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CEND-3107-228
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TID-4500, 38th Edition

PROGRESS REPORT
BURNABLE POISON ADDITIONS TO UO_2

For Period
October 1 to December 31, 1964

Prepared by:

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Contract AT (30-1)-3107
U. S. Atomic Energy Commission

Issued February 1965

Unclassified
Classification

Wm. H. Simon 2/26/65
Authorized Classifier Date

NUCLEAR DIVISION
COMBUSTION ENGINEERING, INC.
WINDSOR, CONNECTICUT

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BURNABLE POISON ADDITIONS TO UO_2

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FOREWORD

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.

BURNABLE POISON ADDITIONS TO UO_2

By

R. C. Brayer and W. P. Chernock

ABSTRACT

Metallographic evaluation of the UO_2 -0.05 wt% B_4C element which was thermally tested under both radial and axial thermal gradients, has indicated formation of columnar grains; thus substantiating the estimated fuel centerline temperature of about 2400°C . No boron migration was detected in this element.

Attempts to obtain additional information on the influence of temperatures in excess of 1800°C in inducing boron migration in closed systems, operating with an axial thermal gradient for times in excess of 100 hours were unsuccessful as a result of observed cracks in the tungsten cladding after test. Additional tests utilizing a tungsten liner in more ductile cladding have been planned.

The enrichment for the fuel rod irradiations in Saxton has been fixed at 6% and is based on anticipated fuel centerline temperature of 2200°C and a power rating of 17.8 kw/ft. Orders for fuel cladding and structural materials have been placed and are scheduled for delivery in January 1965. Rods for the irradiation test program will be fabricated in February and inserted in Saxton in March 1965 for irradiation to target burnups of 2000 and 5000 MWD/MTU.

Additional development of fabrication processes for the irradiation test elements has indicated that a loss of 1% in theoretical density may be encountered as a result of heat treatment of the UO_2 particles to reduce the nitrogen content.

I. SUMMARY

Evaluation of microstructure constituents in thermally tested rods has indicated that the target temperature of 2400°C was probably attained. No significant axial or radial boron migration was observed in this system which was purged continuously with helium in an effort to promote boron movement. It was postulated that, if $\text{UO}_2\text{-B}_4\text{C}$ reactions did occur, radial movement within a given radial section used for analysis would not have been detected. It was also suggested that local conditions may have provided a closed system which would provide the necessary equilibrium pressures to stop the reaction.

The attempt to determine $\text{UO}_2\text{-B}_4\text{C}$ stability in tungsten capsules at temperatures up to 2200°C was unsuccessful as a result of cracking of the tungsten cladding. Thus, a closed system was converted to an open system. Beads containing B_2O_3 were observed on the inner surfaces of the tungsten capsules. The observation of B_2O_3 has required a reassessment of the previously proposed mechanism for $\text{UO}_2\text{-B}_4\text{C}$ reactions. A re-evaluation of the potential for UB_2 formation has substantiated the existence of this material, indicating that the previously proposed reaction mechanisms are at least partially correct.

The fuel enrichment for the Saxton irradiation was established as 6% and will result in a fuel centerline temperature of $2200^{\circ}\text{C} \pm 350^{\circ}\text{C}$ and a power rating of 17.8 kw/ft. These rods are scheduled for insertion in March 1965. Fuel densities of about 87% of theoretical are anticipated as a result of evaluations performed on depleted UO_2 indicating a loss of 1% in theoretical density resulting from the nitrogen removal heat treatment.

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 redistribution of the burnable poison within the fuel element by means of out-of-pile thermal gradient tests.
- C. To develop fabrication procedures for fuel elements containing large self-shielded poison particles.
- D. To irradiate test rods containing burnable poison to determine the potential in-pile boron redistribution.

During the first year's effort, electromechanical vibratory compaction methods were utilized to produce UO_2 fuel rods containing 0.05 wt% B_4C additions to densities of 88 to 89% of theoretical. The local boron contents were maintained within $\pm 20\%$ of the nominal loading.

Fabrication methods were subsequently developed for introducing self-shielded B_4C particles in fuel elements containing vibratory compacted UO_2 - B_4C mixtures. This effort was successfully concluded during the last reporting period.

