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BURNABLE POISON ADDITIONS TO UO_2

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Prepared By:

R. C. Brayer
W. P. Chernock

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Argonne, Illinois

4

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General Electric Company
Atomic Power Equipment Department
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San Jose, California

1

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Westinghouse Electric Corporation
Atomic Power Department
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BURNABLE POISON ADDITIONS TO UO_2

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INTRODUCTION

The United States and the European Atomic Energy Community (EURATOM), on May 29 and June 18, 1958, signed an agreement which provides a basis for cooperation in programs for the advancement of the peaceful applications of atomic energy. This agreement, in part, provides for the establishment of a Joint U. S. -Euratom research and development program which is aimed at reactors to be constructed in Europe under the Joint Program.

The work described in this report represents the Joint U. S. -Euratom effort which is in keeping with the spirit of cooperation in contributing to the common good by the sharing of scientific and technical information and minimizing the duplication of effort by the limited pool of technical talent available in Western Europe and the United States.

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Project Engineer	R. C. Brayer
Chemical Analysis	W. C. Taylor
Thermal Testing	C. E. Burdg
Sample Preparation	R. C. Brayer
Metallography and X-Ray Diffraction	G. Zuromsky

BURNABLE POISON ADDITIONS TO UO₂

By

R. C. Brayer and W. P. Chernock

ABSTRACT

Microscopic evaluation of samples containing fused UO₂-3 w/o B₄C after heat treatments for up to two hours from 1600 to 2100°C indicated reaction product particles which were irregularly shaped and partially embedded in the UO₂. The reaction products have been tentatively identified as UB₄ and/or UB₂ on the basis of X-ray diffraction analyses.

Re-evaluation of thermodynamic data for a closed system, typical of that encountered in an operating fuel element, reconfirmed previous conclusions that the formation of significant amounts of volatile boron species is improbable. Additional tests to confirm these conclusions are being initiated under conditions in which UO₂-B₄C mixtures are heated to temperatures up to 2300°C in an axial gradient of 10°C/cm with no artificially imposed radial gradient. Other tests are being initiated with the aim of inducing boron migration by removing gaseous reaction products during testing under conditions in which the ratio of radial to axial thermal gradient is approximately 250.

An irradiation program has been formulated and requests for quotation have been issued for irradiation testing of two elements for burnups up to 5000 MWD/MTU.

I. SUMMARY

During the past quarter an investigation of the characteristics of the reaction between UO_2 and B_4C in flowing argon atmosphere at temperatures up to 2200°C was initiated. This investigation was continued during the present reporting period.

Microscopic evaluations of samples containing fused UO_2 -3 w/o B_4C previously analyzed by X-ray diffraction showed no evidence of sintering up to 2000°C and only slight evidence of sintering above that temperature. Microstructures of all samples containing B_4C were similar and were independent of the reaction temperature. They showed the presence of a globular to irregularly shaped white phase partially embedded in the fused UO_2 . Since UB_4 and/or UB_2 have been identified by X-ray diffraction as reaction products in UO_2 - B_4C mixtures, this phase has been tentatively identified as UB_4 and/or UB_2 . The possibility that the globular appearance of some of the reaction product particles resulted from partial melting has been considered and was discarded since UB_4 and UB_2 have melting temperatures in excess of 2400°C and the presence of B_2O_3 (m. p. 450°C) in the system could not be substantiated.

A reassessment of thermodynamic equilibrium for the postulated UO_2 - B_4C reaction in a closed system for temperatures of 2000°K and 2500°K shows that 0.0002% and 0.1% of the original B_4C content will be reacted before the equilibrium gas pressure necessary to halt the reaction is attained.

A re-evaluation of the B_4C fraction dissociating in an operating fuel element was shown to be no greater than 10^{-8} .

In an attempt to verify the above conclusions, $\text{UO}_2\text{-B}_4\text{C}$ mixtures will be enclosed in tungsten tubes with UO_2 liners and will be heated to temperatures up to 2300°C for time in excess of 100 hours. An axial thermal gradient of $10^\circ\text{C}/\text{cm}$ will be maintained over the fuel element length throughout the test period. Post-test analysis will concentrate on evaluation of boron migration.

