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Metallurgy and Ceramics

ARGONNE NATIONAL LABORATORY

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ROLL CLADDING OF URANIUM-NIOBIUM ALLOYS FOR
PLATE TYPE FUEL ELEMENTS

by

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Assigned to Argonne National Laboratory

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ROLL CLADDING OF URANIUM-NIOBIUM ALLOYS FOR PLATE TYPE FUEL ELEMENTS

by

C. H. Bean and R. E. Macherey

I. ABSTRACT

The feasibility of cladding plate type, corrosion resistant uranium-niobium fuel elements with Zircaloy-II by roll bonding has been demonstrated. Plates with cores of uranium alloyed with 3 w/o and 6 w/o Nb intended for irradiation testing in a high temperature water test loop in the MTR have been finished within specified tolerances.

It should be possible to produce finished rolled plates with the following dimensional tolerances:

Over-all width tolerance	- ± 0.005 " in the width range of 1-1/2" to 3"
Over-all thickness tolerance	- ± 0.001 " in the thickness range of 0.062" to 0.250"
Straightness	- Less than 1/8" camber in 2 ft
Cladding thickness	- $\pm 10\%$ on 0.010" to 0.020" thick cladding.

The preparation of cladding billet core and clad components and the assembly of billets by enclosing cores in welded Zircaloy-II jackets can be readily accomplished with conventional fabrication equipment. Some machining operations and all billet evacuations, as used in the preparation of most picture frame billet assemblies, have been eliminated.

Roll bonds were obtained with reductions of 75% to 80% in thickness. Reductions in excess of 90% in thickness, although not necessary for bonding, can be used for economical production of long plates.

Plates can be made with clad to core bond strengths from 30,000 psi to 60,000 psi. Properly heat-treated plates have sufficient ductility to allow cold finishing by rolling, forming, bending, or twisting, with reductions of 20% to 30%.

Edge bonds of Zircaloy to Zircaloy have been obtained which were corrosion resistant to 260°C water. End seals which were also corrosion resistant to water at this temperature have been made by shielded arc welding inserts to the clad.

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The core alloy of clad plates quenched from solution temperature by pressing between water-cooled platens had the same corrosion resistance as cores which were water-quenched from the solution temperature. Plates held in this way were free from warping and distortion after quenching.

The roll cladding procedure developed for uranium-niobium alloys should be applicable to the cladding of other corrosion resistant uranium alloys having similar hot rolling characteristics.

II. INTRODUCTION

Recent improvements in the development of uranium alloys for reactor fuels have indicated the desirability of fabricating these alloys into reactor fuel elements. A plate type element was proposed as being desirable for use in a water-cooled or water-moderated thermal reactor operating at temperatures up to 260°C . These elements must be integrally clad with an inert material to prolong their life and to prevent contamination of the water from fuel fission products. A sound metallurgical bond between the core and clad was requisite to securing maximum heat transfer. Fuel elements were also needed which would be suitable for irradiation tests of these alloys in the MTR water test loop.

Gamma-treated uranium alloys containing either 3 w/o or 6 w/o niobium were reported as having good corrosion resistance to water at $260^{\circ}\text{C}^{(1)*}$ and excellent dimensional stability on thermal cycling. Because of the corrosion resistance and stability of these two alloys, they were selected for the initial alloy cladding program.

Because of its low thermal neutron absorption cross section, good corrosion resistance, and availability in quantity, a zirconium alloy, Zircaloy-II, was selected as a desirable cladding material.

It was intended that all fabrication steps be simplified as much as possible and be adapted to conventional or existing equipment consistent with the production of sound clad plate.

A procedure developed at ANL for roll cladding commercially pure uranium with crystal bar zirconium⁽²⁾ suggested certain operations which could be directly applied to the roll cladding of uranium alloys. Cladding billets consisting of uranium alloy cores jacketed with Zircaloy-II cladding material were to be hot rolled in a protective steel can. Optimum rolling temperature, reduction, speed of rolling, and method of material preparation was to be established for each core alloy.

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Hot-rolled plates would require special finishing operations before they could be used as fuel elements. To obtain maximum dimensional tolerances, cold finishing and plate end closure methods would have to be investigated. To secure maximum corrosion resistance and stability of the alloy core, plates would have to be gamma heat treated following all rolling and welding operations. Surface preparations were to be studied which would result in the most corrosion resistant cladding surface.

Finished clad plates were to be tested and the test results used to evaluate cladding procedures. These tests included measurements of bond strength, completeness of bonding, uniformity of the cladding, distribution of the core material within the clad plate, diffusion between the core and cladding, resistance to failure in a water corrosion test, and amount of deformation which the material could withstand on cold finishing or forming into special shapes.

III. MATERIAL PREPARATION AND EQUIPMENT

A. Melting and Casting of U/Nb Core Alloy

The uranium rich uranium-niobium alloys used for core material in this investigation were made by the solution of niobium metal in biscuit uranium during a melting operation.

Melting and casting was done in a 6" Vycor tube vacuum induction furnace. This furnace had a capacity of approximately 6 kg. The vacuum in this furnace was produced by a 6" fractionating oil diffusion pump backed by a 4" booster oil diffusion pump, Distillation Products MCF-700 and MB-100, respectively. The mechanical pump for this system was a Central Scientific Hypervac-20. A chevron-type, water-cooled baffle was provided at the high vacuum end of the MCF-700 to stop back diffusion of pump oil into the furnace.

Power for induction melting was supplied by a TOCCO motor generator set rated 30 KW, 220 volt, 10,000 cycle output. The capacitor bank for regulation of the power factor was located directly behind the furnace. The arrangement for melting and casting consisted of a high purity MgO crucible, 3-1/4" I.D. x 3-3/4" O.D. x 7-1/2" deep with a tapered bottom, supported on but separated from the water-cooled copper mold by means of a MgO stool. For bottom pouring, the magnesia crucible was bottom drilled and fitted with a tapered nose, high purity magnesia stopper rod, 5/8" diameter. For melting, a tantalum heater was provided which closely surrounded the crucible. The Vycor tube was shielded from the heat by means of a stabilized zirconia insulating tube.

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The charge consisted of niobium, either presintered bar or rolled sheet, and Mallinckrodt biscuit uranium. The niobium was prepared for charging by breaking the presintered bar into 1/4" to 1/2" cubes, or cutting the 30-mil sheet to fit in the crucible. In loading, the niobium was placed on the bottom of the crucible and uranium lumps were placed on top of the niobium. After loading, the furnace was closed and evacuated to an absolute pressure of less than 5×10^{-5} mm Hg. The power was then turned on. After melting the uranium, the charge was heated to between 1550°C and 1750°C and held for a short time (approximately 30 minutes) to permit solution of the niobium. The temperature was then dropped to approximately 1400°C and the melt bottom poured into a water-cooled copper mold, 1-3/4" diameter x 9" deep. A summary of melting and casting data is presented in Table I.

Very good recovery of niobium in the casting was obtained. Very little segregation of the alloying element, either alone or associated with segregation of carbon or nitrogen, was found upon analyses of samples from the top and bottom of the ingots. Typical chemical and spectrographic analyses for these castings may be found in Table II.

B. Fabrication of U/Nb Core Alloy

The 1-3/4" diameter U/Nb alloy castings required fabrication to bar stock of approximately 1/2" x 1-3/4" cross section for machining into core blocks. The castings were fabricated by a combination of forging and rolling operations.

For forging, the castings were preheated 30 - 45 minutes in a helium atmosphere to a temperature of 850°C. From three to four reheats of 10 minutes each were required to maintain the working temperature between 750°C and 850°C. Using reductions of 1/8" to 3/16" per blow, the castings were forged to a cross section of 7/8" x 1-3/4". Both the 3% and 6% Nb alloys were forged in the above temperature range without cracking, checking, or spalling.

Immediately following forging, the bars were preheated to 850°C and rolled to 0.50" or 0.60" thick, depending upon the required finished core size. Reductions of 6% to 8% per pass were made on the 16" mill. To maintain rolling temperature the bar was reheated 5 minutes after every pass. Hot rolled bars were straightened, then cooled in an inert helium atmosphere or by water quenching. Air cooling this material from 850°C resulted in severe oxidation and flaking of the metal surface.

The core material was finished to sizes required for cladding billets by machining and grinding.

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C. Fabrication of Zircaloy-II Cladding Material

All Zircaloy-II cladding material was fabricated from Ingots No. 1X081 and 1X082, melted from consumable electrodes at Allegheny Ludlum Steel Corporation. Grade P zirconium sponge from the U. S. Bureau of Mines was mechanically mixed with the alloying elements, compacted into electrodes, and melted in a 9" diameter crucible. The 9" diameter ingots were then remelted into 12" diameter ingots. Melting was done in an argon atmosphere. The chemical and spectrographic analyses of these ingots is reported in Table II.