Early in the program, the potential for axial and radial boron redistribution was evaluated for rods containing UO_2 - B_4C mixtures which were vibratory compacted around a tungsten heater rod. An axial thermal gradient of 8 to 12 $^{\circ}\text{C}/\text{cm}$ was maintained for a period of two hours with a maximum fuel centerline temperature of 2000 $^{\circ}\text{C}$.

These tests indicated that significant radial or axial boron migration did not occur under these conditions.

As a result of investigations of $\text{UO}_2\text{-B}_4\text{C}$ reactions conducted at 2200°C in an open system, a mechanism was postulated for the $\text{UO}_2\text{-B}_4\text{C}$ reaction. A thermodynamic evaluation of this mechanism indicated that, in a closed system, the extent of the $\text{UO}_2\text{-B}_4\text{C}$ reaction would be negligible.

The effort during the present reporting period included the heat treatment of $\text{UO}_2\text{-B}_4\text{C}$ mixtures in sealed tungsten capsules at temperatures up to 2250°C with an axial gradient of 16 to $21^\circ\text{C}/\text{cm}$. In addition, a further analysis was made of the attempt to induce boron migration in a thermal gradient test conducted for a period of 30 hours in a flowing helium atmosphere at temperatures in excess of 2400°C . Flowing helium was used in an attempt to simulate a truly open system. Axial and radial thermal gradients in this element were $15^\circ\text{C}/\text{cm}$ and $4500^\circ\text{C}/\text{cm}$. No significant axial or radial boron migration was detected.

Major emphasis during this reporting period was placed upon making the necessary preparations for the irradiation, in Saxton, of two fuel rods containing vibratory compacted UO_2 with burnable poison additions.

III. THERMAL GRADIENT STUDIES

Results¹ of previous efforts on this phase of the program failed to indicate significant boron migration even when an attempt was made to induce such migration by simulating open system conditions in a slowly flowing helium atmosphere. The UO_2 -0.05 wt% B_4C fuel element was vibratory compacted to 86.8% of theoretical density and was operated for thirty hours at a fuel centerline temperature in excess of 2400°C , a radial thermal gradient of $4500^\circ\text{C}/\text{cm}$ and a radial to axial thermal gradient ratio of about 300.

A typical transverse section of a thermally tested rod is shown in Figure 1. The region of grain growth and the oriented pores are typical of those observed in similar tests of pelletized UO_2 . It is believed that the metallic particles are tungsten which has been transferred from the heater rod probably by means of vapor transport. Previous investigations² performed as part of another joint United States-Euratom Research and Development Program has indicated that the onset of massive grain growth occurred in the temperature range of 1800 to 2000°C . Utilizing these data as an approximate "benchmark," it was concluded that a centerline temperature of 2400°C was probably achieved.

It is interesting to note that significant axial or radial boron migration was not observed even when attempts to induce such migration were introduced by flowing helium through the test sample. This observation is particularly puzzling in view of previous work on this program which indicated significant boron losses for UO_2 - B_4C powder mixtures heated to comparable temperatures in a completely open system in flowing argon.

Fig. 1
TRANSVERSE SECTION OF THERMALLY TESTED ROD

On the basis of this last observation, as well as metallographic indications that temperature in excess of 2000°C were achieved, it is likely that a local $\text{UO}_2\text{-B}_4\text{C}$ reaction may have occurred. Since the significant axial or radial boron migration was not detected, one of the following must have occurred:

1. The reaction zone was limited to a volume small enough to permit boron migration within the limits of accuracy of the methods used to determine its presence. In this case, radial movement of short distances within the specific radial sample width would not be detected. Axial movement would have been detected.

2. Local densification of the fuel in the highest temperature regions of the rod may have provided a local closed system in which the volatile products of the reaction reached their equilibrium pressure resulting in a cessation of the reaction.

It is concluded from the above results that significant axial or radial boron migration has not been observed in out-of-pile thermal gradient testing. It is further concluded that the greatest unknown in these data is the potential for radial boron redistribution within a radial sampling zone (see Figure 4 of Reference 1).

