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DEVELOPMENT OF MANUFACTURING PROCEDURES
FOR HFIR FUEL PLATES TO ORNL SPECIFICATION

R. W. Knight
F. L. Sherman

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R. W. Knight and F. L. Sherman

SEPTEMBER 1971

OAK RIDGE NATIONAL LABORATORY
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DEVELOPMENT OF MANUFACTURING PROCEDURES FOR
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R. W. Knight and F. L. Sherman¹

ABSTRACT

Techniques to manufacture the High Flux Isotope Reactor (HFIR) fuel plates were originally developed at the Oak Ridge National Laboratory (ORNL). Since ORNL is not a manufacturing facility and many techniques had to be worked out to improve the efficiency of manufacture of precision parts at a high production rate, a great deal of the work had to be left to the contractor, Texas Instruments Inc. This report covers the development of these production procedures based on the ORNL original fabrication concept. Problems in production such as fuel plate blisters, fuel core outlines, homogeneity measurements, and material are discussed.

INTRODUCTION

The HFIR fuel plates are manufactured according to ORNL specification² HFIR-FE-2 under subcontract 91X-70500-C by Texas Instruments Inc.³ The basic method for the manufacture of these fuel plates was developed at ORNL on a pseudo-production basis.⁴ Texas Instruments Inc developed the basic ORNL process into a highly efficient production process⁵ with production losses presently less than 10% based on over

¹An employee of Texas Instruments Inc, a Division of Texas Instruments, Inc., Attleboro, Mass.

²R. W. Knight and G. M. Adamson, Jr., Specifications for High Flux Isotope Reactor Fuel Elements, HFIR-FE-2, October 1966. Available from R. W. Knight, Oak Ridge National Laboratory.

³Texas Instruments Inc, 34 Forest Street, Attleboro, Mass., 02703, a corporate Division of Texas Instruments, Incorporated.

⁴G. M. Adamson, Jr., Fabrication Procedures for the Initial High Flux Isotope Reactor Fuel Elements, ORNL-4342 (February 1969.)

⁵R. W. Knight, J. Binns, and G. M. Adamson, Jr., Fabrication Procedures for Manufacturing High Flux Isotope Reactor Fuel Elements, ORNL-4242 (June 1968).

60,000 fuel plates manufactured. The results of this development are being reported in a series of reports of which this is the first. Almost 2 years was required to develop processes for the manufacture of the fuel elements and fuel plates. Although this long a development period was not anticipated, the delivery of the first fuel assembly coincided very well with the reactor startup.

The HFIR fuel assembly contains two different types of fuel plates as shown in Fig. 1. The inner plate contains 15.8 g ^{235}U and 0.0164 g ^{10}B . The core of the fuel plate comprises a fuel section with the fuel in the form of U_3O_8 in 10l aluminum powder and a filler section with the ^{10}B in the form of B_4C in 10l aluminum powder. The outer fuel plate has a similar fuel core with 18.44 g ^{235}U and 10l aluminum powder in the fuel section and only 10l aluminum powder in the filler section. The fuel and filler sections mate in a rectangular parallelepiped. The core is made by pressing and sintering all those ingredients into a rectangular parallelepiped sized and shaped to attain the correct dimensions after bonding and rolling. The manufacture of the fuel plates follows the now familiar method with cover plates and frames used throughout the research reactor fuel element industry.

The outer annulus contains 369 fuel plates and the inner annulus 171 for a total of 540 fuel plates in each reactor fuel element assembly (see Fig. 2). Further data on the fuel element manufacture were reported by Knight, Binns, and Adamson.⁵ This report covers the development of the fuel plate manufacture. A second report will be issued to cover the development of the side plate fabrication, fuel element assembly, fuel element assembly, fuel element welding, and fuel element machining.

MANUFACTURING DEVELOPMENT OF FUEL PLATES

Fuel plates for HFIR represented a new breed with closer tolerances of the fuel and boron distribution and fuel plate dimensions. The fuel and filler sections had to be contoured very accurately (Fig. 1) to produce the required flux distribution during reactor operation as well as prevent hot-spot burnout of the fuel plates under the rigorous operating conditions of the reactor. Finished fuel plates for previous reactors were either flat or curved in an arc. The HFIR fuel plates are formed to involute shapes that differ for the inner and outer annuli.

Fig. 1. HFIR Fuel-Loading Cross Section.

Fig. 2. Complete HFIR Fuel Assembly.

Materials

Uranium Oxide

The uranium in the form of U_3O_8 is manufactured by Union Carbide Nuclear Division's Y-12 plant,⁶ as no other manufacturer was available who would make the fuel to the exacting specifications required. These specifications are given in the Appendix. The only change from the original specification requested of the manufacturer has been that the surface area of the fuel be as close as possible to $0.047 \text{ m}^2/\text{g}$. The U_3O_8 is made in approximately 16-kg batches, which are in turn cross blended to a single lot of 225 kg. Each 16-kg lot is analyzed for conformance to the specifications before it is committed to cross blending. This gives the manufacturer an opportunity to select appropriate batches or to recalcine batches, if necessary, to achieve the surface area requested. All U_3O_8 shipped to Texas Instruments Inc is recertified by an independent agency before use, and pilot lots of inner and outer fuel plates are manufactured to test the material under production conditions before it is released to full production use. The precise control over surface area was not required for nuclear properties but to assist in measuring the homogeneity of the fuel plate. The x-ray homogeneity scanners because of their sensitivity respond to differences in particle size and distribution as well as to quantity of uranium in the fuel plate.

The limitation on surface area controls the shape of the particles, the number of fines, and the porosity of the U_3O_8 . The concern over surface area and concomitant particle size led to the requirement that the contents of each shipping container be blended before use to assure a uniform particle size distribution among the fuel plates.

⁶W. J. Werner and J. R. Barkman, Characterization and Production of U_3O_8 for the High Flux Isotope Reactor, ORNL-4052 (April 1967).

Aluminum Powder

The only aluminum powder used thus far has been Alcoa 101 with greater than 80% -325 U.S. standard mesh and 100% -100 U.S. standard mesh. At ORNL such powder had been vacuum annealed at temperatures between 540 and 590°C in lots of 10 lb or less. In production, 50-lb quantities were annealed at one time. Since aluminum annealed above 512°C reacts exothermically and sinters, thick beds cake and the powder becomes useless. For the large quantities, the annealing temperature was reduced to between 510 and 490°C min, and the powder bed thickness was limited to 2 in. Aluminum powder proved to have its own individual characteristic response to vacuum annealing. For a metal, this material is a reasonably good insulator. The original vacuum annealing furnaces at Texas Instruments Inc had, in addition to control and override instrumentation, three probe thermocouples to qualify the furnaces for heating and temperature holding characteristics. Some irregularity was found in the time required to heat various lots of powder; the longest time found was used for production, and no effort was applied to optimize the heating time.