Ingot No. 1X082 was forged to a 2" thick by 10" wide slab at 980°C in air. The total preheating and forging time did not exceed 2 hours. Reductions of 1/2" to 1" per blow could be made on forging. Approximately 1/16" of metal was machined from the surface of the forged slab to remove all scale and oxide or nitride contamination.

Two-inch thick slabs cut from one end of cast Ingot No. 1X081 were hot rolled at 980°C. The slabs were preheated 1-1/2 hours at 980°C in a helium atmosphere and rolled 92-1/2% reduction in thickness to 0.100". This rolled sheet had a slight "orange peel" type of surface, but was otherwise free from defects.

All Zircaloy-II finished from the forged and scalped Ingot No. 1X082 was hot rolled at 850°C after 45 minutes of preheating. Reductions of 17% per pass with a reheat time of 5 minutes per pass were found to be most satisfactory. Sheet was hot rolled to within 10% or 20% of the desired finished thickness and cold finished to size after cleaning.

Hot rolled Zircaloy-II sheet was cleaned by sand blasting with alundum sand followed by pickling in a 10% HNO₃ - 2% HF acid bath. The surface of hot rolled and cleaned material was smooth, bright, and free from visible defects.

Maximum cold reductions of 20% to 25% at 6% to 10% per pass could be made on hot rolled or annealed Zircaloy-II. Cold worked sheet was effectively annealed by heating 10 minutes at 850°C in a helium atmosphere followed by water quenching.

A commercial grade of low-carbon, low-nitrogen steel was used for protective steel cans on assembled cladding billets. This material, Ti-Namel steel, was used to prevent contamination of the Zircaloy-II by nitrogen from the steel.

Existing facilities of the ANL shops and laboratories were used in the preparation, fabrication, and testing of all clad fuel plate assemblies. Detailed description of specific fabricating and testing equipment may be found elsewhere.⁽²⁾

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IV. CLADDING BILLET ASSEMBLY

The billets designed for roll cladding uranium alloys with Zircaloy-II were a modification of the true picture frame type of assembly and were patterned after the type used at ANL for roll cladding uranium with zirconium.⁽²⁾ Since it is questionable whether contamination of the metal surfaces at the interface can be entirely avoided, it seemed imperative that some contamination be tolerated in a process which could be used for quantity production of plates. Such contaminants could either be attenuated by resorting to reduction by rolling or by diffusion into the core or clad metal. Previous tests with zirconium-clad uranium billets, which were carefully assembled with the cladding material sealed around the core by welding and not evacuated, resulted in bonded plate which was equal to or better than plate rolled from evacuated billets. By maintaining a minimum air gap between the clad and core on assembly and by sealing the core inside the cladding jacket to eliminate further contamination while heating prior to rolling, the most favorable conditions for minimal effects of billet contamination should be obtained. The cladding billet designed for this process had a minimum air gap between the core and clad of 0.001 cubic inch per square inch of interface on assembly.

Particular attention was given to the surface preparation of materials prior to assembly. The effect of deviations in the preparation of the Zircaloy-II cladding material was rolled and sheared or machined to size. Most cladding material was cleaned with an acid bright dip in hot concentrated nitric acid containing a trace of hydrofluoric acid (less than 1%), followed by washing in hot water. Some of the material was finished by machining all over, degreasing, and washing; some was vapor blasted with a silica grit and washed. All uranium alloy core material was machined from hot rolled bars and sand blasted with a silica sand just prior to assembly. The Ti-Namel steel cans were machined from hot rolled plate, degreased, and washed. Figure 1 shows the essential parts of a cladding billet prior to assembly. Dimensions of components for cladding billet assemblies are given in Table III. A drawing of the Ti-Namel steel can is presented in Figure 2. The inside dimensions of the can were altered to suit other cladding billets.

In assembling a cladding billet the alloy core was jacketed with top and bottom cover plates, side plates, and end plates of Zircaloy-II which were pressed tightly against the core and held in position while the Zircaloy-II was manually welded with an inert-gas shielded arc to seal the core inside the Zircaloy-II jacket. The top and bottom surfaces of this assembly were cooled by means of water-cooled copper plates during welding to prevent overheating and minimize contamination of the mating surfaces. A pressure of 3 to 4 tons per square inch was maintained on the cladding assembly during welding. The jacketed core was assembled in the Ti-Namel can which was then clamped and held in a vice while the can and end plugs were welded with carbon steel filler rod. No attempt was made to keep this assembly cool while sealing the can.

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Billet No. 10 was assembled with a 0.005" thick, high purity niobium foil barrier between the core and both the top and bottom cladding cover plates.

V. CLADDING BILLET PROCESSING

Several series of cladding billets were processed to study roll bonding variables to establish optimum conditions for producing a satisfactory fuel plate.

Rolling temperatures were selected which were considered most likely to contribute to a good bond at the Zircaloy-II to Zircaloy-II interface along the sides and ends of the clad plate, and at which the resistance to deformation of the core material would most nearly match that of the cladding. The gamma-transformation temperature for the two uranium-niobium alloys is very nearly the same, 680°C for the uranium - 3 w/o niobium and 690°C for the uranium - 6 w/o niobium. Since the roll bonding temperatures to be investigated were in the gamma range, it seemed probable that both core alloys would have almost the same rolling characteristics. This was partly confirmed by results from hot working the cast alloys.

All cladding billets in Ti-Namel steel cans were preheated 45 minutes in air prior to rolling, and reheated 5 minutes after each pass to maintain the rolling temperature. Rolling temperatures of 740°C, 770°C, 800°C, and 850°C were investigated. Reductions on rolling were varied from 10% to 17% per pass with total reductions of 50% to 90% in thickness. The effect of rolling speeds of 25, 50, and 75 feet per minute on rolled plate dimensions was also determined. The results effected by these variances in procedures are discussed in Section VIII.

Billets were rotated 180°C after each pass to reverse the direction of rolling relative to the elongation of the core and to minimize the accumulation of core material at one end of a clad plate.

Two billets were rolled without a protective steel can. Billet No. 5 was given an initial 50% reduction in a Ti-Namel can, removed from the can, heated in a helium atmosphere, and hot rolled an additional 40%. Billet No. 39 was hot rolled 80% reduction in thickness without a protective steel can. This billet was heated in a carbonate salt bath, Houghton Liquid Heat 235.⁽³⁾ The composition of this salt was about 28% LiCO_3 , 33% NaCO_3 , and 39% KCO_3 . This salt bath effectively protected the Zircaloy-II jacket welds and billet surface from scaling during heating at 770°C.

Roll cladding variables introduced in the fabrication of cladding billets No. 1-10, inclusive, and billet No. 39, are given in Table IV.

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A group of 6 billets, No. 11-16, inclusive, were rolled to produce clad plates with both uranium - 3 w/o niobium and uranium - 6 w/o niobium alloy cores. The surface preparation of the cladding material and the rolling speed were the only variables introduced in this group of billets as may be noted in Table V.

The protective Ti-Namel steel cans were readily removed from hot rolled clad plates by shearing the edges of the can around the clad plate and stripping off the steel.

VI. FINISHING METHODS

To determine the limitations for specifying physical properties, shapes, and dimensional tolerances in the production of uranium-niobium alloy fuel elements, methods of finishing hot rolled plates were studied.

The maximum corrosion resistance and dimensional stability of the uranium-niobium alloys is obtained by quenching from the gamma temperature range. Several methods of heating and quenching these clad alloys to obtain a satisfactory gamma treatment were investigated. The simplest procedure was to heat the clad plates for 15 to 30 minutes at temperatures of 800°C to 850°C in either a lead bath or a carbonate salt bath followed by a water quench. Heat treatment of 1/8" thick plates by any of these processes resulted in a complete gamma quench. However it was almost impossible to quench a flat plate which was more than 6" or 7" long without having it warp and become distorted. Several plate sections were heated 30 minutes in a lead bath at 850°C and quenched between pressurized, water-cooled copper plates on a 50-ton hydraulic press. Plates heat treated in this manner were free from warping and distortion. Corrosion test results on alloy core material quenched between chill plates indicated that a corrosion resistant gamma treatment had been obtained.

The surface finish and dimensional tolerances of hot-rolled plate can generally be improved by cold finishing. This is particularly true when the hot rolling is done inside a sheath or protective can. Thickness variations of as much as ± 0.005 " have been noted on hot-roll clad plate. Attempts to cold finish hot-rolled plates from billets No. 1 to 6 resulted in severe cracking and disintegration of the plate with total reductions of 5% or less. Plates from the same billets, heat treated by water quenching from a solution temperature of 800° - 850°C, could be readily cold rolled 25% to 30% reduction at 6% to 7% per pass without rupture or edge cracking. Cold-rolled plates had a smooth, lustrous finish and were uniform in thickness within ± 0.001 ".