IV: UO₂-B₄C STABILITY

An unsuccessful attempt was made to determine UO₂-B₄C compatibility at elevated temperatures in a closed system. Two sealed tungsten tubes containing UO₂-0.05 wt% B₄C powder mixtures were heated to temperatures of 2200°C and 1810°C in vacuum for a period of 100 hours. These rods were run with no induced radial thermal gradient and with an axial thermal gradient of 16 °C/cm and 21 °C/cm for the lower and higher temperature rods. After heat treatment, a large axial crack was found in the weld seam of one test element and a loss in cladding integrity was revealed by means of helium leak testing in the second element. Loss of cladding integrity prohibited the planned collection and identification of gaseous reaction products of the UO₂-B₄C reaction. Furthermore, loss of clad integrity converted the planned test of a closed system into one typical of an open system. Unfortunately, the result of this test was merely verification of results obtained previously in this program in a flowing argon, open system. As was expected, large losses in boron occurred. However, one significant new observation was made. Small (1 to 2 mm diameter), colorless, transparent beads were found deposited at the top of the plenum in the coldest portion (about 1500°C) of one of the capsules. An X-ray diffraction analysis indicated that this material was nearly amorphous. However, two lines of B₂O₃ were observed. Previous investigations³, on this program, have indicated UO₂-B₄C reactions in open systems with the subsequent formation of UB₄ and/or UB₂, depending upon temperature. However, B₂O₃ was not observed, probably because the experimental arrangement did not provide for a cooler deposition surface.

The formation of UB_2 as one of the products of the UO_2-B_4C reaction has been reconfirmed through an extensive re-evaluation of previous data. Although no reactions between UO_2 and W* have been reported in the literature for temperatures in the range of interest to this program, it was suggested⁴ that such reactions were possible after only three hours at $2000^\circ C$ and that one of the reaction products may exhibit a crystal structure which was isomorphous with UB_2 . Although such a reaction was unlikely, it was imperative that this observation be evaluated since the formation of UB_2 has considerable influence on the mechanisms which can be postulated for the UO_2-B_4C reaction. Pellets containing 9 and 50 wt% UO_2 in tungsten were heated for three hours at $2000^\circ C$ in an argon atmosphere. Diffraction patterns of these materials revealed lines for tungsten and UO_2 with no trace of extra lines. On this basis, as well as the re-evaluation of the previous diffraction patterns of UO_2-B_4C reaction products, it was firmly established that UB_2 can be formed in addition to UB_4 . Thus, any accurate description of the mechanism for UO_2-B_4C reactions must permit the formation of UB_2 . Thus, the reaction mechanism proposed earlier³ in this program must be at least partially correct. However, the observation of B_2O_3 on the surface of the failed tungsten capsules, noted earlier in this section, was not included as a factor in this mechanism. Thus, a re-evaluation of reaction mechanisms is in order. Unfortunately, experimental data are inadequate to provide a firm basis for the establishment of a single mechanism. Nevertheless, such a re-evaluation has been initiated in an effort to factor in the influence of B_2O_3 formation on previously proposed mechanisms.

* In these open system evaluations, the UO_2-B_4C mixtures were placed on a tungsten sheet.

V. IRRADIATION TESTING

During this reporting period, considerable effort has been directed toward the preparation for the irradiation in Saxton of two fuel rods containing vibratorily compacted UO_2 plus 500 ppm boron as B_4C . The principal objective of the irradiation program is to determine the potential for boron migration in irradiation and thermal environments typical of those encountered in large water-cooled power reactor fuel rods.

Two fuel rods are to be irradiated in a 2 x 2 removal rod assembly in the Saxton reactor to target burnups of 2000 and 5000* MWD/MTU. The fuel rod design shown in Figure 2 is similar to that for Saxton rods, with minor modifications to provide for vibratory compacted fuel. The bottom end plug design was modified to prevent collection of fine particles of fuel or B_4C in the space between the bottom end plug and the cladding. In addition, a high density ceramic disc has been placed at the top of the fuel column to provide support for the fuel expansion marker, a device which is aimed at providing information concerning fuel thermal expansion.

The cladding material will conform as closely as possible to that utilized for the Saxton core loading and will be fabricated from welded and cold drawn (10% cold work) type 304 stainless steel. Cladding specifications are given in Appendix A.

The fuel loading will contain 6%**enriched, compaction-grade, fused crystalline UO_2 , to which 500 ppm boron will be added in the form of fused B_4C . Specifications for the UO_2 are given in Appendix B.

*The maximum burnup will be fixed by the scheduling of the plutonium loading for Saxton. Although 5,000 MWD/MTU is a desired target a burnup in the range of 3000 to 5000 MWD/MTU will be considered as an adequate test of the concept.

