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1968 - 1969 USAEC/AECL
COOPERATIVE PROGRAM
ON
 U_3Si COEXTRUSION DEVELOPMENT
AND BILLET PREPARATION

P. A. Ard

February 1970

AEC RESEARCH &
DEVELOPMENT REPORT

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Fuels and Materials Department
Chemistry and Metallurgy Division

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1968-1969 USAEC/AECL COOPERATIVE PROGRAM
ON U_3Si COEXTRUSION DEVELOPMENT AND BILLET PREPARATION

P. A. Ard

ABSTRACT

This report covered work performed in two cooperative USAEC/AECL programs on U_3Si coextrusion and U_3Si billet preparation. The purpose was to develop technology for coextrusion of zirconium alloy clad fuel rod stock with small central axial holes to accommodate radiation induced swelling, and to develop alternate methods for producing cast uranium silicon alloy billets with a fine uranium and U_3Si_2 microstructure necessary for reducing the deoxidizing heat treatment time.

Several 0.600-in. OD coextrusions with small 0.200-in. diam central coaxial holes were produced from 2-1/4-in. billets containing castings of uranium silicon alloys with compositions of U-3.6 wt% Si to U-4.15 wt% Si. The U_3Si material was successfully coextruded with both Zr-2 and Zr-2.5Nb cladding materials.

Several 3-in. diam coextrusion billets were extruded at reductions of from 15.7/1 to 21/1, and at extrusion ram speeds of 40, 60, and 120 in./min. The billets were produced from cast U-3.8 wt% Si using both Zr-2 and Zr-2.5Nb for cladding material. These billets were all successfully extruded with a 0.250-in. diam coaxial void except for the billet extruded at the slower 40 in./min speed in which the mandrel overheated and failed. Several billet end designs were tested on the 3-in. diam billets in an effort to reduce the "rattlesnake" breaking which occurs near both ends of extrusions from both the 2-1/4-in. and 3-in. billets. Two lead end designs proved successful in preventing this breaking, but attempts at tail end designs were not satisfactory. Small defects in the U_3Si

core immediately under the cladding U_3Si interface were prevalent in most of the 3-in. billet extrusions. These defects are thought to be caused by billet chilling against the relatively cold tooling and small inclusions within the U_3Si cores.

Attempts to produce a finer uranium and U_3Si_2 microstructure in uranium silicon alloy billets by vacuum consumable electrode melting were not successful. However, a very fine microstructure was produced in the cast uranium silicon alloy shot made by the spinning disk method.

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1968- 1969 USAEC/AECL COOPERATIVE PROGRAM
ON U_3Si COEXTRUSION DEVELOPMENT AND BILLET PREPARATION

P. A. Ard

INTRODUCTION

This report covers the development work performed in two cooperative USAEC-AECL programs on U_3Si coextrusion development and U_3Si billet preparation methods. The program work performed in fiscal year 1968 covered coextrusion development using 2-1/4-in. billets. The fiscal year 1969 program continued the coextrusion development focusing on evaluation of billet and extrusion parameters using 3-in. billets. Also the 1969 program included both vacuum arc casting of uranium silicon alloy billets and the preparation of uranium silicon alloy shot..

The objectives of these programs were to develop the technology for coextrusion of zirconium alloy clad fuel rod stock with a small central axial void and to develop alternate methods for producing U_3Si billets with finer microstructures.

A fine evenly dispersed U_3Si_2 and free uranium partical size in the as-cast material would reduce the time required for the sluggish deltizing heat treatment, which converts the undesirable U_3Si_2 and free uranium to the more workable and corrosion resistant U_3Si . The central coaxial void in the coextrusions has the purpose of accommodating radiation induced swelling by providing a void into which the unconstrained fuel can expand. Thus, with this void, the fuel can operate for longer exposures without unacceptable external growth caused by fuel swelling. Fuel with a coaxial void has performed very satisfactorily under irradiation showing a very noticeable decrease in external swelling.

Studies conducted at Chalk River Nuclear Laboratories* indicate that lower power costs can be realized by using a higher density fuel than UO_2 in heavy-water-moderated power reactors. After studying several fuel possibilities, Chalk River personnel decided to test in detail U_3Si (U-3.8 wt% Si), which has a high density (15.58 g/cm^2 compared to 9.7 g/cm^3 for UO_2) with a low parasitic adsorption and good corrosion resistance in high temperature water in both the unirradiated and the irradiated condition.