Plates which were roll clad 80% and 90% reduction in thickness usually had irregularly shaped ends which tapered by as much as 3/8" from one side to the other, with variations of 0.020" to 0.050" in cladding thickness

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over the last 1" of plate length. Corresponding irregularities were present in the distribution of the core at the plate end. To obtain accurate plate lengths and core end geometry, finished plates were cut to the desired length and the exposed core material sealed. The exposed uranium-niobium alloy core was etched to a depth of $1/8"$ to $3/16"$; a thin strip of cold-rolled Zircaloy-II sheet was then fitted into the cavity and sealed by welding with an inert gas shielded arc. Some difficulty was encountered in selecting an etchant which would preferentially attack the core at a desirable rate. The uranium-niobium alloys could be etched with warm concentrated aqua regia but not without considerable pitting of the exposed Zircaloy-II. A solution of 80% HCl and 20% water (by volume) appeared to be the best. The temperature was maintained between 60°C and 90°C . A temperature greater than 90°C resulted in attack of the clad, less than 60°C retarded the reaction with the core. Etching time varied with the composition of the core alloy and volume of solution but was generally between one and two hours.

Several methods were used to finish the surface of clad plate after welding, annealing, or gamma heat treatment to obtain a smooth bright metallic finish which would be corrosion resistant to water temperatures of 260°C or higher. This was successfully done by either vapor blasting the plate surfaces with - 400 mesh Alundum, or by removing 0.005" to 0.010" of metal from the plate surfaces by machining, using a vacuum chuck. Machine cuts were limited to 0.002" to 0.003" per cut to avoid contamination due to overheating the Zircaloy-II. Although finish machining is costly, a more lustrous finish is obtained and operations in which metal is removed from the surface could be utilized for accurate control of over-all plate dimensions.

Roll-clad plate which could be formed might find applications in the design of specially shaped fuel elements. Since heat-treated plate could be rolled 25% to 30% reduction in thickness, additional means of cold forming heat treated plates were investigated. Bending tests showed that roll clad plate could be bent up to 80° around a radius equal to the thickness of the plate without rupture. Strips of plate 6" long, $1/2"$ wide, and $1/8"$ thick, roll clad with 0.010" of Zircaloy-II, were twisted through 900° before breaking. Figure 3 shows a similar strip which has been twisted 540° without breaking. There was no evidence of separation between the cladding and core on any of these specimens. It is possible that fuel elements such as corrugated plates, welded tubes, twisted ribbons, or folded plate might be fabricated from flat roll clad plate.

VII. TESTING METHODS AND RESULTS

Clad plates were inspected by metallography, autoradiography, physical measurements, mechanical testing, and corrosion testing. Results of these tests were used to evaluate the effects of material preparation, methods of processing, and finishing operations.

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The high purity niobium foil barrier which was inserted between the core and clad of billet No. 10 was found to be evenly distributed and well bonded to both the core and cladding during hot rolling. This barrier may be clearly seen in a photomicrograph of the polished specimen in Figure 9.

Examination of the metallographic structure and bond between Zircaloy-II and Zircaloy-II at the cover plate to side strip interface on a plate rolled 80% reduction in thickness at 770°C showed no evidence of voids or inclusions and no clearly defined interface between the two layers of Zircaloy-II. A slight difference in grain size between the two layers of Zircaloy-II is the only indication of the original interface, as may be seen in Figure 10.

Autoradiograph examination of clad plate was found to be an effective means of locating the core material relative to the edges and ends of rolled plates and to show variations in the cladding thickness. Radiation emanating from the uranium core resulted in exposures on x-ray film resembling normal x-ray exposures. Although no cladding defects or core segregation was noted on any of the uranium alloy plates clad with Zircaloy-II which were examined, such defects have been readily located on zirconium-clad uranium plates.

Clad plates were placed with one side in contact with Kodak Type K Industrial X-ray Film. Portions of the film extending beyond the plate edges and ends were exposed with a flashlight to furnish an accurate outline of the entire plate. An exposure time of 16 to 20 hours gave a gradient contrast to the film through 10 to 20 mils of zirconium cladding.

Figure 11 shows two contact prints made from autoradiographs of uranium - 6 w/o niobium plates roll clad 80% reduction in thickness at 770°C. Plates were cut to 7-1/4" lengths and the ends sealed by welding. Both plates have a nominal cladding thickness of 0.015" as finished. The plate in Figure 11a was finished by machining 0.010" to 0.015" of metal from the surface. The light area on the right side of Figure 11a indicates a considerable variation in clad thickness, which probably resulted from the plate being warped on one end and consequently more metal was machined from one side of the plate than from the other. The plate in Figure 11b was finished by vapor blasting and the thickness of the cladding appears to be uniform throughout its length. The core distribution relative to the edges and ends of both plates is quite regular.

A method was devised for tensile testing alloy clad plate which proved satisfactory and reliable. The test specimen was prepared by percussion welding a 1/4" diameter pure zirconium stud to the cladding on one side of a clad plate. The cladding at the base of the stud was then machined down to the core as shown in Figure 12. With the plate supported in a holding jig, the cladding adhering to the stud could be pulled from the core in a tensile testing machine. A welding technique was developed which gave

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reproducible welds. Weld penetration was obtained between the stud and cladding without affecting the structure of the cladding or core metal adjacent to the roll bond. The metallographic structure of a typical weld cross section is shown in Figure 13. Data obtained on bond tensile strength from experimental clad plates is given in Table VIII.

Representative samples from rolled and heat treated plates were given a bend test to obtain data on plate ductility and formability. Test strips 1/2" wide, taken at 90° to the direction of rolling, were subjected to a guided bend test around mandrels with 1/8" and 1/16" radii. Measurement of the angle through which strips could be bent before rupturing furnished a quantitative evaluation of plate formability. Table IX summarizes the bend test results.

Hardness measurements were taken on the core alloys as cast, before and after roll bonding, and after heat treatment. The results of these measurements are reported in Table X. Hardness values for both uranium-niobium alloys were found to be quite similar at each stage of fabrication, i.e., casting, rolling, and heat treating.

Perhaps the most valuable test for clad fuel elements, short of an actual service test, is a water corrosion test at temperatures corresponding to the intended service temperatures. Sections cut from roll clad alloy plates were corrosion tested in static water at 300°C with the alloy core material exposed to evaluate methods of heat treatment. Integrally clad plates with both welded and as rolled end closures were tested in static water at 260°C to compare end closures and to appraise the Zircaloy -II edge bonds. The effects of surface finishing procedures on the corrosion resistance of Zircaloy-II were also noted.

The results of corrosion tests on clad plates with exposed core material are given in Table XI. Clad plates were sectioned with the core material exposed across the width of the plate. Figure 14 shows a photomacrograph of sections cut from heat treated plates No. 7-10, inclusive, after corrosion testing 410 hours in 300°C degassed distilled water with the core material exposed. Note the selective attack of the bond area on all samples except No. 10 which had a 0.001" thick niobium barrier between the clad cover plates and core.

Corrosion test data on integrally clad plates with the ends sealed is given in Table XII. All plates were heat treated 1/2 hour at 850°C prior to corrosion testing.

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VIII. DISCUSSION OF RESULTS

The first group of plates rolled, from billets No. 1-6, inclusive, were clad with Zircaloy-II which had been fabricated by hot rolling a total of 92-1/2% reduction in thickness at 980°C without sufficient cleaning after hot rolling to remove surface contamination. Little or no bond was present between the Zircaloy-II and Zircaloy-II at the edge or end of any of these plates. The cladding could also be chipped loose at the clad to core interface after bonding, particularly on plate No. 2 which was rolled at 850°C. The wide range of bond tensile strength values noted on these plates, 15,000 psi to 47,000 psi, may be due to an erratic surface condition in the cladding material.

Plates clad with Zircaloy-II which had been fabricated from the forged and scalped billet, finish hot rolled at 850°C, and thoroughly cleaned prior to assembly into the cladding billet, all had satisfactory Zircaloy-II to Zircaloy-II bonds at the plate sides and ends as well as good bonds between the clad and core. Various surface preparations of the cladding material prior to billet assembly (pickling, vapor blasting, or machining) resulted in equally good bonds. The preparation of cladding and core materials had no apparent effect on cladding thickness variations.

Incomplete bonding between the core and clad on several plates could be related to defective pre-rolling welds. The use of automatic welding equipment or a more thorough study of the effect of manual welding variables on pre-rolling welds would be desirable.

Casting alloy core material directly into a pre-fabricated Zircaloy-II jacket would eliminate several steps in the processing of core materials and cladding billet assemblies. The feasibility of such a procedure should be established.