**The basis for the selection of this enrichment will be discussed later in this section.

Fig. 2
FUEL ROD DESIGN

The nitrogen content of the fuel has been limited to 100 ppm on the basis of minimizing internal gas pressure. It is believed that this low nitrogen level can be achieved as a result of a four hour heat treatment at 1400°C in vacuum or hydrogen. This heat treatment will be performed by the UO₂ supplier. The particle size formulation selected for use in fabricating these fuel rods is shown in Table I and is based upon results of vibratory compaction investigations performed earlier in this program. Recent studies have verified the selection of this particle size distribution as optimum for rods of this diameter, especially in view of the superimposed requirement of maintaining a uniform axial boron loading (within $\pm 20\%$ of the nominal loading).

Recent studies were aimed at determining the influence of the nitrogen-control heat treatment on compaction density and homogeneity. A lot of UO₂ was split into two sections. One section was vacuum annealed at 1400°C, whereas the other section was not heat treated. Compactions were made utilizing both types of powder, formulated according to Table I. The dimensions of the rods were similar to those selected for the irradiation test program and the method of loading and compaction was identical to that used previously in this program. In all instances, a loss of 1% in theoretical density was observed for the rods containing heat treated UO₂. Thus, a density of 87% of theoretical is anticipated for the irradiation test elements.

A limited evaluation of the difference between the two types of UO₂ powder, described above, failed to show significant positive trends. The O/U ratio remained at 2.01 for both powders and mercury pycnometer determinations indicated similar particle densities.

TABLE I

FUEL FORMULATION

Allowable Distribution				
Group	U.S. Mesh Size	U.S. Mesh Size	%	Quantity %
I	-6 +16	-6 +8	37 - 40	60
		-8 +10	18 - 20	
		-10 +12	17 - 18	
		-12 +16	23 - 27	
II	-30 +100	-30 +60	40 - 60	15
		-60 +100	40 - 60	
III	-200	-200 +325 -325	15 85	25

Grade	U.S. Mesh Size	Quantity
Norbide	-325	To provide 500 ppm natural boron

Microscopic evaluation at high power failed to show differences in the extent of cracking in particles taken from both types of fuel. However, low power microscopic observations under oblique lighting indicated a bright orange color reflected by most heat treated particles. Only very few particles from the as-received (non-heat treated) powder showed this effect. The cause of this phenomenon has not been established. During handling of the powders, it was observed that the heat treated UO_2 was less free-flowing than the as-received material. This may result in more sticking, less than optimum distribution and slightly lower densities.

The fuel enrichment was established by Westinghouse⁵ on the basis of a nuclear analysis for nominal (expected) specific power and the most adverse peak specific power in Saxton for enrichments of 6.0, 6.48 and 6.66%. Specific power was calculated for a Saxton 2 x 2 assembly positioned in the N1 position for the following conditions:

1. All four rods contain UO_2 pellets (93% theoretical density) with 8.52% U-235 enrichment.
2. Two of the four rods containing pellets are replaced by vibratory compacted rods (88% theoretical density) containing 6.48% enriched UO_2 and 500 ppm natural boron.
3. Same as condition 2, with 6% and 6.66% enriched UO_2 .

In all cases, the reactor power was assumed to be 20 MW (thermal) producing 12-13 kw/ft in reference Saxton elements. Results of peak power calculations are shown in Table II. Maximum fuel centerline temperatures were calculated on the basis of utilizing an $\int k d\theta$ for vibratory compacted powder of 80% of that for pellets.

TABLE II
SPECIFIC POWER (kw/ft) IN 2 x 2 ASSEMBLY

Enrichment (%)	8.52	6.00	6.48	6.66
Fuel Type	Pellets	Vipak	Vipak	Vipak
Nominal (expected) Maximum	24.2	17.8	19.2	19.7
Most Adverse Peak	28.7	21.1	22.8	23.4

The basis for selection of this value was the result of irradiation tests performed by Westinghouse in GETR. Results of peak fuel centerline temperature calculations are shown in Table III. Evaluation of the data shown in Table III indicates that 6% enrichment will provide power ratings and peak fuel centerline temperatures consistent with present trends in water reactor technology. On this basis, this enrichment was selected for this program.

A cross-check of analytical procedures for boron analyses at Westinghouse and CE was successfully completed. Excellent agreement has been obtained on analyses of homogenized $\text{UO}_2\text{-B}_4\text{C}$ powder mixtures.