If for some unforeseen reason volatile boron species are produced in a closed system and boron migration initiated, condensation of the volatile boron phase should occur at the nearest coldest location which is in the radial direction. No axial migration should occur. However due to the gross consequences of axial migration, the potential for this condition has to be completely assessed. A series of tests designed to purposely generate volatile boron species in test elements operating at thermal gradients in the radial direction 250 times that in the axial direction were initiated. These tests are conducted at centerline temperatures of 2500°C with water cooled cladding. The fuel mixture will be open to a vacuum chamber during test in order to continuously remove the gaseous products, CO_2 , CO , and O_2 , from the reaction zone. The elements for these tests have been compacted and modifications to the test apparatus have been completed. Testing will be completed during the next reporting period.

An invitation for bid for an irradiation test program to determine the extent of axial or radial boron migration under typical power reactor irradiation conditions has been issued. The following two options have been included in the bid request:

Option A: Irradiation of full length rods in a water cooled

power reactor.

Option B: Irradiation of 12-inch long fuel rods contained in capsules in a test reactor.

Preference was expressed for Option A.

Two rods containing UO_2 -0.05 w/o B_4C compacted to $88 \pm 1\%$ of theoretical density will be irradiated for approximately four weeks and 5000 MWD/metric ton U, respectively. An axial gradient typical of water reactor conditions will be maintained on the test samples. Post-irradiation testing will include visual and dimensional inspection, gas collection, and analysis for Xe, Kr, He, CO_2 , CO, radial and axial boron distributions and metallography.

II. INTRODUCTION

This program is aimed at developing methods for fabricating fuel elements containing burnable poisons homogeneously distributed in UO_2 .

The principal objectives of the program are:

A. To develop vibratory powder compaction methods for producing a uniform distribution of a burnable poison in high density UO_2 fuel elements.

B. To investigate the potential for redistribution of the burnable poison within the fuel element by means of out-of-pile thermal gradient tests.

C. To investigate the potential leaching of the burnable poison under defected clad conditions by means of loop testing of defected rods.

D. To irradiate test rods containing burnable poison to determine the potential in-pile boron redistribution.

E. To develop fabrication procedures for fuel elements containing large self-shielding poison particles.

During the first year's effort, electromechanical and pneumatic compaction methods were utilized to produce high density UO_2 fuel rods containing 0.05 w/o B_4C additions with a ternary UO_2 formulation containing:

<u>w/o</u>	<u>Mesh Size</u>
60	6-16
15	30-100
25	< 200

All B_4C was minus 325 mesh.

A series of fuel rods containing 45, 20-gram, incrementally loaded, charges of UO_2 -0.05 w/o B_4C powder were compacted to 88 - 89% of theoretical

density by the electromechanical method. The local boron contents were maintained within the prescribed level of variation of +20% from the nominal loading.

The method for boron analysis evolved as part of the past year's effort was based on fusion of UO_2-B_4C mixtures in sodium carbonate at $1000^{\circ}C$ in order to transform the contained boron to a water-soluble salt which was then used to produce boron quinalizarin complex solutions. These solutions were subsequently analyzed colorimetrically for boron.

At the end of the first year's effort, the potential for axial migration of boron under exaggerated axial thermal gradient conditions and decreased radial thermal gradient conditions at centerline temperatures approaching the melting point of the fuel was investigated. Fuel rods, containing an axially positioned tungsten heater and compacted to 85 - 86.5% of theoretical density were tested in a helium and argon atmosphere and were subjected for two hours to axial* thermal gradient of $40 - 60^{\circ}C/cm$. The radial thermal gradients were minimized by maintaining a clad temperature of $800 - 1150^{\circ}C$. A slight trend toward lower local boron concentration in higher temperature regions of the fuel was observed, although the local concentration of +20% of the average boron value for the rod was maintained.

During the first reporting period of this fiscal year, the potential for axial and radial boron redistribution was evaluated under an axial thermal

* Emphasis was placed on studies of axial migration early in the program on the basis that preferential loss of boron from regions of the fuel in power peaks would increase the peaks and lead to potentially unsatisfactory conditions.

gradient* from 8 to 12°C/cm at a centerline temperature up to 2000°C for approximately two hours. The geometry and method of preparation of these test rods were similar to those used in the first series of tests described previously.**

The results of this later series of tests indicated that:

- A. Significant axial boron migration had not occurred.
- B. Significant radial boron migration had not occurred.
- C. A higher boron concentration was present in the outer 20 v/o of the fuel in both the as-compacted and thermally tested conditions. There was no significant increase in the content in this "skin" layer as a result of testing under thermal gradient conditions. Furthermore, there was no significant decrease in boron content in the other 80 v/o of the fuel as a result of thermal gradient testing. The higher boron content at the "skin" of the fuel was attributed to the increase in fines in this region resulting from the decreased average interstice size found when large particles and physical boundaries (cladding) interact.