The use of a new Marshall cold-wall vacuum oven with a 20 1/4-in.-ID x 26-in.-long hot zone (Fig. 3), supplied by ORNL, provided answers to the previous variations. This oven has six available thermocouples for qualification, and the trays containing the aluminum powder remain in one location during annealing. This allows very accurate location of the test thermocouples. During heating a difference as much as 260°C was possible at locations in the powder as close as 1/2 in. To overcome this problem, the distance the heat was required to travel through the aluminum powder was decreased by the insertion of a stainless steel grid. The grid extended through and above the powder; its blackened surfaces absorb the radiant heat from the surrounding heating elements and baffles and distribute it to the powder. This arrangement has proved very successful, and the total heating time for annealing has stabilized at 6.5 hr; previous times were greater than 11 hr.

The aluminum powders at ORNL were used shortly after annealing, but at Texas Instruments Inc the powders were backlogged for

Fig. 3. Marshall Furnace for Annealing Aluminum Powder, Showing Arrangement of Trays.

substantial periods. When powders were stored in air, the adsorption of moisture made blending difficult. This problem was subsequently solved by storing the powders under vacuum and controlling the time, temperature, and relative humidity during screening, weighing, and blending. Figure 4 shows the vacuum storage containers. These controls also made the contouring of the powders in the die tops much more uniform. An added unanticipated benefit was that the aluminum powder, being kept substantially dry, absorbed the moisture from the more deliquescent B_4C and provided excellent B_4C homogeneity in the core filler section.

Since aluminum powders are manufactured for commercial use, particle size variations exist in the -325 mesh portion. As a result before production starts a special fuel plate run is made - usually one lot of inner plates and one lot of outer plates, for a total of 48 fuel plates. This is done to determine the pressing pressure required to meet the desired fuel compact gage to produce acceptable fuel cores. Pressing pressures have varied from 22 to 30 tsi, dependent upon the characteristics of the lot of aluminum powder used. In no case has variability been found within a given lot. The variations are only between lots, and these powders are bought in as large a lot size as is practical for production (up to 15,000 lb). Thus, this variability has caused no real problem. All aluminum powders are purchased with a certified chemical analysis and particle size distribution provided by the seller. The aluminum powder purchased with greater than 80% -325 U.S. standard mesh will, after vacuum annealing, be between 74 and 80% -325 U.S. standard mesh, and this range is considered acceptable.

Boron Carbide

All boron carbide to date has been purchased from the Norton Company. While this is not the only source of the material, previous experience by Texas Instruments Inc had indicated that the boron carbide supplied by them, while not meeting all specifications for chemical composition and particle size, would be considered acceptable. From time to time the specifications have been changed to accommodate the material; however, this was impractical since variables could not

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Fig. 4. Vacuum Storage of Annealed Aluminum Powder.

be predicted for the succeeding lots. In the case of the boron carbide, as well as the aluminum powder, one must recognize that these materials are not made primarily for the nuclear industry. The material is taken from a standard production run, and considerable variability can result from manufacturing equipment conditions and is permissible in the use for which the material was originally intended. The boron carbide is purchased with a full chemical analysis, and ^{10}B analysis is certified by an independent laboratory. The cost of this analysis is greater than the cost of small lots of material (2-10 lb). To check a particular lot of boron carbide under production conditions before it is committed to general use, one lot of inner fuel plates is manufactured, and total boron homogeneity is determined on one plate.

Aluminum Frame and Cover Plate Material

Since both frames and cover plates are 6061 aluminum alloy clad with 1100 aluminum and problems with both parts are similar, it seems appropriate to deal with them together. At ORNL this material was bought in 3×8 ft sheets in small lots, 1 to 2 tons at a time. For production, the smallest order was for 10 tons and the largest for 100 tons. We cannot see why no manufacturer of this material has been able to produce it in large quantities without substantial difficulty. It appears at this time that alloy 6061 clad with 1100 aluminum will no longer be available, since all three manufacturers have suffered substantial financial losses in trying to manufacture this material in these larger quantities. The major problems are in cladding blisters and surface quality. Meeting the chemical composition for nuclear application has never been a problem. As an alternate to use of the aluminum clad material, a technique for bonding alloy 6061 to itself has been developed.⁷ This technique is in use at ORNL and will be tried on a production basis at Texas Instruments Inc. There is evidence that

⁷J. H. Erwin, Metals and Ceramics Div. Ann. Progr. Rept. June 30, 1969, ORNL-4470, p. 122.

the quality of the finished fuel plate made from unclad alloy 6061 will be as good as that of plates presently manufactured from the alclad material.

Fabrication

Frame and Cover Plates

The 3 × 8 ft sheets of frame and cover plate stock are sheared into 6-in.-wide × 4-ft-long strips with the 4-ft dimension in the rolling direction. The strips are cold rolled to the desired gages, since the as purchased thickness variability is much too great for use in fabricating fuel plates. Commercial tolerance is 0.019 in. total for frame material and 0.006 in. total for cover plate material. Finish frame and cover plate tolerance is ±0.001 in.

An example of the effect of the variability of the sum of the thickness of two covers and one frame on fuel length is as follows:

1. assume a deviation of 0.004 in. from a nominal 0.482 in.,
2. final fuel plate gage 0.0505 in. or a total reduction ratio of 9.546, and
3. final nominal fuel length of 20.00 in.

$$\frac{0.004}{9.546} \times \frac{20.00}{0.0505} = 0.166 \text{ in. change in the fuel length due to a}$$

0.004 in. total change in thickness of two covers and one frame.

At ORNL the frames were blanked in a die to produce two cavities approximately 0.015 in. undersize; they were then blanked in a second die to remove the remaining 0.015 in. The frame sides were reversed during these operations to minimize the rolled edge, which normally occurs during blanking. At Texas Instruments Inc (TI) the preliminary blanking was done the same as at ORNL. However, TI thought that broaching would be better for final sizing, since more than one frame could be broached at a time and a better finish to the cavity would result. After several tries on the horizontal broaching machine at TI, it became apparent that the frames could not be held so that uniform cavities could be produced. Therefore, a die was made for the high-speed press to remove 0.015 in. from each face of the cavities and also

to trim the leading and trailing edge of the frame within 0.005-in. TIR square with the reference edge. The cavities are also maintained square with the reference edge. In this way, both ends, one edge, and both cavities are produced on one tool and have good dimensional control in reference to each other. Because of the tolerance in the width of the as sheared strip, the other edge may be no more than 0.007 in. out of parallel with the reference edge. This technique produces a frame that minimizes fuel core boundary problems in the as-rolled fuel plates.

