodium Annulus (a) (Mils)	Cycle No.	Temp. (°F)	Time at Temp. (Hrs)	Type of Mechanical Energy (b)	Approx. Acceler. (g)	Duration Mech. Energy (Hrs)
W2X	32	1	1000	2.5	V	—	2.0
		2	1000	2.5	V	—	2.0
W8X	20	1	1000	2.5	V	—	2.0
W3F	26	1	1000	1.5	V	2.2	1.0
		2	650	1.0	C	—	0.33
		3	1000	1.5	V	5.7	1.0
		4(c)	1000	1.5	V	5.7	1.0
W4F	26	1	1000	1.5	V	5.7	1.0
W5F	10	1	1000	1.5	V	5.7	1.0
W6F	10	1	1000	1.5	V	5.7	1.0
W7F	26	1	1000	1.5	V	5.7	1.0
W8F	26	1	1000	1.5	V	5.7	1.0
W10F	10	1	1000	1.5	V	5.7	1.0
		2	650	0.5	C	—	0.33
		3	600	—	None	—	—
		4	1000	1.5	V	5.7	1.0
		5(d)	600	0.5	V	5.7	0.25
		6(d)	1000	1.5	V	5.7	1.0
W12F	10	1	1000	1.5	V	5.7	1.0
W14F	26	1	1000	1.5	V	2.2	1.0
		2	650	1.0	C	—	0.33
		3	1000	1.5	V	2.2	1.0
		4	600	—	None	—	—
		5	1000	1.5	V	5.7	1.0

(a) Nominal diametral gap.

(b) V = vibrational; C = centrifugal (400 rpm)

(c) This cycle was used to settle dust-like fuel particles to the bottom of the pin.

(d) These cycles were required because the pin was reclaimed from a capsule with a defective weld and rebonded. During the reclamation process, the original sodium bond integrity was destroyed.

Table 16. Sodium Bonding History of EBR-II Capsules

Capsule No.	Sodium Annulus (a) (Mils)	Cycle No.	Time at 1000°F (Min.)	Type of Mechanical Energy (b)
W2X	17.5	1	68	I
W8X	17.5	1	68	I
W3F	17.5	1	90	V
		2	68	I
W4F	17.5	1	90	V
		2	68	I
W5F	42.5	1	90	V
		2	68	I
W6F	42.5	1	90	V
		2	68	I
W7F	17.5	1	90	V
		2	68	I
W8F	17.5	1	68	I
		2	68	I
W10F	42.5	1	68	I
W12F	42.5	1	68	I
W14F	17.5	1	90	V
		2	68	I

(a) Nominal radial gap.

(b) I = Impact bonded; 1000 blows during 38 minutes at 1000°F; unidirectionally cooled to R.T.

V = Vibration bonded for 60 minutes at 1000°F; unidirectionally cooled to R.T.

Figure 26 Flow Diagram for Capsule Fabrication and Assembly

SECTION 8

CAPSULE FABRICATION AND ASSEMBLY

The sequence of operations for the fabrication of the capsules is presented in Figure 26. The weighed charge of extruded sodium was melted in the capsule, the vanadium specimen container and fuel pin were placed in the capsule, and the sodium remelted to allow sufficient seating of these components to permit insertion of the top end plug. After welding of the end plug, the capsules were bonded. Bonding techniques are described in Section 7.5.

Bond quality was determined by eddy current surveys and by full length radiography to determine disposition of the sodium. Other characterization tests shown in Figure 26 were performed in a manner similar to that described for fabrication of the fuel pins.

Standards and specifications for the capsules were generally the same as for the fuel pins. Radiographic documentation of component locations and end plug welds was extensive. End plug weld radiographs were inspected to an ANL accepted specification for capsule end plug welds. No leaks were detected after 2,000 psi helium pressurization for any fabricated capsule.

The container which enclosed the vanadium alloy tensile specimens required no special testing other than radiography and eddy current testing to ensure that they were completely filled with sodium.

SECTION 9

EXPERIMENT PROGRESS

Two capsules, W2X and W8X containing Cr_{23}C_6 -modified fuel were incorporated into subassembly X050 and loaded into EBR-II in February 1969. The subassembly was scheduled to remain in-pile for 7500 MWd (150 full-power days).

Calculations based on chemical analyses of the fuel, and current operating conditions gave the following results.

	<u>W2X</u>	<u>W8X</u>
Linear rating (kw/ft)	24.1	27.0
Specific rating (w.g ⁻¹)	219	219
Target burnup (a/o-HA)	3.6	3.6
Target burnup (MWd/T-fuel)	32,800	32,800
Peak clad mean temp. (°C)	580	600
Peak fuel center temp. (°C)	1,030	1,100

Figure 27 shows peak burnups as a function of time, calculated from exposure data published in EBR-II Operations Reports and ANL Monthly Progress Reports.

It is anticipated that the target burnup will be reached during run 39 and that the subassembly will be discharged at the end of that run early in December 1969.

Exposure of subassembly X050 will be extended to 12,000 MWd, equivalent to a peak burnup of 5.75 a/o (HM) in pins W2X and W8X. The new target exposure will be reached at the end of run 42, approximately the end of June 1970. If the operating power of EBR-II is raised to 62.5 MW before completion of run 42, the subassembly will be discharged. As a result of the increased exposure, pin W8X will achieve a higher burnup than originally designated. On the basis of the swelling data shown in Figure 1, fuel cladding contact will not occur unless the actual swelling rate exceeds the nominal value by 40-100%. The anticipated total burnup of W2X will be within 5% of the design burnup.

Pin W8F was included in subassembly X070 which was loaded into position 3A2 at the start of run 39 B (January 1, 1970).

Figure 27 Cumulative Burnup History of Capsules W2X and W8X

SECTION 10
PREVIOUS REPORTS

All work related to the present investigations and performed prior to the present period was 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,"
- WCAP-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,"
- WARD-3791-27 "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending September 30, 1968,"
- WARD-3791-31 "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending December 31, 1968,"
- WARD-3791-36 "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending March 31, 1969,"
- WARD-3791-40 "Uranium-Plutonium Carbide Development Quarterly Progress Report for the Period Ending June 30, 1969."

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