Insertion of fuel rods in Saxton was rescheduled for the first week in March on the basis of delays in enrichment selection, resulting in postponement of delivery of UO_2 . In addition, the delivery of the type 304 stainless steel tubing was also delayed as a result of rejection by the vendor of the first lot of tubing. Fuel and cladding material are scheduled for delivery in January. All other hardware is being fabricated.

TABLE III

PEAK FUEL CENTERLINE TEMPERATURE

Vibratory Compacted $\text{UO}_2\text{-B}_4\text{C}$
500 ppm boron
88% Theoretical Density

<u>Enrichment %</u>	<u>Nominal Power Rating (kw/ft)</u>	<u>Peak Fuel Centerline Temperature ($^{\circ}\text{C}$)</u>
6.0	17.8	2200 $\pm$ 350
6.48	19.2	2350 $\pm$ 350
6.66	19.7	2400 $\pm$ 350

VII. PLANNED EFFORT DURING THE NEXT REPORTING PERIOD

Major emphasis will be placed upon the fabrication of fuel rods for irradiation testing in Saxton. Rods containing depleted UO_2 will also be fabricated and analyzed for local density and boron variations to provide baseline data for post-irradiation evaluations.

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APPENDIX A

Specification for Welded and Cold Drawn Stainless Steel Tubing

1. This specification covers welded, cold drawn, bright annealed, cold worked (approximately 10% reduction in area), and flash pickled stainless steel tubing (AISI 304), manufactured from cold rolled strip and having close tolerances. This specification covers tubing sizes having an inside diameter of less than $\frac{1}{2}$ inch and wall thickness of 0.010 to 0.030 inches.
2. No change shall be made in the quality of successive shipment of material furnished under this specification without first obtaining the approval of the purchaser.

MANUFACTURE

3. PROCESS: The steel shall be made by the electric furnace process.
4. MANUFACTURE: (4.1) The tubing shall be formed from cold rolled stainless steel strip continuously welded along its entire length. It shall then be cold drawn, bright annealed, cold drawn to size, and flash pickled.
(4.2) The amount of cold reduction shall be sufficient to remove any dimensional indications of welding, and the weld shall be metallurgically sound through the full wall thickness of the tubing.
5. ANNEALING: After the first cold reduction, the tubing shall be thoroughly and uniformly annealed to subsequently meet the mechanical properties after the final cold reduction. All traces of drawing compound shall be removed before any annealing operation.
6. FINAL COLD REDUCTION: After bright annealing, the tubing shall be given a final cold draw of approximately 10 per cent reduction in area, but in no case more than 15 per cent, to obtain the mechanical properties specified in Sections 13 and 14.

7. PICKLING: After the final bright anneal, cold finish, straightening, cleaning, and inspection operations, the tubing shall be flash pickled to remove traces of surface oxidation and contamination. The tubes shall be thoroughly rinsed in hot and cold water to remove residual pickling solution. Particular attention shall be given to the inside of the tubes and individual rinsing of the inside surface may be necessary. The tubes shall not be pickled, ground, or polished for metal removal as a final operation.
8. REPAIR OF DEFECTS: The repair of defects on finished tubing by welding, grinding, abrading, or any other processes is not permitted. In process repair operations will be acceptable if the finished product conforms to the requirements of this specification.
9. ENDS: The tubing shall be furnished with square cut ends. The ends shall be deburred with a maximum chamfer of 0.002 inches in the inside and outside diameters.

CHEMICAL PROPERTIES AND TESTS

10. CHEMICAL COMPOSITION: The tubing shall conform to the following requirements as to chemical composition as determined by ladle and check analysis:

Carbon	0.06 - 0.08 Percent
Manganese, Max	2.00 "
Phosphorus, Max	.030 "
Sulfur, Max	.030 "
Silicon, Max	.75 "
Cobalt, Max	As specified on P O
Nickel	8.00 -11.00 Percent
Chromium	18.00 -20.00 "
Iron	Remainder

Rare earth additions shall not be made.

11. LADLE ANALYSIS: An analysis of each heat of steel shall be made by the steel manufacturer to determine the percentages of the elements specified in Section 10, the chemical composition thus determined or that determined from a check analysis made by the tube manufacturer, if the latter has not manufactured the steel, shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 10.

12. CHECK ANALYSIS: (12.1) An analysis of either one length of flat rolled stock or one tube shall be made on each heat of steel.