The fuel for the heavy-water-moderated, boiling light water-cooled reactor is anticipated to be delta stabilized U_3Si with extended fuel life obtained through use of an internal expansion void. The element is fabricated from chill cast deltized rods, assembled in clad tubing and closed. Defect behavior of irradiated rods, which tend to bond during irradiation, is reasonably good. Defect behavior of unbonded rods is poor because of water logging. Both initial heat transfer and initial defect behavior could be improved by bonding. The fabrication technology would be substantially advanced if the axial void and the core-to-clad bond could be provided by an economical coextrusion step.

* G. H. Chalder et al. U_3Si as a Nuclear Fuel, AECL-2874. AEC Division of Technical Extension, Oak Ridge, Tennessee, May 1967.

1968 U₃Si COEXTRUSION DEVELOPMENT PROGRAMOBJECTIVES

The overall objective of the 1968 USAEC/AECL program on U₃Si coextrusion was to further the coextrusion technology of zirconium alloy clad U₃Si fuel stock with a small coaxial void using 2-1/4-in. diam coextrusion billets. This work, a continuation of the work previously performed by Battelle-Northwest, established the feasibility of producing Zr-2-clad U₃Si rods by coextrusion with a small coaxial center hole. Cast billets of uranium silicon alloys (U-3.6 wt% Si to U-4.15 wt% Si), heat treated to provide the desired U₃Si microstructure, were supplied by Eldorado Nuclear Limited for use in this coextrusion work. The following were the coextrusion objectives of this program:

- Improve the technique for producing the coaxial voids in the coextrusions
- Produce coextrusions clad from both Zr-2 and Zr-2.5Nb
 - a Solution-quench the Zr-2.5Nb clad coextrusions immediately after extrusion
- Extrude two cladding thicknesses, 0.025 and 0.015 in., for both cladding alloys and two U₃Si alloys
- Produce coextrusions with integral longitudinal ribs on the outer diameter of the clad rods which might serve as spacers for the fuel rods
 - a Coextrude enriched rods for irradiation testing
- Explore methods of straightening the coextrusions.

SUMMARY

Several 0.600-in. OD coextrusions were successfully extruded with a small 0.200-in. diam central coaxial void. These coextrusions were produced from castings of uranium

silicon alloys near the stoichiometric composition (U-3.6 wt% Si to U-4.15 wt% Si) which were induction melted, cast, and deltized. These coextrusions have demonstrated that a coaxial void for controlling irradiation swelling in the fuel can be successfully produced during the high temperature extrusions.

These uranium silicon alloys were successfully coextruded with both Zr-2 and Zr-2.5Nb. Clad thicknesses of 0.015 and 0.025-in. were produced with these two zirconium alloys on 0.600-in. diam rod. The cladding is uniform in thickness even though the uranium silicon alloys are as-cast and deltized with no prework prior to coextrusion.

The attempt to solution quench the Zr-2.5Nb clad material by water quenching the extrusion directly out of the die produced considerable cracking in the U_3Si core and some fracture of the brittle bond between the cladding and core. The purpose of this experiment was to determine whether the Zr-2.5Nb clad could be prepared for aging directly from the extrusion without a subsequent heat treatment. The billet extrusion temperature is adequate for solution heat treating the cladding.

A coextrusion was made with four longitudinal integral ribs on the outside of the Zr-2 clad rods to determine the feasibility of extruding the fuel rods with integral spacer ribs. The results appeared reasonably good, but the cost of producing the billet presently appears greater than the cost of attaching the spacers to the coextruded rods by conventional welding methods.

Two enriched (3% ^{235}U) coextrusions of both uranium alloys clad with Zr-2 were produced for irradiation testing in a Canadian test reactor.

The work, initiated on straightening of coextrusions by hot stretch-straightening, showed promise. However, results

indicated the desirability of vertical rather than horizontal stretching, and the necessity of insulated rod end gripping fixtures to provide more uniform resistance heating.