Attempts to roll clad commercially pure uranium with zirconium by reducing a cylindrical billet in oval-edge-oval passes resulted in irregular cladding thicknesses due to the hardness differential of the two materials. The uranium-niobium alloys and Zircaloy-II have nearly the same resistance to deformation at temperatures from 770°C to 800°C, and it is possible that improved edge bonds and simplified billet assemblies could be had by roll bonding a cylindrical billet in oval-edge-oval passes. Hot hardness data and roll pressure data on these alloys at proposed roll bonding temperatures would be helpful in developing any new rolling process.

Roll bonding procedures which produced the best clad plates with uranium - 3 w/o niobium alloy cores were directly applicable to roll bonding the uranium - 6 w/o niobium alloy core material. Plates with the most uniform over-all dimensions, cladding thickness, best surface condition, and best bonds were effected by rolling 80% to 90% total reduction at 770°C with

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17% reduction per pass. Some control over plate width could be had by regulating the rolling speed. Increased rolling speeds resulted in less variation in over-all plate width and less total width increase. None of the roll clad plates had a bond between the alloy core and the Zircaloy-II along the side of the plate.

The dimensions of clad plates rolled in Ti-Namel steel cans were all fairly uniform in width and thickness. Variations in rolling temperatures from 740°C to 800°C and total reductions of 50% to 90% in thickness had comparatively little effect on width increase. Billet No. 2 which was rolled at 850°C had less increase in width than billets rolled at lower temperatures. All billets (No. 11-16, inclusive) rolled with thicker side strips of Zircaloy-II (see Table III) showed appreciably less width increase on hot rolling, possibly due to additional restraint of the thicker Zircaloy-II strip. Plates which were hot rolled without a protective steel can from billets No. 5 and 39 evinced extensive lateral spread.

Although integrally clad plates can be made by rolling without a protective steel can, there is insufficient side restraint on flat rolling to control plate width effectively. It is expected that the use of edge rolls would control plate width and might also effect a clad to core bond along the side of the plate.

No attempt was made to establish an optimum heat treatment temperature, time, or quenching rate on either of the uranium-niobium core alloys. Satisfactory gamma treatments have been used on clad plates, but until an optimum treatment is established for these alloys, the effect of heat treatment on bond strength, diffusion, or corrosion resistance of the clad to core interface cannot be evaluated.

Present methods for sealing the ends of clad plates finished to specific length have produced corrosion resistant seals. This type of closure often leaves a void or gap between the end of the core and the filler strip which may interfere with heat transfer at the plate end. A gap of this sort may be seen in the cross section of a plate end closure in Figure 15. Improved methods of welding or sealing the ends of clad plates would be desirable.

The surface of plates finished by either machining all over or by thorough vapor blasting with alumina grit, followed by pickling, were equally corrosion resistant in 260°C water. Plates which were machined had a more lustrous appearance prior to corrosion testing than vapor blasted plates.

No serious difficulties were encountered in obtaining plate with uniform cladding thicknesses. Variations of less than 10% in thickness could be maintained for cladding thickness of 0.010" to 0.020". The thickness of clad uranium - 6 w/o niobium was generally more uniform than for clad

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uranium - 3 w/o niobium. The average clad thickness deviation for clad uranium - 3 w/o niobium was about 0.60 mil and for clad uranium - 6 w/o niobium it was about 0.30 mil. The average clad thickness deviation reported for zirconium-clad uranium plates was from 1.0 mil to 1.9 mils.⁽²⁾

Early corrosion tests on samples of plate having the core material exposed, plates No. 2 and 3 of Table XI, showed that the clad to core interface was selectively attacked. It was not known whether this attack was due to a galvanic couple between the core and clad, selective attack of the diffusion layer, or presence of inclusions in the bond interface. The presence of a high purity niobium foil barrier between the cladding and the core (plate No. 10) was found to combat this selective corrosion.

The tests described in this report have served to evaluate fabrication procedures. Thermal cycling tests of clad plates would give valuable data on effective heat treatments and the value of the cladding material to restrain growth of un-heat treated core materials.

IX. DEVELOPMENT OF PLATES FOR THE MTR TEST LOOP

A series of clad plates were prepared which were intended for irradiation tests on uranium-niobium alloy core material in the MTR 260°C water test loop. Measurements of corrosion resistance and dimensional stability are to be obtained on both uranium - 3 w/o niobium and uranium -6 w/o niobium clad with 0.015" of Zircaloy-II.

For testing in the MTR high-temperature water loop, plates finished according to the following specifications were required.

Over-all Length	$7 - 1/4" \pm 1/16"$
Over-all Width	$1.875" + 0.000"$ $- 0.030"$
Over-all Thickness	$0.125" + 0.000"$ $- 0.005"$
Core thickness	$0.095" + 0.000"$ $- 0.005"$
End Clad	0.080" minimum
Side Clad	$3/16" \pm 1/16"$
Minimum Cladding	0.010"

Six plates of each core composition were needed. Three plates of each composition were intended to be located in-pile for irradiation, and three of each composition were intended to be located outside of the reactor for corrosion only.

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A total of 19 billets were prepared and fabricated into plates. Preparation of core alloys and Zircaloy-II cladding materials has been previously described. Two types of plates were made, differing only in cladding thickness. One group (billets Nos. 20 to 26, inclusive, and Nos. 27 and 38) was fabricated to width and thickness by rolling, and another group (billets Nos. 27 to 36, inclusive) was fabricated with the cladding thickness oversize to permit machining to desired dimensions. The dimensions of the core and cladding components for these billets are given in Table XIII.

The U/Nb core alloys used in these plates were prepared by melting and casting as shown in Table XIV. Chemical and spectrographic analyses for these alloys are given in Table XV. These alloys were fabricated to bars, machined, then sand blasted just prior to assembly.

The Zircaloy-II cladding material was used as rolled and bright dipped in hot concentrated nitric acid containing a trace of hydrofluoric acid.

Ti-Namel steel cans described previously were used to encase the cladding billet for protection and support.

All billets were rolled at 770°C from a muffle furnace. Reheats after every pass were employed to maintain rolling temperature. Billets were reduced a total of 78% in thickness using 17% reduction per pass on a 16" x 24", 2-high mill. Billets were turned 180° endwise so as to change rolling direction with each pass to promote uniformity.

After roll bonding, the steel cans were removed from the plate by shearing and stripping. Then, to put the core alloy in the bonded plate in condition amenable to cold rolling, a gamma treatment was performed (1/2 hour at 850°C solution treatment followed by a water quench). All plates were cold rolled to reduce the ± 0.005 " thickness variations to ± 0.001 " and to improve the surface finish of the plate. A 5-3/4" x 16" x 12", 4-high mill was used for the cold rolling. Reductions of from 9-1/2% to 10% were accomplished. Plates which were intended for use as rolled were finished to a thickness of 0.122" to 0.125" (billets 20 to 26, inclusive, 37 and 38). Plates which were machined to final thickness were rolled to 0.150" (27 to 36, inclusive).

Plates were adjusted to length by cutting to 7-5/16", two plates being obtained from each rolled billet. This cutting exposed core alloy at each end of the plates which required sealing. Sealing was accomplished by etching back the core 1/8" to 3/16", inserting a Zircaloy-II filler strip, and fusing cladding to filler strip by welding with an inert gas shielded arc.

To insure corrosion resistance of the core alloy, a gamma treatment was given each plate after the welded and closures were made, the same treatment used to secure ductility for cold rolling. Even though the prior

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treatment for cold rolling promotes corrosion resistance it was feared the cold rolling and possible tempering from welding would cause reversion to the non-corrosion resistant state.

At this point plates from billets 20 through 26, 36, and 37 were finish fabricated and heat treated. Heat treating and welding scale were removed by vapor blasting with -400 mesh alumina. Plates from billets 27 through 35 were machined to thickness by removing 10 to 12 mils from each face. Machining was done by taking light cuts of 2 to 3 mils with a fly cutter. Plates were held for machining in a vacuum chuck.

All rolled plates were straight, had smooth clad surfaces free from visible defects and were within the specified tolerances. Plates appeared to be well bonded at their edges and ends except plate No. 23 which had one edge unbonded for a distance of approximately 5". Plate No. 22 developed an edge split, 3" in length, upon gamma solution treatment and water quench. All other plates were visually satisfactory after heat treatment.

Fabrication procedure for these roll clad MTR plates is contained in Table XVI. Photographs of plates prior to corrosion testing are shown in Figures 16 and 17. The plates shown in Figure 16 were finished by machining; those in Figure 17 were finished by vapor blasting. Cladding thickness analysis and bond tensile strength measurements for some of the 6 w/o Nb alloy clad plates are given in Table XVII. Of the 38 possible plates from the 19 billets rolled, 4 plates (Nos. 22, 23) were rejected because of faulty edge bonds, and 4 plates (Nos. 25, 27) were rejected because of faulty machining. The remaining 30 plates were considered satisfactory for further acceptance tests.