Frame cavities were originally manufactured to produce a 0.003 to 0.008 in. shrink fit at room temperature. It was necessary to heat the frame to 500°C to insert the core. At present the frame cavities are 0.003 to 0.005 in. oversize, and the frame heating is unnecessary. This change widened the finished fuel by 0.003 to 0.005 in. on the average and up to 0.008 in. in the narrowest location. This was desirable because the finished cores were on the narrow side of the allowed range. There was no measurable effect on the fuel length or rate of blister formation.

The cover plates were originally sheared oversize in length, and one end was resheared for squareness. Instead, now a cover plate is blanked directly from the strip, cutting both ends at the same time with one die.

Both cover plates and frames were scratch brushed on the mating surfaces for rolling at ORNL. This process was adopted at Texas Instruments Inc and used until recently. It has now been replaced by an ORNL-developed etching process using Oakite 160 (ref. 8) for preparing bare alloy 6061 for bonding. The etching process has proven to be very successful at Texas Instruments Inc. This change removed from the process a step that was both dirty and difficult. Since the frames and covers had to be cleaned anyway, the small addition to the pickle room was insignificant compared with the time required for scratch brushing, and an added bonus was that now the frame cavities could also be cleaned efficiently. The cleanliness of solutions is controlled by frequent

⁸A product of Oakite Products, Inc., General Offices: 50 Valley Road, Berkeley Heights, New Jersey 07922.

changes. The Oakite solution is changed every 108 pieces; the HNO_3 solution after 216 pieces and the alcohol after 360 pieces. These numbers were developed by production experience. The Oakite process has been previously documented,⁷ but a word of caution is in order. During the use of this process, fuel plate blisters have occasionally occurred. These blisters are now explainable. Organic contaminants in commercial nitric acid leave on the surface of the plate a film that is undetectable until it is heated; then it appears from yellow to brown on the 1100 aluminum side and a dull gray on the alloy 6061 side. Because of this contamination, all frames and covers are now desmuted with reagent-grade nitric acid.

So that processing characteristics are consistent, all covers and frames are orientated so that the direction during hot and cold rolling phases of fuel plate fabrication is the same as the direction of rolling during fabrication of original sheet or plate. Completed frames and covers for both inner and outer fuel plates have the same respective thicknesses, so identical mill settings and reductions are used during fuel plate fabrication.

Weighing and Blending

As was previously stated, the aluminum powder was stored in vacuum containers after annealing. In preparation for weighing and blending, slightly more aluminum powder is taken from vacuum storage than would be weighed out in 4 hr. The powders are screened through three (needed to meet screening stack height) U.S. standard 100-mesh sieves for 2 min. Screening for longer times passed foreign particles, such as eye lashes, lint, and dandruff, which fit the 100 mesh size in two dimensions but are substantially large in the other dimension. All powders except B_4C are weighed on the same balance in a dry box approved for uranium. Separate weighing cups are used for aluminum and U_3O_8 , and before weighing starts all are precoated on the interior surfaces with the material to be weighed. The weighing cups are not brushed or cleaned between weighings, so a constant residue weight is provided. The balance with cup is adjusted to zero. All 24 uranium charges are weighed first, and the weight of every charge is double-checked by

adding a weight to the balance pan to bring the dial to the next higher multiple of 10 g. This means that if a charge of 22.312 g of U_3O_8 is weighed, a 7.688-g weight is placed beside the weighing cup on the balance pan, and the balance should read 30 g even. Since six different weighings are made on this balance, six check weights are provided, each having a different shape for quick identification. The aluminum charges are weighed in the same manner. At ORNL all weighing was done on an analytical balance, which was more time-consuming but more accurate. Since this high degree of accuracy was not required, the more convenient but less accurate top-loading balance could be used. All charges are weighed in the weighing room, in which relative humidity is controlled at 30 to 50% and temperature at 70 to 75°F to control the moisture content of the charge. Next, the inner annulus filler section charge jars are transferred to the chemical laboratory, and the B_4C is added. This is somewhat cumbersome, but necessary, since the accuracy of the B_4C weighing is much more stringent than that of the aluminum powder and U_3O_8 .

The powders are blended at Texas Instruments Inc the same as at ORNL. Several other techniques were tried during development, such as "V" blenders and in-line blenders; however, the oblique blender, formerly sold by the Chemical Rubber Company, is still the best blender available for this type of work. Figure 5 shows this blender loaded.

The need for the next step is not well founded. All charge jars are hand-tumbled obliquely end for end several times just before the charge is added to the die. Although we do not know whether this serves a useful purpose, the fact that satisfactory cores are obtained makes us reluctant to try eliminating this relatively simple step. This hand-tumbling is also done at ORNL on every charge poured into a die, regardless of the contents of the jar.

Charge jars have been of special importance. The original jars were plastic with steel caps and a vinyl-coated paper gasket. Since the abrasive action of the powders was scouring the interior of these jars, a change was made to glass jars. Except for infrequent chipping of the top edge, glass jars have been highly successful. Jar caps, on

Fig. 5. Blending of Fuel Charges.

the other hand, required a much longer development to obtain satisfactory performance. The protective paint or lacquer abraded, particularly in the threads, and pieces of the coating were found in the blended charges. Steel caps were replaced with Bakelite caps, but these proved to be unsatisfactory. The threads were damaged by abrasion, especially in some caps that had fragile porous areas on the threads. Chromium-plated steel caps proved very adequate. The plating adheres well, protects the steel from rusting, and resists abrasion well in the threaded area.

The vinyl-coated paper gasket of the original metal caps was very unsatisfactory. Pieces of both new and used gaskets were found in blended charges. This gasket was replaced by an aluminum disk backed by a neoprene disk. The combination of glass jars, plated jar caps, and aluminum and neoprene disks gave the least contamination by foreign material. No way has been found to clean charge jars, caps, or gaskets without leaving some residue or piece of cleaning material. As a consequence, all new charge jars, caps, and gaskets are cleaned as well as possible with lint-free cloths; then the jar is filled with a charge of aluminum powder, capped, and blended. The emptying of this charge

seems to remove most of the extraneous material and leaves the jars coated for subsequent use.

Compacting

The HFIR fuel compact is duplex, having a fuel section and a filler section. The filler section is 101 aluminum in the outer plates, and 101 aluminum with added boron carbide in the inner plates. The fuel section in the finished outer plate is graded across the compact (see Fig. 6), varying from 0.0095 in. at the thin edge to a maximum of 0.027 in. at the hump or highest point and back to 0.0183 in. on the other edge in the rolled fuel core. The finished core thickness is approximately 0.030 in. The pressed and degassed compact is 0.267 in. thick, and the "as-contoured" powders are 0.750 in. thick.

To achieve the contour, dies with the desired rectangular cavity were manufactured. Die tops with a calculated contour were made with the same die cavity dimensions and are aligned by pins to the die and to each other. Since both inner and outer compacts are duplex and the same problems exist with both, we shall treat them as a single task.