(12.2) Samples for chemical analysis shall be taken in accordance with ASTM E-59, Method of Sampling Steel, Cast Iron, Open Hearth Iron, and Wrought Iron for Determination of Chemical Analysis.

(12.3) The chemical composition thus determined shall conform to the requirements specified in Section 10.

(12.4) If the analysis of one of the tests specified in Sections 11 or 12 does not conform to the requirements specified in Section 10, an analysis of each length of flat rolled stock or tube from the same heat may be made, and all lengths of flat rolled stock or tubes conforming to the requirements will be acceptable so far as chemical composition is concerned.

MECHANICAL PROPERTIES AND TESTS

13. ROOM TEMPERATURE TENSILE PROPERTIES: (13.1) The tubing shall conform to the following requirements as to room temperature yield strength.

Yield Strength, Psi (Guarantee Value)	75,000 - 100,000
Yield Strength, Psi (Aim Value)	80, 000 - 90,000

(13.2) The ultimate tensile strength and elongation of the tubing shall be determined for information only. It is desired that the properties conform to the following:

Tensile Strength, Psi, Min	100,000
Elongation in 2", Percent, Min	
Longitudinal, Full Section Specimen	*35

(13.3) The tensile properties shall be determined in accordance with ASTM A-370 using the 0.2 per cent offset method when determining yield strength and using a full size tube section.

*For tubes having a wall thickness less than 0.015 inch and/or an O.D. of less than 0.125 inch, the elongation shall be 30 per cent min.

16. REVERSE FLATTENING TEST: A section of finished tube, 4 inches in length, shall be split longitudinally 90 degrees on each side of the weld. The sample with the weld shall be flattened with the weld at the point of maximum bend. There shall be no evidence of lack of weld penetration over laps from weld flash, or cracks in the sample.

17. HYDROSTATIC TEST: (17.1) Each tube shall be tested at the pressure P computed by the following formula:

$$P = \frac{2ST}{D}$$

Where:

P = hydrostatic test pressure in Psi,
S = minimum yield strength (75,000 Psi)
T = minimum wall thickness in inches
D = outside diameter in inches

(17.2) The hydrostatic pressure shall be maintained for not less than 5 seconds.

18. INTERGRANULAR CORROSION TEST: As evidence that the steel is not susceptible to intergranular corrosion, test specimens representative of the material furnished, when tested in accordance with ASTM A-393 Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steels shall not have lost their metallic ring when dropped on a stone or other hard, flat object. The specimens shall not show cracks or other evidence of disintegration after flattening full size sections to the H value specified in Section 15 or bending strip sections through 180 degrees around a diameter equal to the thickness when examined at a magnification not exceeding 5X to 15X.

19. EDDY CURRENT INSPECTION: (19.1) Each tube shall be subjected to an eddy current inspection and shall be free from cracks, seams, slivers, inclusions and other defects having a depth of 10 per cent of the wall thickness or more.

(19.2) The test procedure shall be as agreed upon by purchaser and manufacturer. Questionable areas showing indications of 10 per cent or less of the wall thickness may be investigated by rubbing with emery paper. Tubing so investigated, shall be re-eddy current tested. If the surface must be prepared for re-eddy current testing, the tubing shall be flash pickled as a final operation.

(19.3) Other types of nondestructive testing which will provide the specified quality of tubing shall be as agreed upon by purchaser and manufacturer.

20. TEST SPECIMENS: (20.1) (Tension) Room temperature tensile test specimens shall be taken longitudinally in accordance with ASTM A-370 and the elevated temperature test specimens shall be in accordance with ASTM E-21.

(20.2) (Flattening) The flattening test specimens shall be taken from the end of finished tubes just prior to the final flash pickling operation.

(20.3) (Corrosion) Corrosion test specimens shall be taken in accordance with ASTM A-393.

21. NUMBER OF TESTS: (21.1) (Room Temperature Tension Tests) Not less than 3 room temperature tension tests shall be made for each group of 5,000 feet or less of finished tubing. A group shall consist of tubes of the same size, from the same heat, and processed in the same manner.

(21.2) (Elevated Temperature Tensile Tests) Not less than 2 elevated temperature tensile tests shall be made for each group of tubing. A group shall be defined as in Section 21.1.

(21.3) (Flattening and Reverse Flattening) Not less than 2 flattening and 2 reverse flattening tests shall be made for each group of 5,000 feet or less of finished tubing. A group shall be defined as in Section 21.1.