All plates were autoradiographed on one side for the determination of uniformity of clad thickness and location of core. The 6 w/o Nb alloy plates appeared to have slightly more uniform cladding thickness than the 3 w/o Nb alloy plates as concluded from the lack of mottle in the autoradiograph of 6 w/o plates. Some of the machined plates showed variation in cladding thickness, one (No. 30-2) being so severe as to cause its rejection (see Figure 11a).

The principal acceptance tests performed on these plates were static and dynamic corrosion in 260°C (500°F) degassed water. The water flow rate in the dynamic test was approximately 20 ft/sec. Plates were first tested in static water for various lengths of time, and then selected plates were corrosion tested in a dynamic loop. A summary of corrosion data is presented in Table XVIII. Although all plates were not tested for the same periods of time, some plates withstood periods up to 449 hours static - 0 dynamic, and 450 hours static - 114 hours dynamic. Of these thirty plates, one (No. 21-2) swelled but did not rupture after 270 hours of static corrosion; two plates (Nos. 32-1 and 26-1) developed white streaks on corrosion, one of

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which (No. 26-1) was salvaged by machining off the white area, and the edge bond on No. 21-1 split, but did not enter the core after 267 hours static - 114 hours dynamic corrosion testing. Photographs of plates after corrosion testing are shown in Figures 18 and 19. The plates in Figure 18 were finished by machining; those in Figure 19 were finished by vapor blasting.

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Table I

MELTING AND CASTING DATA FOR URANIUM RICH U/Nb ALLOYS

Billet No.	Nominal Composition, Wt % Nb	Charged Weight, kg		Form of Nb Alloy Addition	Temperature, °C		Vaccum, mm Hg		Heat Time, Hr-Min	Casting Weight, kg
		U	Nb		Max.	Pouring	Min	Pouring		
H-204	3	5.150	0.169	30-mil Sheet	1750	1390	4×10^{-4}	5×10^{-5}	5-55	5.10
H-205	3	5.125	0.159	30-mil Sheet	1750	1400	7×10^{-4}	8×10^{-6}	5-55	5.20
H-242	3	4.78	0.163	Presintered Chunks	1570	1270	--	--	5-30	4.63
H-248	6	4.50	0.33	Presintered Chunks	1550	1430	6×10^{-3}	5×10^{-4}	5-37	4.80
H-253	6	4.90	0.33	Presintered Chunks	--	1400*	3×10^{-3}	1×10^{-4}	4-35	4.80
H-254	6	5.22	0.35	Presintered Chunks	1650	1425	9×10^{-4}	4×10^{-5}	4-45	5.27

*Approximate temperature, sight glass clouded

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Table II

CHEMICAL AND SPECTROGRAPHIC ANALYSIS FOR URANIUM-NIOBIUM ALLOYS AND ZIRCALOY-II

Material	Nb (%)	N (ppm)	C (ppm)	Sn (%)	Ni (%)	Cr (%)	Fe (%)	Spectrographic Analysis (ppm)										
								Al	B	Cu	Fe	Mg	Mn	Na	Ni	Pb	Si	Ti
H-204	2.84	75	34					5	1	1	20	0.5	0.5		15	2	70	
H-205	2.68	15	20					5	2	1	10				15		40	
H-242	3.40	70	54					5		2	20	0.5	3		15		<10	
H-248	6.48	178	68					5	0.1	1	30				20		60	
H-253	6.54	150	51					5	0.1	1	50	10	5	2	12		60	
H-254	6.33	115	29					5		1	50	2	3	2	18		60	
1X081		51		1.45	0.049	0.106	0.115	60	<.1	7	800	13	18	<10	400	40	<50	1000
1X082		54 64		1.57	0.048	0.104	0.135	60	<.1	7	800	10	18	<10	400	40	<50	1300

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Table III

DIMENSIONS OF COMPONENTS FOR CLADDING BILLET ASSEMBLIES

Billets No. 1-10, inclusive			
Billet Component	Width (In)	Length (In)	Thickness (In)
Core	1.500	3.000	0.400
Cladding Cover Plates	1.700	3.200	0.100
Cladding Side Plates	0.400	3.200	0.100
Cladding End Plates	0.400	1.500	0.100

Billets No. 11-16, inclusive			
Billet Component	Width (In)	Length (In)	Thickness (In)
Core	1.335	3.375	0.475
Cladding Cover Plates	1.725	3.525	0.075
Cladding Side Plates	0.475	3.525	0.195
Cladding End Plates	0.475	1.335	0.075

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Table IV

FABRICATION PROCEDURE FOR ROLL CLADDING URANIUM - 3 w/o NIOBIUM WITH ZIRCALOY-II

Billet No.	Origin of Core Material	Origin of Clad Material	Cladding Billet Red./pass (%)	Total Red. (%)	Rolling Temp. (°C)	Significant Variable and Remarks
1.	H-204	1X081	10	80	770	Temperature and reduction arbitrarily selected as being suitable for rolling U-3w/o Nb and Zircaloy-II.
2.	H-204	1X081	10	80	850	Billet rolled at 850°C to determine effect of temperature on bonding and clad to core diffusion.
3.	H-204	1X081	17	90	770	Total reduction and reduction per pass increased to determine effect of increased reduction on bonding.
4.	H-205	1X081	17	50	770	Total reduction decreased to 50% to determine effect on bonding.
5.	H-205	1X081	17	80	770	Billet rolled 50% reduction in Ti-Namel can, balance after removal from can to determine feasibility of rolling without a protective steel can.
6.	H-205	1X081	17	80	770	This plate was fabricated for corrosion testing as rolled and heat treated.
7.	H-216	1X082	17	80	770	Surface preparation of cladding material improved over that used for billets No. 1-6, inclusive.
8.	H-216	1X082	17	80	800	Rolling temperature varied to study effect on bonding and clad to core diffusion.
9.	H-216	1X082	17	80	740	Rolling temperature varied to study effect on bonding and clad to core diffusion.
10.	H-216	1X082	17	80	770	Billet rolled with 0.005" thick high purity niobium foil between clad and core.
39.	H-238	1X082	17	80	770	Billet rolled 80% reduction out of carbonate salt bath without protective steel can.

Table V

FABRICATION PROCEDURE FOR ROLL CLADDING URANIUM - 3 w/o NIOBIUM
AND URANIUM - 6 w/o NIOBIUM WITH ZIRCALOY-II

Billet No.	Origin of Core Material	Origin of Clad Material	Surface Preparation of Clad Material	Total Red. (%)	Rolling Temp. (°C)	Roll Speed (Ft./Min)	Significant Variable and Remarks
11.	H-240 U-3 w/o Nb	1X082	Machined all over, degreased, water rinse.	80	770	75	Cladding material machined all over prior to assembly to determine effect on bonding.
12.	H-242 U-3 w/o Nb	1X082	Machined all over, degreased, acid bright dip.	80	770	75	Cladding material machined and acid bright dipped to determine effect on bonding.
13.	H-242 U-3 w/o Nb	1X082	As rolled and bright dipped.	80	770	75	Cladding material as rolled and acid bright dipped to determine effect on bonding.
14.	H-237 U-6 w/o Nb	1X082	Machined all over, degreased, water rinse.	80	770	75	U-6 w/o Nb Core with cladding material machined to establish bonding.
15.	H-238 U-6 w/o Nb	1X082	As rolled and bright dipped.	80	770	25	Rolling speed varied to study effect on width increase while hot rolling.
16.	H-238 U-6 w/o Nb	1X082	As rolled and bright dipped.	80	770	50	Rolling speed varied to study effect on width increase while hot rolling.