At ORNL the dies were mounted on a die set with a jack to raise and lower the bottom punch as needed. The general procedure is to assemble the fuel section die top onto the die and raise the bottom punch until the leveled fuel charge just fills the cavity. A stainless steel straight edge is then used to level the powders to the proper contour, and, by raising or lowering the jack, the powders are contoured just filling the die top cavity. The filler portion die top is then added. Extreme care is needed to avoid disturbing the loose fuel powders already contoured. This operation has always required great dexterity on the part of the operator and has been the limiting factor in choosing personnel for this job.

At Texas Instruments Inc several different approaches to contouring powders have been tried. One was inverting the die tops and loading the filler portion first. Another was using only the fuel die top, lowering the fuel into the die, and adding the filler portion. All these methods failed, and the ORNL technique was adopted with several refinements added to assure a more consistent product. It is

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Fig. 6. Configuration of Fuel Cores. (a) Outer element. (b) Inner element.

extremely important for consistency that all operators use as nearly as possible the same technique in loading the die. One method to achieve this is to make dummy compacts that can be separated at the fuel-filler interface for the operator to examine for faults. Although this may sound difficult, the method of parting the sections is simple. This goal took months to achieve, and the solution was discovered quite by accident. The operators used talcum powder on their hands before putting on rubber gloves. They found that if talcum powder was lightly sprinkled over the fuel section before the filler section was added, after pressing, the dummy compact would separate quite readily and show the fuel-filler interface. With this technique, the compactor could experiment with different loading techniques to achieve a smooth continuous interface between fuel and filler.

During the actual operation of compacting, the rollup of powders at the core die interfaces was found to vary. This was attributed to two causes. First, the die lubricant should be restricted to the area of the die cavity that will be filled after pressing; the more area covered, the more rollup of fuel will occur. Second, the U_3O_8 is an excellent abrasive, and scratches on the die cavity surface only 0.0002 to 0.0004 in. deep can cause severe rollup of fuel. This problem has been nearly eliminated by routinely stoning the die cavities with a very fine Arkansas stone at regularly scheduled intervals. The dies are then resized by coating the cavity with the stearic acid- CCl_4 solution and baking at a low temperature, much as one would size a cast-iron frying pan. After sizing, the dies and die tops are thoroughly cleaned with isopropyl alcohol and are ready for use.

The art of contouring has been well refined, but even three or four years experience does not prevent occasional lapses in technique. The core hump can be moved as much as 1/8 in. by changing the leveling technique and the powders can be packed into corners. Both these lapses can cause homogeneity rejections. In our present method, the fuel powder blends are poured into the die-top cavity at the hump. With the straight edge of the leveling tool perpendicular to the die face, not the die-top face, the powders are first pushed away from the operator. The powders resting on the die top are then gently pulled

over the cavity toward the operator to fill the front of the cavity. Precompacting against either the reference or opposite edge (front or back of the die cavity) results from powders tumbling a slope or from being pushed rapidly. The answer has been to concentrate the fuel charge at the hump area as much as possible during filling and then to slowly and gently fill first the back edge and then the front edge. At this point, the bottom punch of the die may be adjusted slightly in order to finish filling the die top. The corners usually are the last areas to be filled. It is extremely important that the leveling tool be kept perpendicular to the top of the die, not the contour of the die top, since the edges of the die are very susceptible to fuel packing.

The second die top is added at this point, and the fuel section is raised slightly; this causes the edges of the fuel to be rolled downward, reducing rollup of fuel during the final operations. The filler is poured out on the die top a small amount at a time and gently pushed into the cavity a little at a time to keep from disturbing the already contoured fuel portion. Both edges of the compact are treated alike from this respect. Once sufficient filler is added to the cavity in this manner, the remainder of the filler material is poured out on the die top with approximately equal portions at both edges. If the filler is poured from the charge jar directly onto the leveled fuel charge, a splash will occur at the interface of the fuel and filler charge and persist in the finished compact. This is sometimes noticed in radiographs of the compacts and frequently during x-ray homogeneity scanning. Figure 7 shows this in an experimental compact separated at the interface line. The leveling tool is used very gently to move the filler over the fuel until all the fuel is covered. After the fuel is completely covered, the leveling can be completed with little difficulty, since the filler powder holds the fuel powder very well. The leveled compact then is lowered into the die cavity in one continuous stroke. The jack must not strike the die base or the powders will be disturbed as the higher density fuel charge slumps into the low-fuel-content areas. Once the powders are lowered in place, a rolled edge approximately 0.031 in. high will appear around all the cavity edges.

Fig. 7. Split Compacts Illustrating Interface Between Fuel and Filler Sections. (a) Acceptable interface. (b) Results of upset or splash during addition of filler charge.

This roll should be uniform, with no powders sticking to the die or die top corners. If a proper drop has been achieved, a good compact will result. The top punch is inserted and gently pushed by hand until it contacts the powders; then the press is activated to do the final pressing. Pressing pressures from 22 to 30 tsi have been used, depending upon the characteristics of the particular lot of aluminum powder being used.

At Texas Instruments Inc pressing with a punch having parallel top and bottom faces produced compacts with nonuniform thickness, because the fuel portion of the compact is asymmetric. The bottom face of the punch was tapered, giving a greater pressing effort in the area of higher U_3O_8 concentration. A taper of 0.007 in. was necessary for outer compacts and 0.005 in. for inner compacts.

Rolling

Originally, assemblies were preheated to 500°C for 1 hr before the first pass and reheated 10 min between passes until the third pass; then the reheat time was reduced to 5 min. A circulating hot air furnace with a high recovery rate is used. Once after the furnace had been repaired, the recovery rate was so fast that a magnesium-aluminum-silicon eutectic was formed at the 6061 aluminum alloy grain boundaries, causing small surface blisters and increasing the overall blister rate by approximately 15%. When the cause of the blisters was identified, all furnaces were reset to 485°C, which is just below the eutectic temperature but still high enough to achieve good grain growth. Once the temperature was changed, the blister rate went back to a normal 5%. More recently the scratch brushing has been eliminated achieving a cleaner operation and reducing the blister rate to 2.5% or less.

During preproduction the hot roll bonding operation was found to substantially affect the quality of fuel plates. The first problem considered was damage to the surface of plates. Because of extreme pickup problems, we decided to use no feed or runout table at the mill. Billets at the hot mill are guided by a series of vertical rollers engaging the bonding assembling edges without any face contact. Figure 8 shows this guide.

Fig. 8. Entry Side of Hot Rolling Mill, Showing Plate Being Fed Between Roll Guides.

The bonding assemblies and partially bonded plates were carried with conventional 18-in. steel tongs with added stainless steel hammer-head jaws and a guard that limited the holding area to the extreme ends of the assembly, which are later discarded.