In order to explain the slight trend toward lower local boron concentration observed in the higher temperature region of the fuel under the 40 - 60°C/cm axial thermal gradient, during the first test series, and the absence of radial boron movement under a 3000 to 4000°C/cm radial thermal gradient, during the second test series, a B₄C-UO₂ stability study was initiated during the

* Typical of that encountered in power reactors.

** Brayer, R. C. and Chernock, W. P., CEND-181 (November 1963), Burnable Poison Additions to UO₂, Summary Report; and Brayer, R. C. and Chernock, W. P., CEND-197 (October 1963), Burnable Poison Additions to UO₂, Quarterly Progress Report.

last quarter. Two series of experiments were conducted under isothermal conditions in flowing argon. Samples containing 3 w/o B₄C-97 w/o UO₂ mixtures were held for 2 hours at temperatures ranging from 1600 to 2200°C. UB₄ was formed at temperatures as low as 1600°C. UB₂ was identified at higher temperature and appeared to be an intermediary stage in the dissociation of UB₄. Samples containing 0.2 w/o B₄C-UO₂ mixtures were heated for up to 2 hours in flowing argon to temperatures in the range of 1600 to 2130°C and showed a loss of 50 w/o of the boron present at 1600°C and a retention of less than 6 w/o of the boron at temperatures in excess of 1800°C. A comparison of observed weight losses with those predicted by various possible reactions indicated that the reaction of UO₂ plus B₄C to form CO₂ (or CO plus 1/2 O₂) plus UB₄, followed by dissociation of UB₄, provided good correlations. Thus, in a flowing argon system, where gaseous reaction products can be continually removed, a mechanism for boron volatilization and subsequent migration appeared to have been established. An evaluation of the free energy of the UO₂-B₄C reaction between 2000 and 2500°K indicated that the equilibrium pressure of CO₂ varied from about 7×10^{-5} to 4×10^{-2} atmospheres between 2000 and 2500°K, respectively. The amount of B₄C which will be reacted in an operating element to produce this pressure was found to be negligible. The formation of a volatile boron species was dependent upon whether an "open" or "closed" system was used for testing. Since fuel elements are closed systems, it was concluded that boron volatility should not be a problem. However, in-pile tests as well as additional out-of-pile tests are required to verify this conclusion.

III. B₄C-UO₂ STABILITY

During the last quarter an investigation of the characteristics of the reaction between UO₂ and B₄C in a flowing argon atmosphere at temperatures up to 2200°C was initiated. This investigation was continued during the present reporting period. Evaluation of the results of X-ray diffraction studies conducted on samples containing UO₂-3 w/o B₄C and evaluation of boron chemical analysis combined with weight change data obtained on samples containing 0.2 w/o B₄C verified the following conclusions previously made in CEND-203* for an "open" system:

- A. A UO₂-B₄C reaction occurred.
- B. The reaction proceeded at temperatures as low as 1600°C in flowing argon.
- C. UB₄ seemed to be formed first at lower temperature and apparently decomposed to UB₂ at higher temperatures.
- D. A volatile boron species was formed at the higher temperatures.

The microscopic evaluation of the samples containing fused UO₂-3 w/o B₄C previously analyzed by X-ray diffraction (see CEND-203*) was completed during this quarter. No sintering of the UO₂ was observed up to 2000°C with only slight sintering above that temperature. In order to retain the loosely packed particles of UO₂, B₄C, or reaction products "in situ" during polishing and examination, the heat treated samples were impregnated with "Loctite". Even after this consolidating treatment only a fraction of the polished surface

* Brayer, R. C. and Chernock, W. P., CEND-203, (January 1964), Burnable Poison Additions to UO₂, Quarterly Progress Report.

was retained. However the surface was good enough to identify reaction products absent in the pure UO_2 controls.