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Table VI

PHYSICAL MEASUREMENTS OF HOT ROLLED CLAD PLATES

Billet No.	Thickness as Rolled (In.)	Width as Rolled (In.)	% Width Increase on Rolling	Camber	Cladding Dimensions (mils)					
					Average ⁽¹⁾ Thickness	Average ⁽²⁾ Deviation	Average ⁽³⁾ Variation	Range of ⁽⁴⁾ Deviation	Minimum Clad Thickness	90% of ⁽⁵⁾ Measurement Range
1.	0.126 to 0.129	1.820 to 1.834	7 to 8	None	22.1	0.69	0.69	+2.0/1.6	20.5	20.5 to 23.5
2.	0.118 to 0.121	1.798 to 1.815	5-1/2 to 6-1/2	None	20.1	0.65	0.60	+2.1/1.8	18.3	18.5 to 21.5
3.	0.063 to 0.065	1.857 to 1.875	9 to 10	None	11.2	0.37	0.48	+1.0/1.6	9.6	10.5 to 12.5
4.	0.296 to 0.298	1.822 to 1.825	7 to 7-1/2	None	48.0	0.71	0.66	+2.0/2.0	46.0	46.5 to 49.5
5.	0.121 to 0.123	1.936 to 1.952	14 to 15	1/8" per ft	21.1	0.62	0.53	+1.7/2.0	19.1	20.0 to 22.5
6.	0.123 to 0.128	1.840 to 1.867	8 to 9-1/2	None	(No measurements taken of cladding thickness)					
7.	0.118 to 0.122	1.870 to 1.884	10 to 11	None	21.6	0.66	0.48	+2.1/1.6	20.0	20.0 to 22.5
8.	0.118 to 0.124	1.844 to 1.868	8-1/2 to 9-1/2	None	21.6	0.63	0.53	+1.7/1.8	19.8	20.5 to 23.0
9.	0.122 to 0.126	1.853 to 1.875	9 to 10	None	22.3	0.39	0.52	+0.9/1.3	21.0	21.5 to 23.5
10.	0.117 to 0.123	1.864 to 1.876	9-1/2 to 10	None	22.4	0.72	0.55	+2.1/2.6	19.8	21.0 to 24.0
39.	0.125 to 0.126	2.056 to 2.087	21 to 22-1/2	None	19.6	0.64	0.49	+2.0/-1.6	18.2	18.5 to 20.0

- (1) Determined by averaging thickness measurements taken at 0.100" intervals across both sides of six sections, equally spaced along length of plate.
- (2) Determined by summing the differences (not algebraically) between average clad thickness and each measurement and dividing by total number of measurements.
- (3) Sum of differences between each pair of measurements (0.100" apart), divided by number of individual variations determined.
- (4) Greatest positive and negative deviation observed.
- (5) The frequency of occurrence of various clad thicknesses observed was plotted. The narrowest range of thickness embracing 90% of the measurements was recorded.

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Table VII

PHYSICAL MEASUREMENTS OF HOT ROLLED AND COLD FINISHED CLAD PLATES

Billet No.	Thickness as Hot Rolled (inches)	Width as Hot Rolled (inches)	% Width Increase on Hot Rolling	Thickness as Cold Finished (inches)	Width as Cold Finished (inches)	% Width Increase on Cold Finishing	Cladding Dimensions (mils)					
							Average(1) Thickness	Average(2) Deviation	Average(3) Variation	Range of(4) Deviation	Minimum Clad Thickness	90% of(5) Measurement Range
11	0.134 to 0.140	1.801 to 1.821	5.5	0.122 to 0.123	1.832 to 1.851	1.6	16.9	0.94	0.46	+2.1/-1.5	15.4	15.0 to 18.5
12	0.139 to 0.141	1.792 to 1.816	4.9	(Not cold finished)			(No measurements taken of cladding thickness)					
13	0.135 to 0.140	1.801 to 1.831	6.1	0.123 to 0.124	1.828 to 1.859	1.0	17.1	0.63	0.30	+1.5/-0.9	16.2	16.0 to 18.5
14	0.136 to 0.139	1.794 to 1.808	4.8	0.124 to 0.125	1.807 to 1.826	0.77	18.3	0.44	0.29	+1.5/-0.7	17.6	17.5 to 19.5
15	0.136 to 0.138	1.821 to 1.845	6.7	0.123 to 0.124	1.835 to 1.858	0.76	16.8	0.53	0.42	+2.0/-0.9	15.9	16.0 to 18.0
16	0.136 to 0.139	1.813 to 1.832	6.1	0.124	1.829 to 1.846	0.76	16.5	0.31	0.27	+1.4/-0.9	15.6	16.0 to 17.5

- (1) Determined by averaging thickness measurements taken at 0.100" intervals across both sides of six sections, equally spaced along length of plate.
- (2) Determined by summing the differences (not algebraically) between average clad thickness and each measurement, and dividing by total number of measurements.
- (3) Sum of differences between each pair of measurements (0.100" apart), divided by number of individual variations determined.
- (4) Greatest positive and negative deviation observed.
- (5) The frequency of occurrence of various clad thicknesses observed was plotted. The narrowest range of thickness embracing 90% of the measurements was recorded.

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Table VIII

BOND TENSILE STRENGTH OF ZIRCALLOY-II CLAD URANIUM -
NIOBIUM ALLOY PLATE

Billet No.	Roll Bonding Temperature (°C)	Total Reduction (%)	Fabrication	Ultimate Tensile Strength (psi)
1	770	80	As hot rolled	40,500 to 41,200
1	770	80	Heat treated ⁽¹⁾	35,500 to 47,100
2	850	80	As hot rolled	22,100 to 32,400
2	850	80	Heat treated ⁽¹⁾	21,000 to 35,600
3	770	90	As hot rolled	21,600
3	770	90	Heat treated ⁽¹⁾	15,000
4	770	50	As hot rolled	20,000 to 28,300
5	770	80	As hot rolled	22,300 to 33,200
6	770	80	Heat treated ⁽¹⁾	35,700
7	770	80	As hot rolled	43,200
7	770	80	Heat treated ⁽¹⁾	28,800
8	800	80	As hot rolled	44,200
8	800	80	Heat treated ⁽¹⁾	28,200
9	740	80	As hot rolled	44,000
9	740	80	Heat treated ⁽¹⁾	24,700
10	770	80	As hot rolled	47,700
11	770	80	Cold finished ⁽³⁾	28,400
13	770	80	Cold finished ⁽³⁾	21,800
14	770 ⁽²⁾	80	Cold finished ⁽³⁾	39,200
16	770 ⁽²⁾	80	Cold finished ⁽³⁾	30,000

(1) Heated 1/2 Hour at 850°C in lead followed by a water quench.

(2) Core Alloy Uranium - 6 w/o Niobium.

(3) Cold finished 6% reduction in thickness after heat treatment.

REF ID: A69110

Table IX

BEND TEST DATA

Plate No.	Core Alloy, % Nb	Rolling Temp. (°C)	Heat Treatment	Angle to Rupture (Degrees)	
				1/8" Radius	1/16" Radius
1	3	770	None	25	25
1	3	770	1/2 hr at 800°C, water quench	63	58
2	3	850	None	14	10
2	3	850	1/2 hr at 850°C, water quench	58	58
2	3	850	1/4 hr at 850°C, water quench	43	45
3	3	770	None	25	26
3	3	770	1/2 hr at 800°C, water quench	81	71
5	3	770	None	28	20

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Table X

HARDNESS VALUES OF CORE ALLOYS

Fabrication	Hardness - 3% Nb Alloy		Hardness - 6% Nb Alloy	
	DPH	Rockwell C	DPH	Rockwell C
As cast	475	47	423	43
As hot rolled, 45% reduction at 850°C, Air Cooled	512	50	272	26
As roll clad, 80% reduction at 770°C, Air Cooled	620	57	591	55
As roll clad, 80% reduction at 850°C, Air cooled	762	63		
As heat treated after cladding, 1/2 hr 800°C, water quenched	319	32	330	33
Heat treated as above and aged 84 hrs at 350°C	450	45		
Heat treated as above and aged 24 hrs at 500°C	423	43		

Table XI

CORROSION TEST DATA ON CLAD PLATES WITH CORE MATERIAL EXPOSED TO 300°C STATIC WATER

Plate No.	Rolling Temp. (°C)	Special Plate Characteristics	Exposure and Results					
			72 Hrs.	144 Hrs	214 Hrs	282 Hrs	410 Hrs	895 Hrs
2	850	Plate heat treated 1/2 hr. at 800°C, quenched between pressurized, water-cooled chill plates.	No change in appearance. Corrosion rate on core same as for water quenched U-Nb alloy.	Bond area selectively corroded to a depth of 3/4" from edge.	Complete failure of bond area. Test discontinued.			
3	770	(Same as 2, above)	(Same as 2, above)	Bond selectively attacked at spots to a depth of 0.050" from exposed edge.	Slightly more attack of bond.	Attack at bond extended to a depth of 0.150" from the exposed edge. Test discontinued.		
7	770	Plate heat treated 1/2 hr. at 850°C, water quenched.	(Same as 2, above)	Two slight bulges in clad due to selective attack of the bond area.	Previous two bulges showed increase in size.	Two bulges noted on each side of plate. Increased attack at interface.	Clad separated from 70% to 80% of core area. Test discontinued.	
8	800	(Same as 7, above)	(Same as 2, above)	No change.	Two slight bulges in clad due to selective attack of the bond area.	One additional bulge noted in clad. Slight increase in two previous bulges.	Clad separated from core over approx. 50% of area due to selective attack. Test discontinued.	
9	740	(Same as 7, above)	(Same as 2, above)	Two noticeable bulges in clad due to selective attack of the bond area.	Noticeable increase in selective attack at bond area.	Large bulges noted in clad over 1/2 of plate area.	Clad almost completely separated from alloy core due to selective attack at bond. Test discontinued.	
10	770	(Same as 7, above) Niobium foil barrier between core and clad.	(Same as 2, above)	No change.	No. change.	No change.	No change. No selective attack at clad to core interface.	Slight bulges at edges of plate which were not protected by the niobium foil barrier. Test discontinued.