The furnace rack was given a great deal of consideration. Bonding assemblies laid flat on the hearth of the oven picked up a great deal of contamination, so this practice was abandoned. When stacked on themselves or any other material, bonding assemblies tend to slide slightly, causing pickup or torn metal on the contact faces. This

produces inclusions and other types of surface defects that subsequently reject plates. The solution was a 24-station rack in two tiers. The rack supported the bonding assemblies diagonally with tapered pins, so that only corners contacted the rack. Even with this minimum contact, aluminum accumulates on the rack and must be periodically removed by etching in caustic. The next problem at the hot roll operation was the aluminum-faced asbestos gloves used by the rolling mill operator. These gloves both were short lived and seriously contaminated the bonding assemblies with asbestos fibers. The gloves were replaced with insulated leather welders' gloves.

The catching phase of the hot mill operation proved more difficult in preventing handling damage. Both tongs and vise grip pliers proved quite impractical. With these holding devices it was not possible to turn the assemblies to the proper orientation for the next rolling pass. In addition, they cut badly into the hot assemblies and made loose or semiattached chips. The use of asbestos gloves was also extremely impractical because they shed asbestos over the assemblies. Chain-mesh gloves worn over cotton canvas gloves proved to be a very workable solution. With these gloves, the catcher could grasp the assemblies as they emerged from the rolls, turn them to the desired orientation, and hand them back over the rolls to tongs of the rolling mill operator. All these steps may seem excessive, but it should be remembered that no other operation has as much opportunity to produce surface damage and inclusions in fuel plates as the hot roll bonding operation.

Another phase of the effect of the hot roll bonding operation on the quality of finished fuel plates is the fuel end configuration. The product requirement is for each end of the fuel of a finished plate to lay within a given half inch. With variation from other sources requiring a portion of this tolerance, only a limited part could accommodate out-of-square ends. The roller guides previously mentioned along with highly trained operators have been able to control fuel end configuration to acceptable deviations from square. A roll guide for one side of the bonding assembly has been found to be entirely adequate. The second guide needed constant adjustment to allow for the spreading of the bonding assemblies as the rolling sequence progressed. A special

square referencing the two rolls is used to align the working guide. Analysis shows that, if an assembly is entered into the rolls at an angle during the initial passes, out-of-square ends are developed. This condition cannot be corrected during subsequent passes but becomes increasingly larger. The average out-of-squareness has been limited to 0.10 to 0.15 in. When a plate entered the mill $1/4$ in. out of square for the first bonding pass, the core deviated from square by 0.0118 in. at the leading end and 0.0135 in. at the trailing end. On completion of the rolling operations these deviations would increase to 0.096 and 0.110 in., respectively.

A problem at the hot mill was the rolling of flat assemblies, particularly during the initial passes. Despite lubrication with oil and kerosene spread by felt wipers, many assemblies took the shape of the rolls and were difficult to catch on the exit side, to orient, and place in the reheating oven, and, particularly, to feed into the mill for the next pass. The solution was to construct a set of wiper brackets sufficiently strong to act as a brake on the rolls during the idling period and prevent a pause, particularly of the top roll, during entry of the assemblies. When the top roll paused, the bottom roll curved the assembly around the top roll. It was found desirable to use high grade wool felt as the pads (SAE specification F-10). The felt, by its good wicking and even density, provides both constant lubrication and constant drag on the rolls. Mineral spirits replaced kerosene, because kerosene was found to hold foreign material.

When aluminum is hot rolled, the roll surface becomes coated with a mixture of aluminum and oxide. This results in smoothing of the roll surface during the roll bonding of fuel plates. When the finish of the surface reaches 5 to 10 microinches rms the rolls will not accept the fuel plate billets, particularly during the initial passes. Before this stage is reached, the rolls will start skidding and then grab a billet by the corner, pulling it through the mill diagonally and producing a trapezoidal core. After the rolls have been ground to a finish of 10 to 15 microinches rms, the mill can bond about 4000 plates before rejection or slipping occurs, if the billets have been assembled

with cover plates prepared by scratch brushing the mating surface, leaving the oxide film on the outer surface. However, fewer than 1000 can be bonded when the covers are prepared by etching. A surface finish of 30 to 40 microinches rms extends the roll life to about 2000 plates with etched covers. An interesting phenomenon is that slipping or refusal occurs on the dummy assemblies used to heat and condition the rolls before it occurs on fuel plates; this warns of the need for roll refinishing without sacrificing fuel cores.

The two plates made from each bonding assembly after the seventh pass of the hot roll operation were originally cut apart with a power shear located away from the rolling mill. This caused delays for cooling, handling, and reheating. A three-blade one-handle hand-operated shear was fastened to a bench in a convenient location so that the assemblies could be simultaneously trimmed and sheared in two and then replaced in the reheating oven without the delay and extra handling.

The fuel plate core has, until recently, always had a slight taper from end to end. The numbered end, which enters the rolls on the first pass, was always approximately 0.008 in. wider than the trailing end in the finished plate. On occasions this taper caused rejections. (The powder dies had been designed to produce a core slightly under the nominal fuel width to provide metal for reworking the die cavity to remove scratches resulting from the abrasive U_3O_8 particles.) Plates rejected because of fuel width and length are submitted to the ORNL Operations Division for final acceptance or rejection, and in most cases they are acceptable. Several times experimental changes were made in the hot bonding schedule to correct the taper, with no real success. As was previously stated, the problem was solved when the frame cavity size was changed to accommodate a loose fitting core rather than a shrink fit.

The rolling schedule at ORNL was different for the inner and outer plates at Texas Instruments Inc. The core and frame were so designed that the same rolling schedule could be used for both types of plates. This design has the further benefit that rolling to a predetermined gage that is common for all plates produces a core outline that is within specification. This eliminated the need for fluoroscoping the

fuel plate before final cold rolling, thus saving a complicated and time-consuming step in the operation. Also each mill setting is predetermined and strictly adhered to by the operators. Further, the camber of the core was proportional to the camber of the fuel plate, so a camber gage was installed at the cold mill. Before the final four passes through the mill the camber is measured, and the two bearings of the rolls are adjusted accordingly to reduce the camber to an acceptable level. The specifications call for a fuel plate thickness of 0.050 ± 0.001 in., but to obtain reproducibility in forming, the mill operator is given a tolerance of 0.0005 in. This tolerance may seem abnormally tight and difficult to achieve; however, the mill operator can achieve it with excellent reproducibility within his measuring capability.

Annealing

The annealing procedure has not been changed from the original ORNL process, except for the reduction in temperature from 500 to 485°C. At Texas Instruments Inc the annealing is done strictly on a time basis. Engineering development established the time to reach temperature, the time at temperature, and the cooling rate, so during the anneal after hot rolling the operator is concerned only with time. The final anneal is controlled by a cam timer, so all the operator has to do is insert the plates and set the timer. All furnaces have override alarms to alert the operator to occasional malfunctions. Ovens used for production are tested regularly to assure that temperature specifications are being met.