The microstructure of all samples containing B_4C were similar and were independent of the reaction temperature. A typical microstructure is shown in Figure 1 and consists of a globular to irregularly shaped white phase partially embedded in the fused UO_2 . UO_2 has been identified as the gray phase in Figure 1. The white phase particles, some of which contain single or multiple gray colored cores, were only observed in thermally treated samples containing UO_2 - B_4C mixtures. Since UB_4 and/or UB_2 have been identified by X-ray diffraction as reaction products in UO_2 - B_4C mixtures, the white phase reaction product in Figure 1 has been tentatively identified as UB_4 or UB_2 . The central gray core in these particles is probably unreacted B_4C .

The possibility that the globular appearance of some of the reaction product particles resulted from partial melting has been considered. These rounded particles were observed after heat treatments at temperatures as low as 1600°C . The melting temperatures of UB_2 and UB_4 were reported in excess of 2400°C^* and are considerably greater than the lowest reaction temperatures attained in these tests. Re-evaluation of X-ray diffraction data reaffirmed the previous conclusion that UB_4 and/or UB_2 were the only new reaction products. A specific evaluation of the X-ray diffraction data was made in search of evidence supporting the presence of B_2O_3 . B_2O_3 is a liquid between 450 and 2250°C and, at 2000°C , its vapor pressure is estimated

* Howlett, B. W., Jnl. Inst. Metals, 2, (1959-1960), 467.

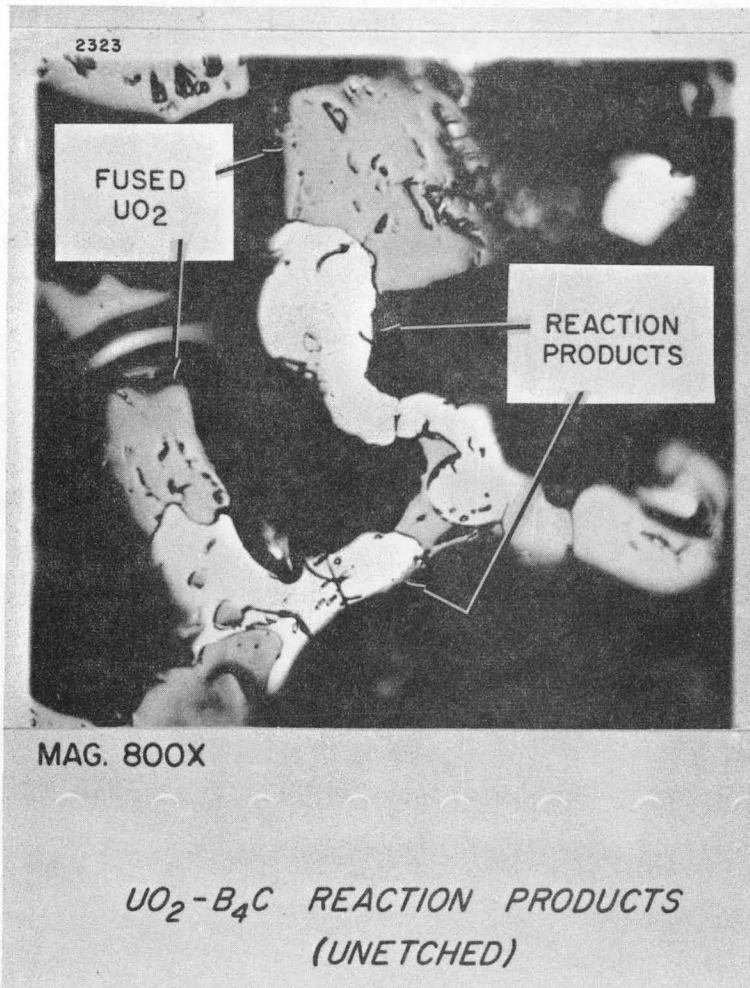
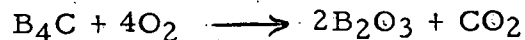


Fig. 1

as 10^{-1} to 10^{-2} atmospheres. B_2O_3 could be formed through the reaction:



If B_2O_3 were found in an "open" system, it is probable that it would volatilize and leave the system. It is improbable that the white phase in Figure 1 is B_2O_3 . On the basis of all of the above data, it was concluded that melting probably did not occur and that the reaction products, shown in Figure 1, are probably UB_4 and/or UB_2 .