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Table XII

CORROSION TEST DATA FOR INTEGRALLY CLAD PLATES EXPOSED TO 260°C STATIC WATER

Plate No.	End Closure	Pre-test Surface Preparation	Test Time Hrs.		Remarks
			Test Interval	Total Time	
6	One end as rolled. One end sealed by welding.	Surface vapor blasted with silica grit. Welded end of plate not completely sealed by welding. No Zircaloy-II bond at edge or end of rolled plate.	137	137	Adherent lustrous gray film.
			181	318	One corner ruptured and swollen.
			98	416	Large blister ruptured approx. 2" from one end of plate and cracks noted along edge bond line.
			134	550	Little change in appearance - test discontinued.
7	(Same as 6 above)	Surface vapor blasted with silica grit.	154	154	Coating of ZrO_2
		Pickled, then machined.	648	802	Iridescent oxide film, white ZrO_2 streak on one edge.
8	(Same as 6 above)	Surface vapor blasted with silica grit.	154	154	Coating of ZrO_2
		Pickled	648	802	Dark gray oxide film. Several white spots and one white streak on one edge.
9	(Same as 6 above)		154	154	White Coating.
		Pickled, then machined. Core alloy exposed by machining.	648	802	Iridescent film. One small white spot. Swelling on both sides near exposed core alloy. Swelling approximately 8 mils.
10	(Same as 6 above)	Surface cleaned by vapor blasting with -100 mesh silica after heat treatment. Acid bright dipped.	154	154	Lustrous iridescent film. Few white spots.
			336	490	A very small bulge $1/8" \times 1/4"$ developed.
			648	1138	No defect or opening detected in cladding. Cause of bulge not known.
11a	Both ends sealed by welding.	(Same as 10 above)	585	585	Adherent dull gray coating, white spots.
11b	(Same as 11a above)	(Same as 10 above)	585	585	Adherent dull gray coating with faint white streaks.
13	(Same as 11a above)	(Same as 10 above)	566	566	Adherent gray iridescent coating.
14a	(Same as 11a above)	Pickled	566	566	Adherent gray iridescent coating.
14b	(Same as 11a above)	(Same as 10 above)	61	61	Adherent dark gray coating. Plate showed small rupture.
			505	566	Bulge increased in size to approximately 10 mils high. Bulge continued to area over core alloy - end insert interface.
15a	(Same as 11a above)	Machined	61	61	Adherent lustrous dark gray coating. Black loose coating observed around crack at one end of plate which was present before testing. Crack probably caused by machining through cladding.
			505	566	Crack remained essentially the same size with no bulging of clad. No appreciable deposits of black product were observed.
15b	(Same as 11a above)	(Same as 10 above)	566	566	Lustrous dark gray coating and faint mottled white wash on several areas.
16a	(Same as 11a above)	Machined	385	385	Lustrous iridescent film. Small bulge on bond line.
			181	566	Slight increase in bulge on bond line.
16b	(Same as 11a above)	(Same as 10 above)	566	566	Adherent lustrous dark gray coating and faint mottled white wash on several areas. Hole was cut through cladding during identification marking exposing core alloy. No bulging observed during test.

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Table XIII

DIMENSIONS OF CORE AND CLADDING COMPONENTS OF BILLETS
USED FOR THE MANUFACTURE OF MTR TEST PLATES

Description of Component	Width (in.)	Length (in.)	Thickness (in.)	Billet Nos.
Uranium Alloy Core	1.410	3.375	0.475	20 to 26 incl.
Zircaloy-II Cover Plates	1.800	3.525	0.075	
Zircaloy-II Side Plates	0.475	3.525	0.195	
Zircaloy-II End Plates	0.475	1.410	0.075	
Uranium Alloy Core	1.410	3.375	0.475	27 to 36 incl.
Zircaloy-II Cover Plates	1.800	3.525	0.137	
Zircaloy-II Side Plates	0.475	3.525	0.195	
Zircaloy-II End Plates	0.475	1.410	0.075	
Uranium Alloy Core	1.410	3.375	0.450	37 and 38
Zircaloy-II Cover Plates	1.800	3.525	0.087	
Zircaloy-II Side Plates	0.450	3.525	0.195	
Zircaloy-II End Plates	0.450	1.410	0.075	

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Table XIV

MELTING AND CASTING DATA FOR URANIUM-NIOBIUM CORE ALLOYS FOR MTR TEST PLATES

Ingot No.	Nominal Composition, w/o Nb	Charge, kg U	Weight, kg Nb	Form of Nb Alloy Addition	Temperature, °C		Vacuum, mm Hg		Time of Heat, Hrs-Min	Casting Weight, kg	Cladding Billet Nos.
					Max.	Pouring	Min.	Pouring			
H-239	6	4.28	0.270	Presintered chunks	1580	1335	1.2×10^{-3}	1×10^{-5}	4 - 40	4.30	32 & 33
H-243	3	5.350	0.178	Presintered chunks	1560	1260	7×10^{-4}	5×10^{-6}	6 - 0	5.25	20, 21, & 27
H-246	3	5.48	0.190	Presintered chunks	1600	1300	1×10^{-3}	9×10^{-6}	5 - 30	5.13	37 & 38
H-253	6	4.90	0.33	Presintered chunks	--	1400	1×10^{-3}	1×10^{-4}	4 - 35	4.80	22, 23 & 24
H-254	6	5.22	0.35	Presintered chunks	1650	1425	9×10^{-4}	4×10^{-5}	4 - 45	5.27	25, 28 & 29
H-255	6	5.10	0.34	Presintered chunks	1675	1400	9×10^{-4}	1×10^{-4}	4 - 25	5.08	26, 30, & 31
H-259	6	4.15	0.28	Presintered chunks	1640	1430	1×10^{-3}	3×10^{-6}	3 - 40	3.98	34
H-260	6	4.77	0.32	Presintered chunks	1630	1430	9×10^{-4}	9×10^{-6}	1 - 43	4.74	35 & 36

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Table XV

CHEMICAL AND SPECTROGRAPHIC ANALYSES FOR MTR TEST PLATE CORE ALLOYS

Ingot No.	Chemical Analyses			Spectrographic Analyses										
	Nb, %	N, ppm	C, ppm	Al	B	Cu	Fe	Mg	Mn	Na	Ni	Pb	Si	Ti
H-239	6.03	--*	35			2	7				5		10	
H-243	3.26	--	27	5		2	10			5	15			
H-246	3.43	--	29	5	0.1	2	20	0.5		5	15		10	
H-253	6.01	--	51	5	0.1	1	50	10	5	2	12		60	
H-254	6.33	--	30	5		1	50	2	3	2	18		60	
H-255	6.43	--	37	5	0.1	1	40		3	2	18		30	
H-259	6.37	--	40	5		1	50	1	5	2	12		50	
H-260	5.76	--	30	5	0.1	1	30	1	3	3	18		40	

*Nitrogen Analytical Method found inaccurate, results not reported.

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Table XVI

FABRICATION PROCEDURE FOR ROLL CLADDING MTR TEST LOOP PLATES

Billet No.	Origin of Core Material	Origin of Cladding Material	Rolling Speed, Feet Per Minute	Comments on Hot Rolled Plate	Surface Finishing of Plate
20	H-243 U-3 w/o Nb	1X082	75	Good surface condition. Plate free from camber.	Vapor blasted with Alundum grit.
21	H-243 U-3 w/o Nb	1X082	75	Slight defect in edge bond 1" long x 0.050" deep on one side.	Vapor blasted with Alundum grit.
22	H-253 U-6 w/o Nb	1X082	50	Edge bond discontinuous for 2-3" along one side.	Plate heat treated, cold finished, and used for physical testing.
23	H-253 U-6 w/o Nb	1X082	50	Intermittent bonding between clad and core on one side.	Plate used for physical testing.
24	H-253 U-6 w/o Nb	1X082	50	(Same as 20, above)	Vapor blasted with Alundum grit.
25	H-254 U-6 w/o Nb	1X082	75	(Same as 20, above)	(Same as 24, above)
26	H-255 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 24, above)
27	H-243 U-3 w/o Nb	1X082	75	(Same as 20, above)	From 0.010" to 0.012" machined from plate surfaces.
28	H-254 U-6 w/o Nb	1X082	75	(Same as 20, above)	(Same as 27, above)
29	H-254 U-6 w/o Nb	1X082	75	(Same as 20, above)	(Same as 27, above)
30	H-255 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 27, above)
31	H-255 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 27, above)
32	H-239 U-6 w/o Nb	1X082	75	(Same as 20, above)	(Same as 27, above)
33	H-239 U-6 w/o Nb	1X082	75	(Same as 20, above)	(Same as 27, above)
34	H-259 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 27, above)
35	H-260 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 27, above)
36	H-260 U-6 w/o Nb	1X082	50	(Same as 20, above)	(Same as 27, above)
37	H-246 U-3 w/o Nb	1X082	75	(Same as 20, above)	(Same as 24, above)
38	H-246 U-3 w/o Nb	1X082	75	(Same as 20, above)	(Same as 24, above)

NOTE: All billets were hot rolled 78% reduction in thickness at 770°C, 17% reduction per pass.
Annealed plates were cold finished 9-1/2% to 10% after hot rolling.