Fuel Plate Sizing

The fuel plate remains in its as-rolled and annealed condition through fluoroscope punching. At ORNL the plates were scribed at the fluoroscope and rough sheared to length and width. Sufficient end cladding was left to allow the plates to be drilled for mounting, and the plates were machined to size in lots of approximately 30. At Texas Instruments, Inc., the holes are punched at the fluoroscope operation.

A 1/2-in.-diam hole, a 1/2 × 3/4-in. slot and two 1/8-in.-diam holes are punched. The 1/2-in.-diam hole and the slot are matched to a blanking die, which finish sizes a plate in a single operation. The down side of the plate is deburred to remove the rolled edge and the plate is ready for forming. The sizing operation is typically geared for production, and the die yields plates with consistent lengths and widths to extreme accuracy. A very important point that should be stressed here is that the punches at the fluoroscope must relate with the pins on the blanking die within ±0.002 in. to assure that the core is properly located in the finished fuel plate.

In no case after cold rolling and annealing is a fuel plate ever laid on a bare table. All table tops are covered with rubber floor mats that have a dimpled surface to allow any chips or foreign matter to fall away from the plate and prevent scratching. Figure 9 shows a fuel plate being deburred on a rubber mat.

Forming

The fuel plates are formed in essentially the same manner as at ORNL. The die design is the same, and a modified marforming process is used. The die base contained a 2-in.-thick × 26-in.-long × 6-in.-wide piece of 97-durometer polyurethane elastomer with a 26-in.-long × 6-in.-wide × 1/4-in.-thick overlay of the same material. ORNL used a special rubber in the die, and the 1/4-in.-thick piece was used for 19 plates on each side and replaced. With the polyurethane a life expectancy of the 2-in. pad has not been established after 65,000 fuel plates. The 1/4-in.-thick polyurethane pad has been replaced from time to time for cutting that occurs at the ends and edges of the fuel plate and possible damage to a plate by a loose piece from the cut area. Acceptable forming results are always achieved.

The fuel plate holder was also similar to the ORNL holder and was adjusted from the front face of the die. During early stages of production, it was adjusted often on a lot-to-lot basis. However, after production was established, the plate holder position was seldom changed. On one occasion one of the hot rolling operators changed his method of adjusting the rolls on the seventh pass, which is the last

Fig. 9. Deburring Fuel Plate Edges, Showing Use of Rubber Mat.

pass before the duplex frame is sheared in half. This minor adjustment caused these plates to form slightly different than those produced by the other rolling operators. This problem was minor, and no great effort was made to solve it. However, engineers and operators were aware of the problem, and after almost two years the cause of the difference was located and corrected. What made the problem so hard to isolate was the fact that the fuel plate reduction difference was only about 0.003 in., and the fuel plate memory retained the difference through five succeeding hot passes, an anneal at 500°C, then 20% cold

work with a 500°C long-term anneal with slow cooling to the dead soft condition. Metallurgically it doesn't seem possible, but it happened.

Pickling

The fuel plates at ORNL and Texas Instruments Inc are pickled with a solution containing hydrofluoric and nitric acids. Controls were maintained at ORNL, but not rigidly as at Texas Instruments Inc. At Texas Instruments Inc, a maximum of 0.0003 in. is removed from each side of the fuel plate. Control is maintained in accordance with a standard operation, and a final check is made by measuring thickness before and after pickling. The measurement is made on both the fuel plate and a coupon taken from an end trimming of a fuel plate, which represents the same aluminum treated in the same way. A visual inspection by the operator is also made, and if any defect is found inspection is notified and an in-process rejection is written and proper disposition is made.

Inspection

Texas Instruments Inc inspects HFIR fuel plates according to ORNL specifications. At ORNL the inspections were done by several groups and divisions, dependent upon their capabilities and equipment. At Texas Instruments Inc, inspection and quality assurance is a separate organization reporting to top management, and operators are prohibited from inspecting any portion of their work.

During the normal flow of manufacture, each operator checks and cleans each fuel plate before and after each operation. If the operator observes a defect in a fuel plate, he notifies inspection, and the suspect plate is inspected at once. If the fuel plate is acceptable, the process is continued; if it is rejected, an in-process rejection is written and is disposed of by engineering. When engineering designates the rejected part as scrap, it is removed from the lot and the rest of the lot is processed. This in-process rejection capability serves a double purpose: (1) it may indicate a deficiency in an operation, and (2) it saves time for the inspection department; otherwise it would

have to hunt for and find a defect without the knowledge that the operator has concerning the defect.

Control Charts

The Quality Assurance Group keeps constant records on charts and graphs that define nominal 2σ and 3σ limits for the attributes being checked. Some of the attributes for which control charts are maintained are cladding thickness, boron homogeneity, bond integrity, and fuel width. By keeping these charts and graphs updated as closely as production will permit, any change that may occur in a given attribute can be followed very closely, and the operation can be stopped before it gets out of control. On several occasions boron content has shown a trend toward the lower 2σ limit. This indicates a need to change reagents in the chemistry laboratory. In no case has boron homogeneity been a problem, but the chemical reagent purity has. The trend is gradual and easily detectable with the control charts. When the charts indicate a trend toward narrow fuel cores the process is checked. Generally this is caused by a temperature control problem, indicating a need to overhaul the hot bonding furnace. These control charts have been most effective. Although there is no way to assess their financial saving, it does exist and is substantial.

A quality performance report is prepared by Texas Instruments Inc semiannually to keep ORNL informed on all phases of production and also provide information on material needs for future production.

Dimensional Inspection

At ORNL a highly sophisticated inspection routine was not needed because of the small number of fuel plates involved. At Texas Instruments Inc inspection is mechanized wherever possible. Frames and cover plates are inspected dimensionally with gages having dial indicators with go no-go limits. Fuel plate length and width are measured similarly, but the thickness is measured with fixed spindle micrometers, which minimize scratching of the surface and are more accurate than conventional micrometers.

The thickest part of the fuel section (hump) is located with an eddy-current probe in the same way as was done at ORNL. The location of the fuel plate's identification number in relation to the fuel is confirmed for the fluoroscopic inspection that follows. A phase-sensitive instrument has been used in some instances to locate the hump. This was done on inner annulus fuel plates for which the Magnaflux instrument did not provide adequate resolution and sensitivity. The fluoroscope inspection is critical, since it not only positions the core on a go no-go gage template for fuel core outline but also blanks three holes and a slot that locates the fuel plate for blanking and homogeneity inspection. The ability to locate the fuel outline within the limits of the fluoroscope template was originally quite a problem. At that time the template had a continuous strip of platinum wire to indicate the maximum and minimum limits. Cutting this wire into segments substantially improved the locating accuracy. Figure 10 reproduces a radiograph of a template. At present, an experienced operator can center the fuel outline, considering both edges, with an average bias of less than 0.005 in. and a maximum bias of no more than 0.010 in. It is critical that the pins on the fuel plate blanking die and the homogeneity scanner are positioned correctly relative to the punches on the fluoroscope, so that the fuel location can be maintained during blanking and scanning.