(21.4) (Hydrostatic) Each tube shall be tested hydrostatically.

(21.5) (Corrosion) Not less than 3 intergranular corrosion tests shall be made on finished tubing of the same size, from the same heat, and manufactured by the same procedures representing the same finishing treatment.

(21.6) (Eddy Current Test) Each tube shall be eddy current inspected.

22. RETEST: If the results of the mechanical tests of any group or lot do not conform to the requirements specified in Sections 13, 14, 15, 16 and 18, retests may be made on additional tubes of double the original number from the same group or lot, each of which shall conform to the specified requirements.

DIMENSIONS

23. PERMISSIBLE VARIATIONS: (23.1) The dimensions of the tubing shall not vary from that specified more than the following:

<u>Dimensions</u>	<u>Permissible Variation</u>
Inside Diameter	± 0.0005 inch
Wall Thickness	± 5 per cent
Ovality of I.D.	Within I.D. tolerance
Length	As specified
Camber	Camber shall not exceed 0.010 inches per foot

(23.2) Once the manufacturing procedures have been established for attaining nominal size value, all tubing shall be processed without change.

(23.3) The O.D. and I.D. dimensions representative of each group of tubing defined in Section 21.1 shall be determined for the tubing after bright annealing and before the final cold drawing operation.

FINISH

24. FINISH: The tubing shall be flash pickled as a final manufacturing operation. It shall be clean, free of surface contamination, and shall be uniform in quality and condition. The surface shall be smooth and free from cracks, seams, tears, slivers, checks, laps, sinks and any other imperfection.

TEST REPORTS

25. TEST REPORTS: The manufacturer shall furnish 4 copies of a test report showing the results of the tests specified in Section 11, 12, and 21. In addition, 4 copies of the dimensional measurements obtained under Section 23.3 shall be submitted for information only. The report shall include the Purchase Order Number; Purchasing Department Specification Number; Test Identification Number correlating test results with group of tubing represented; and Name of Manufacturer.

PACKING AND MARKING

26. PACKING: The tubes shall be properly packed to prevent injury during shipment.

27. MARKING: Each package or bundle for shipment shall be plainly marked as follows:

Purchase Order Number; Stainless Steel Tubing, Purchasing Department Specification Number; Heat Number; Dimensions; Total Footage; Net Weight; Test Identification Number correlating the test results with the lot represented; Name of Manufacturer.

APPENDIX B

Specification for Fused Crystalline UO₂ Powder

1. This specification covers compaction grade, loose fused crystalline uranium dioxide powder for use in the manufacture of vibrationally compacted fuel elements.
2. No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

MANUFACTURE

3. LIABILITY: The manufacturer shall be financially responsible for all losses and contamination occurring in his manufacturing operation, including repurification, reconversion, into a form acceptable to AEC and unrecoverable losses of uranium, all related shipping charges, and any use charges unless otherwise specified on the Purchase Order.
4. COMPLIANCE WITH REGULATIONS: The manufacturer shall have appropriate licenses and clearances indicating that he is aware of and responsible for complying with all applicable regulations of Federal, State and local regulatory bodies with respect to receiving, accounting for, processing, storing and shipping of UO₂ powder and pellets.

CHEMICAL PROPERTIES AND TESTS

5. CHEMICAL COMPOSITION: The powder shall conform to the following chemical composition:

(5.1) Group I Requirements:

(5.1.1) Elements

Macroscopic Cross Section
Imparted by this Level
of Impurity
 $\text{cm}^2/\text{cm}^3 \times 10^{-5}$

Element	ppm, (Max)	
Al	300.0	1.5
B	1.5	63.7
Bi	2.0	0.0002
Ca	100.0	0.66
Cd	1.0	28.0
Cl	10.0	-
Co	6.0	2.3
Cr	500.0	18.3
Cu	50.0	1.75
Fe	500.0	13.6
In	3.0	3.0
Mg	50.0	0.08
Mn	10.0	1.5
Mo	150.0	2.4
Ni	300.0	14.4
Pb	20.0	0.010
Si	500.0	1.4
Sn	5.0	0.015
Ti	40.0	3.1
V	1.0	0.059
W	50.0	3.0
Zn	20.0	0.19
C	100.0	0.015
F	10.0	0.0003
*N	100.0	8.3

*A vacuum or wet hydrogen heat treatment shall be given to the UO_2 fuel to remove nitrogen present. This heat treatment shall be conducted at a temperature of not less than 1400°C and for a period of not less than 4 hours.