A reassessment of the thermodynamic equilibria for the postulated UO_2 - B_4C reaction for temperatures of $2000^\circ K$ and $2500^\circ K$ was made during this reporting period. It was found that the reaction:



should be inhibited when the CO_2 pressure exceeds 6.7×10^{-5} and 4×10^{-2} atmospheres at $2000^\circ K$ and $2500^\circ K$, respectively. Attainment of these equilibrium pressures involves the consumption of 0.0001% and 0.05% of the amount of B_4C present in the element. The equilibrium in the above reaction will be influenced by the dissociation of CO_2 to form CO plus O_2 . The dissociation constant for this reaction in the temperature range of $2000^\circ K$ to $2500^\circ K$ was calculated as approximately 0.3. An increase of 43% of the amount of B_4C consumed in the UO_2 - B_4C reaction was attributed to the influence of CO_2 dissociation. Therefore, the amount of B_4C which may be reacted before the equilibrium gas pressure necessary to halt the reaction is attained is less than 0.0002% and 0.1% of the original B_4C content at $2000^\circ K$ and $2500^\circ K$, respectively. Dissolution of oxygen, produced by CO_2 decomposition, in the UO_2 lattice could alter the equilibrium gas pressures discussed

above. Specific tests with completely closed systems are necessary before final verification of boron transport characteristics can be made. A description of such tests for out-of-pile and in-pile conditions is presented later in this report.

The potential for B_4C dissociation was also thermodynamically re-evaluated for an operating fuel element (closed system). B_4C can dissociate into gaseous monatomic boron and graphite.* The B_4C dissociation pressure, derived from experimental data has been estimated as 2.5×10^{-6} atmospheres at $2100^\circ C$. The calculated dissociation pressure, using heat of formation and calculated thermodynamic data has been estimated as 5×10^{-6} atmospheres at $2100^\circ C$. The agreement is good and the fraction of B_4C dissociating in an operating fuel element should be negligible and no greater than 10^{-8} .

It is thus improbable that UO_2 - B_4C reactions and B_4C dissociation will present problems with respect to increased internal gas pressures or boron migration in an operating fuel element which thermodynamically closely approximates a closed system.

In an attempt to verify the above conclusion by means of out-of-pile tests, a series of "closed-system" experiments have been designed. UO_2 - B_4C mixtures will be enclosed in tungsten tubes with UO_2 liners and will be heated to temperatures up to $2300^\circ C$ for times in excess of 100 hours. An axial thermal gradient of $10^\circ C/cm$ will be maintained over the fuel element length throughout the test period. An artificial radial thermal gradient will not be introduced. Post-test evaluation will concentrate on measurements of boron

* Gilles, P. W. and Robson, H. E., CCC-1024-TR-191 (June 1956), "Vaporization and X-Ray Studies of Boron Carbide".

migration. It is believed that measurements of axial boron migration from these tests will be on the conservative side on the bases that no radial gradients will be imposed and potential absorption of oxygen (produced from decomposition of CO_2 resulting from $\text{B}_4\text{C}-\text{UO}_2$ reactions) by tungsten will drive the system in the direction of enhanced formation of volatile boron species. Even under these conditions, significant boron migration is not anticipated.

Tungsten tubing for the above tests has been received and arrangements have been made for electron beam welding of end caps on vibratory compacted elements. Arrangements are being made for long-term heat treatments with the desired axial gradients.