After the fuel plates are blanked to size, each plate is given a second fluoroscope inspection to assure that the fuel is properly located within the fuel plate envelope. Suspect plates are sent to x-ray for final disposition, and three plates from each group of seven lots are x-rayed routinely and measured to assure fuel plate quality and keep a constant check on fluoroscope operation bias. When biases in operation are detected the operator is observed during inspection and retrained if necessary.

Fig. 10. Radiographic Reproduction of the Fuel Outline Template for Fluoroscopy.

Homogeneity Inspection

The homogeneity scanners⁹ were designed and built by ORNL. The operating premise is that the amount of x-ray attenuation through a fuel plate is directly related to the amount of uranium in the plate. This relation was confirmed for any given lot of U_3O_8 but did not necessarily hold true from lot to lot, since the x rays are sensitive to U_3O_8 particle size as well as the amount of U_3O_8 . We did not know that the x rays would be sensitive to particle size and shape until after production of fuel plates had started. The aluminum standards used to compare with the quantity of fuel in the plate at times indicated variations in fuel loading up to 5% for any given lot of fuel. An adjustment in the aluminum standards would be time consuming and of questionable value, since each lot of fuel would require a new set of standards. The most expedient way to minimize the problem was to exercise greater control of the manufacture of the U_3O_8 . The U_3O_8 was and is controlled by controlling the surface area of the particles to $0.047 \text{ m}^2/\text{g}$ as closely as possible. This is not an absolute method,

⁹B. E. Foster, S. D. Snyder, and R. W. McClung, Continuous Scanning X-Ray Attenuation Technique for Determining Fuel Inhomogeneities in Dispersion Core Fuel Plates, ORNL-3737 (January 1965).

since it does not control the rate of particle fracture during rolling, and slight changes in fuel manufacture can change the fracture strength of the particles. However, the homogeneity scanner is not used to measure quantity of fuel but to measure how homogeneously the fuel is distributed within the fuel plate. Fuel plates that exceed the homogeneity limits are rejected by Texas Instruments Inc and submitted on waiver to ORNL Operations Division for disposition. Information submitted with these waivers includes the fuel loading bias of the lot of fuel being used. This allows ORNL Operations Division engineers to assess the worth of the plate in the reactor. Approximately 60% of the fuel plates submitted are acceptable, and the present rejection rate at Texas Instruments Inc is 1.5%, showing excellent control of homogeneity.

Ultrasonic Inspection

Ultrasonic inspection is done in the same manner as at ORNL. The fuel plate is compared to a fuel plate with built-in known defects that establish defect limits for the fuel plates. During production, when the ultrasonic signal passes from the fuel section to the end cladding, a rejectable indication sometimes occurs. However, if the plate is turned end for end and rerun the rejection indication does not repeat. Fuel plates with this type of indication are rejected by inspection and visually inspected by engineering. If no visible surface defect is found, the fuel plate is acceptable. Since this constituted a deviation from the specifications, an approval request was submitted to ORNL with sufficient information to show that these plates are acceptable, and the process was approved by ORNL. All plates rejected for nonbond by the ultrasonic method are reinspected visually. It is interesting to note that in almost 90% of the cases of ultrasonic rejection a surface blister is found. This should not be construed as a reflection on the quality of surface inspection given the fuel plates before ultrasonic inspection, since these blisters are so small that they cannot be observed unless the ultrasonic charts are measured and the measurements transferred to the fuel plate to locate the exact spot of the rejection.

Alpha Count

Three fuel plates from each lot are alpha counted to determine the presence of uranium on the outside. If a rejection occurs, the lot is cleaned with nitric acid, and the previously rejected plate(s) and additional plates are counted. Generally only one cleaning is necessary to bring the fuel plates in tolerance, and rejections due to alpha contamination are rare.

Destructive Examination

One plate per fuel element is examined destructively. The fuel plate may be a reject if the rejection does not interfere with the destructive examination. The process used for measuring boron content is the same as ORNL used except that control charts are maintained to constantly monitor the boron content of the punchings. If a drift in boron content is detected, the problem has always been associated with the materials used in the analysis rather than in the fuel plates. No fuel plate has even been found to be out of specification due to boron distribution. When an analysis falls within the 2 to 3 σ limit, new punchings are taken from the same plate and a reanalysis is done to ensure boron distribution.

The fuel plate sections are mounted in epoxy and fine polished. These mounts are measured for cladding thickness top and bottom, edge cladding width, fuel thickness, and grain growth across the bond interface. The edge cladding width and fuel thickness are not required measurements but are used on control charts to assess the quality of work done at the fluoroscope and compacting operations. Cladding thickness and bond quality are determined the same as was done at ORNL.

Involute Inspection

The involute inspection device is the same one used at ORNL. Originally the outer fuel plates were measured at three positions radially at five equally spaced locations along the fuel plate, and the inner fuel plates at four positions radially at five equally spaced locations along the plate. Presently the radial locations remain

the same, but only three locations along the plate are inspected, since we have found that these three locations are sufficient to establish the quality of the involute form. Figure 11 shows the gage being used to inspect an inner fuel plate.

Fig. 11. Inspection of Inner Fuel Plate Involute Curvature.

DISCUSSION

The conversion of fuel plate fabrication from a laboratory scale to a manufacturing facility incorporates a great deal of effort in tooling, formulation of standard operations, drawings, and route cards. It requires tremendous effort on the part of the engineers to educate the operators to a precise method in every operation to assure uniform quality. In many cases during the early stages of manufacture, several different approaches to an operation were tried, requiring retraining of the operators, which was generally more difficult because of the prior training. Laboratory-scale operations are handled on a more or less informal basis, but manufacturing production requires that schedules be met precisely, so that each individual operation of production matches with other operations, resulting in a continuous flow of materials. Scheduling during the early development was extremely difficult, since a permanent operator staff had not been assigned, and schedules had to mesh with other product manufacture. The assigning of available operators rather than the same people all the time provided a means of screening personnel, and as the work load increased, permanent assignment was given to the people best suited to the job.

Quality standards must be continually reviewed to assess losses and find ways to reduce them as well as project future material needs. The quality control charts and graphs are continually monitored to assess future raw material needs, material quality, equipment condition, and operator capability. Employee personal problems have been indicated by changes in fuel plate yield or even more subtle changes such as a change in operator bias at the fluoroscope or a shift in the fuel hump at homogeneity scanning.