(5.1.2) Oxygen-Uranium Ratio. The ratio of oxygen to uranium in the uranium dioxide shall equal to 2.0 ± 0.02 . The ratio shall be determined as follows:

- (a) The uranium dioxide in a mixed sample shall be converted to U_3O_8 in air until a constant weight of sample is obtained.
- (b) The U_3O_8 sample impurities shall be determined spectrographically.
- (c) The uranium content is 84.8% of the U_3O_8 sample weight less the weight of the impurities. Adjustment for impurities shall not be required if the total impurity level of the U_3O_8 sample does not exceed 500 ppm.

(5.2) Group II Requirements:

(5.2.1) The total rare earth composition (Gd, Sm, Eu and Dy) shall not exceed 0.6 ppm. For calculation of the total thermal neutron absorption cross-section, the macroscopic cross-section absorption imparted by 0.6 ppm of these rare earths is $91.44 \times 10^{-5} \text{ cm}^2/\text{cm}^3$.

(5.2.2) The total moisture content shall not exceed 75 ppm.

(5.2.3) The total uranium content shall be 87.7% by weight minimum.

(5.2.4) The isotopic composition of UO_2 shall not be depleted during fabrication. The U-235 content shall be the same as that of the starting uranium dioxide powder.

(5.3) The total thermal neutron absorption cross-section imparted by all impurities listed in Group I, Sections 5.1 and, in Group II, Section 5.2, shall not exceed $100 \times 10^{-5} \text{ cm}^2/\text{cm}^3$.

The impurity level of the rare earth elements shall be included in the calculation of the total macroscopic cross-section absorption when analyzed.

6. CHEMICAL SAMPLING:

(6.1) The manufacturer shall submit for purchaser approval a sampling plan to control chemical composition.

(6.2) The manufacturer shall submit for approval a sampling plan of a test method such as gamma pulse spectrometry to demonstrate that only the specified enrichment has been processed, stored and shipped.

PHYSICAL PROPERTIES AND TESTS

7. DENSITY:

(7.1) The apparent density of the fused uranium dioxide, as measured by a mercury pycnometer, shall be 99.0% of theoretical minimum.

(7.2) The apparent density shall be determined on a 35 gram representative uranium dioxide sample having a mesh size of minus 6 plus 20. The manufacturer shall state his method of testing.

DIMENSIONS

8. PARTICLE SIZE:

(8.1) The particle size distribution as determined on U.S. Standard Sieves shall conform to the following:

Group	U.S. Mesh Size	Allowable Distribution	
		U.S. Mesh Size	%
I	-6 +16	-6 +8	37 - 40
		-8 +10	18 - 20
		-10 +12	17 - 18
		-12 +16	23 - 27
II	-30 +100	-30 +60	40 - 60
		-60 +100	40 - 60
III	-200	-200 +325	15
		-325	85

TEST REPORTS

9. TEST REPORTS:

(9.1) Three (3) copies of certified analyses for total U, U-235, (U-235 to $\pm 4\%$ of the amount present), and the impurities specified in Section 5 shall be submitted to the purchaser when each powder batch is released for production.

(9.2) The manufacturer shall provide three (3) copies of all inspection and test reports as soon as they are generated.

(9.3) The batch size shall be submitted for purchasers approval.

PACKING AND MARKING

10. PACKING: The packaging shall conform to pertinent ICC and AEC regulations regarding nuclear materials and shall be approved by the purchaser.

11. MARKING: Each container shall be individually and consecutively numbered and shall be plainly marked as follows: Purchase Order Number; Uranium Dioxide Powder, Specification BUPO-101; Gross, Tare and Net Weight; UO₂ Lot Number from which the powder was made; Name of Manufacturer.

INSPECTION

12. INSPECTION:

(12.1) Test and inspection shall be made at the place of manufacture.

(12.2) The manufacturer shall afford the purchaser's representatives free of cost, all reasonable facilities to satisfy themselves that the material furnished is in accordance with this specification.

(12.3) Material accepted by the purchaser's inspector at the place of manufacture which subsequently reveals imperfections not detected at the place of manufacture, or which subsequent tests or analysis show not to be in accordance with this specification, is subject to rejection.