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Table XVII

CLADDING THICKNESS ANALYSIS AND BOND TENSILE STRENGTH
OF ROLL CLAD URANIUM - 6 w/o NIOBIUM ALLOYS

Plate No.	Cladding Dimensions - Mils ⁽¹⁾						Ultimate Tensile Strength (psi)
	Average Thickness	Average Deviation	Average Variation	Range of Deviation	Minimum Clad Thickness	90% of Meas. Range	
22	15.8	0.31	0.31	+ 1.0/- 1.0	14.8	15.0 to 16.5	38,900
23	17.9	0.29	0.26	+ 1.1/- 1.0	16.9	17.0 to 18.5	50,000
24	14.9	0.35	0.25	+ 1.5/- 0.7	14.2	14.0 to 15.5	61,500
25	15.8	0.55	0.31	+ 1.4/- 1.1	14.7	14.5 to 17.0	61,700
26	15.4	0.53	0.34	+ 1.8/- 0.8	14.6	14.5 to 16.5	66,500

⁽¹⁾ Explanation of cladding measurements given in the foot notes of Tables VI and VII.

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Table XVIII

ACCEPTANCE TEST DATA FOR MTR HIGH TEMPERATURE WATER LOOP TEST PLATES

Met. Plate No.	Reactor Engr. Plate No.	Core Alloy Composition % Nb	Surface Preparation	Corrosion Test Time, Hrs.		Remarks
				Static	Dynamic	
20-1	4178	3	(1) Vapor Blast & Pickle	267	168	Rejected, clad thickness
20-2	4179	3	Vapor Blast & Pickle	290	54	
21-1	4176	3	Vapor Blast & Pickle	267	114	Edge bond opened part way to core
21-2	4177	3	Vapor Blast & Pickle	270	0	Rejected, plate swelled locally in spots
24-1	4174	6	Vapor Blast & Pickle	290	54	
24-2	4175	6	Vapor Blast & Pickle	290	54	Slight warping
26-1	4172	6	Vapor Blast & Pickle	267	168	Small white streak at one corner in static test. Streak removed by machining did not reappear in dynamic test.
26-2	4173	6	Vapor Blast & Pickle	267	168	White corrosion product at one corner. Area too large to be removed by machining.
28-1	4166	6	(2) Machined & Pickled	290	54	
28-2	4167	6	Machined & Pickled	449	0	
29-1	4162	6	Machined & Pickled	449	0	
29-2	4163	6	Machined & Pickled	290	54	
30-1	4158	6	Machined & Pickled	449	0	
30-2	4159	6	Machined & Pickled	290	0	Rejected, clad thickness
31-1	4156	6	Machined	450	114	Badly scratched
31-2	4157	6	Machined & Pickled	290	54	Rejected, bond test
32-1	4154	6	Machined	291	114	White streak on one corner of
32-2	4155	6	Machined & Pickled	290	54	
33-1	4152	6	Machined	291	168	
33-2	4153	6	Machined	291	168	
34-1	4164	6	Machined & Pickled	290	54	Rejected, bond test
34-2	4165	6	Machined & Pickled	290	54	
35-1	4150	6	Machined	450	114	
35-2	4151	6	Machined	450	114	
36-1	4160	6	Machined & Pickled	449	0	
36-2	4161	6	Machined & Pickled	449	0	
37-1	4170	3	Vapor Blast & Pickle	290	54	
37-2	4171	3	Vapor Blast & Pickle	290	54	Warped in static test
38-1	4168	3	Vapor Blast & Pickle	290	54	
38-2	4169	3	Vapor Blast & Pickle	267	168	

(1) Vapor blasted with -400 mesh alumina, to remove all scale pickled; washed and dried in six operations.
 1. Pickle 35% HNO_3 -3% HF , 2. Pickle 5% HNO_3 -5% $\text{Zr}(\text{NO}_3)_4$, 3. Pickle 5% HNO_3 , 4. Rinse in warm water, 5. Rinse in warm water, 6. Rinse in acetone and dry.

(2) Machining consisted of removing 10 to 12 mils from the faces and edges of plates with a fly cutter with plates held in a vacuum chuck.

NOTE: Plate No's. 24-1, 24-2, 26-1, 37-1, 37-2, and 38-1 were selected for in-pile irradiation and corrosion testing.

Plate No's. 20-2, 28-1, 29-2, 33-2, 34-2 and 38-2 were selected for out-pile corrosion testing only.

NOTES

ANL Negative No. 106-1086

Figure 1. Cladding Billet Components Prior to Assembly.

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Figure 2. Ti-Namel Steel Can Assembly For Cladding Billets 11-16, Inclusive.

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Figure 3. Uranium - 3 w/o Niobium roll clad with 0.010" of Zircaloy II,
twisted 540°

Macro 14,791

7/8X

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Macro 14,524

3-1/3X

Figure 4. Macrosection of Clad Plate No. 3, as Hot Rolled. Plate Sectioned at Equally Spaced Intervals Along its Length.

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Micro 14,664

500X

Figure 5a. Hot rolled 80% reduction in thickness at 770°C followed by slow cooling.

Micro 14,530

1000X

Figure 5b. Same material as 5a above, after heating 1/2 hour at 800°C in lead followed by a water quench.

Figure 5. Structure of Uranium 3 - w/o Niobium Alloy Core Material, Before and After Heat Treatment.

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Micro 14,489

1000X

Figure 6. Clad Plate Rolled 50% Reduction in Thickness at 770°C. Note Voids in the Clad to Core Interface.

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Micro 14,492

1000X

Figure 7a. Diffusion layer in clad plate rolled 80% reduction in thickness at 770°C.

Micro 14,493

1000X

Figure 7b. Diffusion layer in clad plate rolled 80% reduction in thickness at 840°C.

Figure 7. Effect of Rolling Temperature on the Diffusion Layer Formed at Uranium - 3 w/o Niobium to Zircaloy II Interface.

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Micro 14,789

500X

Figure 8. Diffusion Layer in Clad Plate Rolled
80% Reduction in Thickness at 770°C
and Heat Treated 1/2 Hour at 850°C

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Uranium
3 w/o Niobium
Alloy

High Purity
Niobium Barrier

Zircaloy II

Micro 14,659

500X

Figure 9. Niobium Barrier Between the Cladding and Core of a Plate Rolled 80% Reduction in Thickness at 770°C.

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Micro 14,663

100X

Figure 10. Cross Section of Zircaloy II to Zircaloy II Bond at the Edge of a Clad Plate Rolled 80% Reduction in Thickness at 770°C.

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Figure 11a.

Plate surface finished by machining. Cladding thickness becomes progressively thinner toward the right end of plate.

Figure 11b.

Plate surface finished by vapor blasting. Cladding thickness uniform throughout length of plate.

Figure 11. Autoradiographs of Roll Clad Plates Showing Core Distribution and Clad Thickness Variation.

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Figure 12. Specimen for Tensile Test of Bond Strength in Alloy Clad Plates.

Welded Assembly

Machining Detail

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Core Material

Zircaloy II

Heat affected zone
of Zircaloy II and
Zirconium stud.

Zirconium stud

Micro 14,748

50X

Figure 13. Cross Section of Percussion
Welded Tensile Test Specimen

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Macro 14,757

1X

Figure 14. Heat Treated Alloy Clad Plate Specimens Corrosion Tested 410 Hours in 300°C Water With the Core Exposed.

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Macro 14,783

16X

Figure 15. Cross Section of a Welded Plate End Closure Showing Void Between End Filler Strip and Alloy Core.

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ANL Negative No. 106 - 1211

Figure 16. Roll Clad Plates for the MTR Test Loop Prior to Corrosion Testing.
Surface Finished by Machining.

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ANL Negative No. 106 - 1212

Figure 17. Roll Clad Plates for the MTR Test Loop Prior to Corrosion Testing.
Surface Finished by Vapor Blasting.

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ANL Negative No. 111 - 1763

Figure 18. Roll Clad Plates for MTR Test Loop After Corrosion Testing.
Surfaces Finished by Machining.

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ANL Negative No. 111 - 1761

Figure 19. Roll Clad Plates for MTR Test Loop After Corrosion Testing.
Surfaces Finished by Vapor Blasting.

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