EXTRUSION BILLETS

Design

The design of the extrusion billets is shown in Figure 1, and the ribbed extrusion billet is shown in Figure 2. The dimensions of the U_3Si billet core and zirconium alloy sleeve for each extruded billet are given in Table 1. Zircaloy-2 billet end fillers were placed at each end of the U_3Si billet core in an effort to prevent rattlesnake type breakage in the coextrusions near the ends, as well as to reduce the peak forces encountered at both ends of the billet. This rattlesnaking and high force is believed attributable to chilling at the ends of the billet. The effect of the lead end filler is shown in Figure 3 which compares the lead end of a coextrusion from a billet with a lead end filler to an extrusion from a billet without a filler. While the filler did not completely eliminate the rattlesnaking, the length of broken coextrusion was reduced. The extrusion forces were still very high when the lead fillers were used. The force spike at the tail end of the extrusion would not appear when the graphite follower was preheated to the same temperature as the billet (850 °C) instead of the usual 400 °C temperature. The Zr-2 filler pieces have a substantially lower thermal conductivity ($0.04 \text{ cal/cm}^2/\text{cm}/^\circ\text{C}/\text{sec}$) than the Cu-10Ni end caps ($0.095 \text{ cal/cm}^2/\text{cm}/^\circ\text{C}/\text{sec}$) and thus, as the thickness of the Zr-2 end filler pieces increases, chilling of the billet ends should be reduced.

U_3Si Billet Cores

The U_3Si billets were prepared and supplied to BNW by Eldorado Nuclear Limited, heat treated, and machined to 2-1/4-in. OD. The billets were of U-3.6 wt% Si to U-4.15 wt% Si.

FIGURE 1. Standard Design Section Used for 2-1/4-in. Billets

FIGURE 2. Section of Billet Used for Producing Coextrusion with Integral Longitudinal Spacer Ribs

TABLE 1. 2-1/4-in. U₃Si Billet Core and Cladding Description

Billet No.	% Si	U ₃ Si Core		Cladding Sleeve			Comments
		OD, in.	Length, in.	Material	OD, in.	ID, in.	
516-M1T1	4.15	1.953	4.720	Zr-2.5Nb	2.148	1.960	
516-M1T2	4.15	1.954	4.752	Zr-2	2.148	1.965	Several 1/32-in. diam pits in core OD.
517-M1T1	4.15	2.024	4.722	Zr-2	2.143	2.035	Few 1/4-in. diam dark spots and several fine pepper-sized dark spots on core OD.
517-M1T2*	4.15	1.646	4.765	Zr-2	Ribbed 1.828	1.655	Few up to 1/8-in. diam dark spots and a few dark lines on core.
519-M1T1	3.7	1.953	4.997	Zr-2	2.146	1.962	
519-M1T2**	3.7	1.956	4.125	Zr-2.5Nb	2.140	1.988	Cladding wall machined thin
519-M2T1	3.8	2.022	4.995	Zr-2	2.148	2.030	1/16-in. diam dark spots on core OD.
519-M2T2	3.8	1.954	5.001	Zr-2.5Nb	2.123	2.001	Core OD machined undersized
2.95% En 522-B1T1	4.0	1.955	4.996	Zr-2	2.148	1.960	Few 1/16-in. diam dark pits on core OD.
2.95% En 523-B1T1	3.6	1.952	4.997	Zr-2	2.148	1.962	Four 1/64-in. diam dark pits on core OD.

- * Billet 517-M1T2 - Longitudinal cladding rib billet. Cladding OD dimension was taken between ribs. Ribs 0.152 in. high.
- ** Billet 519-M1T2 - Billet can leaked on initial preheat, damaging one end of the core. Damaged end removed, components cleaned, and recanned.

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FIGURE 3. Comparison of U_3Si Coextrusion Lead Ends Produced from Billet with Cone Billet Cap and Zr-2 Billet Filler (upper), and Billet with Flat Cup and No Filler Piece (lower)

The silicon content of each billet is given in Table 1. The deltizing heat treatment which converts the free uranium and U_3Si_2 in the as-cast alloy to U_3Si was performed at 800 °C.

Final machining performed on the U_3Si billets at BNW included drilling the small (0.360-in. diam) center mandrel holes. Drilling this hole with carbide drills proved very difficult and the method of producing the hole was changed to electric discharge machining.

Small pores of various sizes, apparently filled with a dark brittle substance, were noticed on the surface before and after final machining. A description of the defects on the outer diameter surface of the final machined surface is given in Table 1.

The end filler pieces at each end of the U_3Si billet core were Zircaloy-2 machined from bar stock.