IV. THERMAL GRADIENT STUDIES

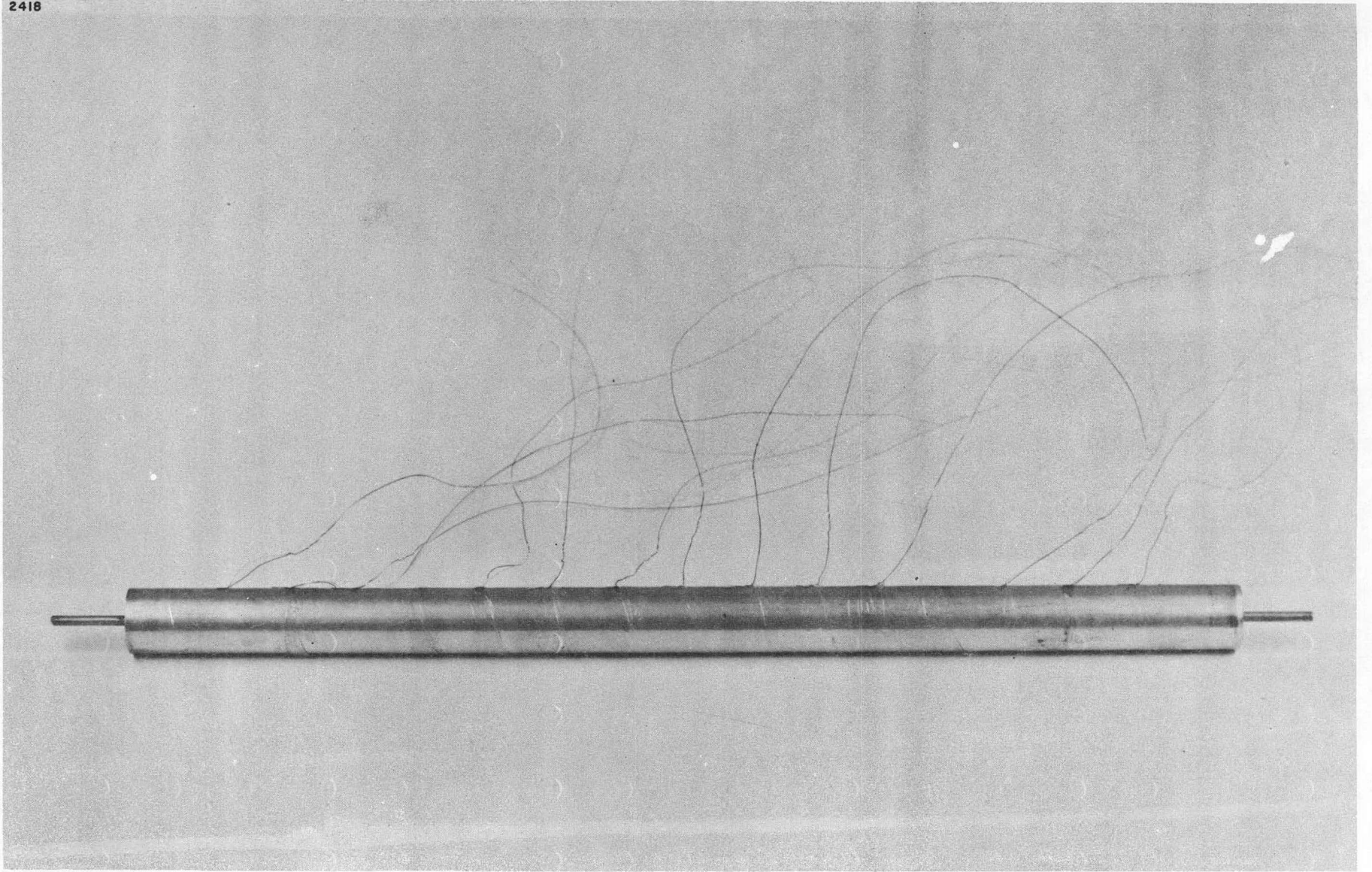
Thermodynamic calculations have shown that, in a closed system, very little UB_4 should be formed as a result of the UO_2-B_4C reaction. In addition, only small amounts of B_4C should decompose to form a volatile boron phase, which is necessary if bulk boron migration is to be encountered. If, for some unforeseen reason, volatile boron species are produced and boron migration initiated, condensation of these volatile phases should occur at the nearest coldest location which is in the radial direction. However, in view of the gross consequences to fuel element operation which might be incurred under conditions of axial boron migration, the potential for such migration cannot be dismissed and must be experimentally assessed. If axial boron migration occurs, movement of boron away from hot spots would increase the magnitude of these hot spots and could lead to burnouts. Radial movement of boron will merely provide minor and acceptable alterations in the self-shielding characteristics of the fuel element.

A series of tests have been designed to purposely generate volatile boron species in test elements operating at thermal gradients in the radial direction 250 times that in the axial direction, representing conditions slightly more severe than those expected in water cooled power reactors. The planned approach is to form volatile boron species at centerline fuel temperatures up to $2500^{\circ}C$ for periods of time up to 30 hours, under conditions in which the gaseous products, CO_2 , CO , and O_2 are removed from the reaction zone, whereas the volatile boron species can condense in the cooler portion of the element.