Tooling is of prime importance in production. The use of operators rather than technicians requires that jigs, fixtures, and dies be extremely accurate and easy to operate, since the operator is more accustomed to repetitive tasks. The operator's prime objective is to manufacture products of the highest quality possible. He must be constantly aware of what he is doing and be on watch for any unusual

occurrence or result and must report them to his supervisor. One tries to arrange tooling and training to minimize the operator's difficulty.

From the time the contract for HFIR was signed by Texas Instruments Inc until the first finished fuel element was delivered, two years had elapsed. During that time the major portion of the tooling, process training, and procedure writing was completed. However, changes were made continually to upgrade the process to improve product quality, save money, or make a job easier. In any case, it is anticipated that these changes, generally minor, will continue as long as HFIR fuel elements are manufactured.

ACKNOWLEDGMENTS

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APPENDIX

Specifications for U_3O_8

September 1, 1970
Sht. 1 Next Sht. 2

DESCRIPTION OF MATERIAL:

Enriched U_3O_8 Powder

PROPERTIES:

1. U_3O_8 powder shall be high-fired dead burned oxide.
2. The density shall be greater than 8.2 g/cc as determined by a toluene pycnometer.
3. The surface area shall be less than $0.07 \text{ m}^2/\text{g}$ as determined by the static Krypton BET determination.
 - a. The technique for making these measurements is contained in the following article by J. L. Bolts, "Surface Area of Solids, Krypton Absorption Method", Method No. 1105 and 900605 (12-28-61) ORNL Master Analytical Manual, TID-7015, Suppl. 5 (Sept. 1963).
 - b. This document may be obtained from the Clearinghouse for Federal Scientific and Technical Information, 5285 Port Royal Road, Springfield, Virginia 22151.
4. The mesh size shall be -170 +325 U.S. Standard mesh and shall not contain more than 10% of -325 U.S. Standard mesh particles as determined by screening of a 100g sample for one hour in a vibratory shaker.
5. The total uranium concentration shall not be less than 84.5 wt. %.
6. The impurities in the U_3O_8 shall not exceed the limits specified in Table I.

TABLE I

Maximum Impurity Level for U_3O_8 (ppm)

Al	100	Co	3	Mg	50
B	0.2	Cr	15	Mn	5
Ba	10	Cu	20	Na	5
Be	0.2	Fe	100	Ni	20
Ca	50	K	20	P	<100
Cd	0.5	F	10	Si	50
		Li	5		2

DWG. OR SPEC. NO. 1-7162		REV. 1
DISTRIBUTION:		TOTAL
E.N. NO.	REV.	DESCRIPTION
67-77	G	Revised
70-23	H	
DRAWN W. Cronin		SCALE none
MFG. APP.		ENGR. APP. F. Sherman 9-1-70
MATERIAL: noted		
CUST. PT. NO.	PROJECT NO. OR-60	
M & C NUCLEAR PRODUCTS P. O. Box 898 Andover, Mass.		
TITLE: ENRICHED U_3O_8 POWDER		

Specifications for U_3O_8

September 1, 1970
Sht. 2 Next Sht. 3

7. A chemical and spectrographic analysis shall be required for each batch of U_3O_8 .
- * 8. The UO_2 content shall be less than 1.0%. The methods of analysis shall be submitted to Texas Instruments Inc. in writing and approved by Texas Instruments Inc. before the analysis for UO_2 is made. (Texas Instruments Inc. must submit the methods of analysis to Union Carbide Nuclear Division in writing and approval received before approval is given the vendor per above).
9. The U_3O_8 shall be cross blended into as large a batch as possible, a minimum batch shall be 100 kg.
10. As furnished to the vendor, the uranium shall be in the form of UF_6 and be enriched to a minimum of 93% of U^{235} . No degradation (within 0.2%) of the material shall be permitted during conversion.
- * 11. All U_3O_8 shall be produced by the precipitation of a uranyl nitrate solution with hydrogen peroxide. Use of any other process must be approved in writing by Texas Instruments Inc. (Texas Instruments Inc. must obtain approval from Union Carbide Nuclear Division before permitting the use of any other process). This approval will require the submission of photomicrographs showing that during rolling, stringing and fragmentation of the oxide are comparable to what has been achieved with the above process.
- * 12. A mass spectrographic analysis for U^{235} enrichment shall be performed by a Texas Instruments Inc. approved facility.
13. Ten certified copies of the chemical and mass spectrographic analyses of U_3O_8 , density, surface area and size distribution of U_3O_8 shall be furnished along with a certification that they comply with the requirements of this specification.
14. No foreign material is to be present in any lot of U_3O_8 .

* CHANGE

DWG. OR SPEC. NO.		1-7162		REV	
DISTRIBUTION:				TOTAL	
E.N. NO.	REV	DESCRIPTION	BY	DATE	
67					
77	G	Revised	FS	11/17	
70-23	H		WC	9/1	
DRAWN		SCALE		DWG. OR SPEC. NO. 1-7162 H	
W. Cronin		none			
MFG. APP.		ENGR. APP. F. Sherman 9-1-70			
MATERIAL:		noted			
CUST. PT. NO.		PROJECT NO.			
		OR-60			
<p>M & C NUCLEAR PRODUCTS P. O. Box 878 Attleboro, Mass.</p>					
TITLE:					
ENRICHED U_3O_8 POWDER					

September 1, 1970
Sht. 3 Next Sht. None

15. A sample of oxide is to be mounted and polished metallographically. In a count of one hundred separate particle cross sections, no more than 25% may show evidence of cracking. Small round voids characteristic of sintering voids are permissible.

* 16. Two samples representative of each lot with a minimum size of 15 gm. shall be furnished the purchaser by the seller for each lot.

PACKING AND SHIPPING:

The U_3O_8 shall be contained in a clean metal can with a full rubber gasket and the volume of the can shall not exceed one liter. The vendor is to limit the amount of U_3O_8 , contained in one liter cans in a given shipping container, to maintain a safe maximum quantity in accordance with Oak Ridge Guide TID-7019 Guide to Shipments of U^{235} Enriched Uranium Material.

The shipping container must fulfill ICC requirements and be marked with a B/E permit number.

* Approval of the proposed shipping container and configuration of fuel must be approved by the U.S. Atomic Energy Commission Division of License and Regulation and Texas Instruments Inc.

This material is to be supplied by a vendor with an approved AEC license.

Each shipping container must be marked with the purchase order number.

Distribution of copies of certification and samples to be per S.O. 54.

* CHANGE

DWG. OR SPEC. NO.		1-7162		REV.	
DISTRIBUTION:				TOTAL	
EN. NO.	REV.	DESCRIPTION		BY	DATE
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W. Cronin		none			
MFG. APP.		ENGR. APP. F. Sherman 9-1-70			
MATERIAL:		noted			
CUST. PT. NO.		PROJECT NO.			
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