Cladding Sleeves

The coextrusion billet cladding sleeves were machined from tubular extrusions of Zircaloy-2 and Zr-2.5Nb. The Zircaloy-2 billet cladding sleeves used for the enriched extrusions were produced from an 11-in. diam ingot reduced by three extrusions to a 2-1/4-in. OD by 1/4-in. thick wall tube. The first two extrusions were made at 650 °C and the final tubular shape was extruded at 750 °C.

The Zr-2.5Nb cladding sleeves were machined from 2-1/4-in. OD by 1/4-in. thick tube extruded from a 3-1/2-in. billet. The billet, supplied by Chalk River Nuclear Laboratories, was extruded into the tubular shape at 750 °C.

Extrusion Can

The outer diameter sleeve of the billet can was of seamless Cu-10Ni tubing. The tubing was cold swaged from 2-3/8-in. OD by 0.085-in. wall tube to 2.240-in. OD by 0.035-in. wall tube.

The original 2-3/8-in. OD tube was purchased in accordance to ASIM Specification B-111-62. The billet sleeves were electro-plated on the inner diameter surface with 0.001 to 0.002 in. of nickel. The purpose of the nickel plate was to provide a barrier to copper diffusion into the zirconium alloy clad during the high temperature extrusion. Areas of excessive copper diffusion are known to occur with pure copper at temperatures above 750 °C and cannot be removed by the usual zirconium alloy bright etching techniques.

The inner extrusion sleeves were Cu-10Ni tubes extruded and swaged to 0.350-in. OD by 0.018-in. wall. The extrusion end caps were machined from Cu-10Ni forged bar.

COMPONENT CLEANING AND ASSEMBLY

The U_3Si billets were first degreased in trichloroethylene to remove grease and oil and then dry blasted with glass bead to remove surface discoloration and foreign materials. The final step consisted of a deionized water rinse and dry. Attempts to find a chemical bright etching technique for cleaning these billets were not successful. The detailed U_3Si billet cleaning procedures are given in the Appendix.

The zirconium alloy and Cu-10Ni billet components were cleaned by bright etching. The can outer diameter sleeves were bright etched prior to electro-plating the inner diameter surface with nickel, and then given a deionized water rinse after plating. Detailed procedure used in the cleaning of the zirconium and Cu-10Ni billet components are given in the Appendix.

The Zircaloy-2 lead end filler pieces were welded into the cladding sleeves by electron beam welding. The tail end filler piece was not welded into the cladding sleeve. Electron beam welding was used to assemble the Cu-10Ni extrusion can,

including final sealing of the can. Details of the welding procedure, weld joint design, and leak testing of the enriched billets after welding are as outlined in the Appendix.

EXTRUSION EQUIPMENT AND TOOLING

All coextrusions were made on the 700-ton vertical hydraulic press operating at a 800 in./min ram closure speed and at 60 in./min maximum ram pressing speed. The 2-1/4-in. extrusion container was resistance heated and controlled at 500 °C. The 0.625-in. extrusion die, which had a 90° included angle cone entry, was preheated in place within the extrusion container.

All mandrels were made from AISI Type T-1 tool steel. A drawing showing these mandrels together with the extrusion die is presented in Figure 4. The mandrels were tapered from 0.205-in. diam at the top to 0.180-in. diam at the mandrel tip. The taper helps reduce the extrusion frictional force on the mandrel. The mandrels were not preheated prior to use as the preheat would contribute to overheating the mandrels during extrusion.

As can be seen in Figure 5, the mandrel was attached to the stem together with the follower block. This arrangement prevents the mandrel (and sacrificial mandrel sleeve) from contacting the billet until the ram closes for extrusion, thus minimizing the mandrel exposure time to the high temperature.

COEXTRUSION

The primary extrusion conditions for the 10 billets extruded in this program are given in Table 2. The billets were coated prior to preheating with a colloidal graphite lubricant and a lubricant consisting of graphite and copper fines. The extrusion tools were coated with a lubricant consisting of graphite suspension in oil.