Three fuel elements, containing UO_2 -0.05 w/o B_4C compacted around a tungsten-25% rhenium heating element, have been fabricated for this investigation using previously developed and described methods. The tungsten-25 w/o rhenium heater was substituted for the tungsten heater, used in previous tests, as a result of better temperature-resistivity characteristics. In order to attain the 2500°C fuel centerline temperature desired for these tests, a heat flux greater than 50 w/cm^2 is required. This higher heat flux exceeds the permissible limits of the equipment previously used in thermal gradient tests. The solid metal heat transfer medium previously used between the test element cladding and the heat sink was replaced by direct water cooling of the test element cladding. Modifications to the test apparatus were made to permit continuous evacuation of the element during thermal gradient testing.

In order to provide the desired axial thermal gradient while maintaining the radial to axial thermal gradient ratio of 250, the stainless steel cladding was insulated from the coolant by means of Mylar films of variable thickness. This was accomplished by wrapping layers of 0.01 mm thick Mylar tape around the clad. The thin Mylar tape was held in place by Dow Corning adhesive #280 which has a curing temperature of 200°C . The temperature of the clad was recorded by means of Teflon insulated thermocouples, directly spot welded on the clad. The fuel element, prior to being inserted in the cooling jacket, is shown on Figure 2. The fuel element was mounted in the jacket in a manner which permitted the thermocouple wires to leave the system through a vacuum seal, which maintained the integrity of the water and vacuum systems.

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TEST SPECIMEN

Fig. 2

This was accomplished by casting the Teflon coated wire in a tin bismuth alloy called Cerrotu. A series of thermal gradient tests using this arrangement have been initiated and will be completed during the next reporting period.

V. IRRADIATION TESTING

Final verification of the feasibility of $\text{UO}_2\text{-B}_4\text{C}$ as stable burnable poison containing fuel elements can only be obtained as a result of an irradiation test program. An "Invitation for Bid" for an irradiation test program has been transmitted to four installations believed to be best qualified to undertake such a program. The specific objective of the irradiation program is to determine the extent of axial or radial boron migration under typical power reactor irradiation conditions. The following two options have been included in the bid request:

Option A: Irradiation of full length rods in a water cooled power reactor.

Option B: Capsule irradiation of 12-inch long fuel rods in a test reactor.

It was specified that prime consideration will be given to those proposals offering an arrangement for irradiation under Option A.

The technical program presented as a guide for preparation of proposals suggested irradiation of two rods containing $\text{UO}_2\text{-0.05 w/o B}_4\text{C}$ compacted to $88 \pm 1\%$ of theoretical density. The first rod will be irradiated for four weeks under Option A or one test reactor cycle under Option B. The second rod, subject to examination of the first rod, would be irradiated to approximately 5000 MWD/metric ton U. The irradiation would be carried under maximum fuel center temperature consistent with the requirement that the melting point of UO_2 will not be exceeded at any time during irradiation. An axial gradient of approximately 10°C/cm will be maintained under Option B. This require-

ment was waived in the case of Option A and the axial temperature profile attained in the power reactor was indicated as acceptable. A program for post-irradiation testing of the fuel elements would consist of visual and dimensional inspection, gas collection, and analysis for Xe, Kr, He, CO₂, CO, radial and axial boron distributions, and metallography. Proposal submittal, subcontractor selection, Commission approval and contract award are scheduled during the first half of the next quarter.

VI. PLANNED EFFORT DURING THE NEXT REPORTING PERIOD

- A. UO₂-B₄C compatibility tests in closed system representative of an operating fuel rod, which have been initiated in the present reporting period, will be completed.
- B. Thermal gradient tests at high fuel centerline temperatures and with a radial to axial thermal gradient ratio of 250, initiated during the present reporting period, will be completed.
- C. Proposals for irradiation test services will be reviewed and a recommendation for a subcontractor will be made to the Commission.
- D. Investigation of the potential for selective boron leaching under defect clad conditions will be completed for exposures up to 100 hours in a pressurized water loop at 120 kg/cm², 310°C and at velocity of 4.5 to 6 m/sec.
- E. Development of fabrication methods for providing a uniform distribution of larger self-shielded B₄C particles in vibratory compacted fuel rods will be initiated and completed.