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FIGURE 4. U_3Si Extrusion Die and Mandrel

FIGURE 5. Section of 2-1/4 in. Extrusion Tooling Showing Ram Closing on 2-1/4 U₃Si Coextrusion Billet and Graphite Cut-off

TABLE 2. Extrusion Conditions and Results for 2-1/4 in. U₃Si Billets

Billet No.	Si in Core, %	Extrusion Material	Cladding Thickness, in.	Billet Preheat, °C	Extrusion Force, tons		Extrusion Constant (k), tons/in. ²		Comments
					Starting	Running	Starting	Running	
516-M1T1	4.15	Zr-2.5Nb	0.025	843	350	270	32.4	25	
516-M1T2	4.15	Zr-2	0.025	856	350	275	32.4	25.5	
517-M1T1	4.15	Zr-2	0.015	843	300	240	27.8	22.2	
517-M1T2	4.15	Zr-2	0.025	843	295	240	27.3	22.2	Ribbed Extrusion
519-M1T1	3.7	Zr-2	0.025	856	300	260	27.8	24.1	
519-M1T2*	3.7	Zr-2.5Nb	<0.025	850	370	260	34.3	24.1	Water quenched out of die. Extrusion bowed.
519-M2T1	3.8	Zr-2	0.015	854	290	250	26.9	23.1	
519-M2T2	3.8	Zr-2.5Nb	>0.015	845	300	245	27.8	22.7	
2.95% En 522-B1T1	4.0	Zr-2	0.025	850	290	245	26.9	22.7	
2.95% En 523-B1T1	3.6	Zr-2	0.025	850	295	240	27.3	22.2	

* Billet 519-M1T2 recanned after first billet leaked.

Extrusion Conditions:

1. Extrusion reduction: 14.6/1 except billet 517-M1T2 was 10.4/1 (based on tooling dimensions)
2. Sacrificial mandrel sleeve: Cu-10%Ni (0.290 in. OD x 0.0218 in. ID); 2 in. of lead end heated in 800 °C graphite block for 30 sec.
3. Lubricant: Billet - Aqua dag with Fel Pro and graphite
Container - Oil dag
Die - Necrolene
Mandrel - Aqua dag and Fel Pro
4. Graphite cutoff: Preheated to 400 °C

The extrusion tooling, except for the extrusion mandrels which were not preheated prior to use was operated at 500 °C. The billets were heated in a closed stainless steel billet can. A graphite block was placed next to the lid of the can to help reduce the O_2 in the can and thus lessen the oxidation of the lubricants.

The coextrusions were extruded through a straightening and guide tube with a 3/4-in. hole. This tube, located beneath the die, straightened out the bows in the rod resulting from the extrusion and guided the extrusion into the extrusion catch tube. This 2-in. diam catch tube was partially filled with vermiculite to reduce the drop of the extrusions from the die and provide a cushioned stop. The straightening tube and cushioned catch tube proved beneficial in reducing bow in the coextrusions.

The extrusion forces, given in Table 2, were the maximum starting forces and the average continuing or "running" forces encountered for each extrusion. A typical extrusion force trace can be seen in Figure 6. The constant "K" is derived from the extrusion formula, $F = K \ln R$, where F is the unit extrusion pressure on the billet exerted directly by the ram, and R is the ratio of the area of the extrusion container less the area of the mandrel to the area of the extrusion.

The Zr-2.5Nb clad coextrusion was water-quenched in an effort to leave the cladding in a solution-quenched condition by filling the 2-in. catch tube with water and extruding directly into the water-filled tube. The coextrusion bowed considerably from this quench.

FIGURE 6. Typical Trace of Extrusion Force Versus Ram Travel for 2-1/4 in. U_3Si Coextrusions

EXTRUSION STRAIGHTENING

Work was initiated on hot stretch-straightening of the coextruded U_3Si rods. The Cu-10Ni extrusion jackets were left on and the rods were surrounded by vermiculite during the heating and straightening operation. A thermocouple was attached to the rods to monitor the temperature. The rods were resistance heated with a high current Reslec heater and pulled on a 50-ton hydraulic stretch straightener. One terminal from the resistance heater was attached to the center of the rod and the other terminal to the frame of the stretch straightener. This method of attaching the terminals was necessary as the stretch straightener rod grips could not be readily insulated from the frame of the stretch straightener.

Attempts to straighten the rods were made at several temperatures up through 700 °C. The 700 °C temperature produced the most satisfactory results. Below this temperature, the rod would not readily stretch and would break. Terminals attached to the center of the rod proved to be unsatisfactory inasmuch as a substantial length of rod on either side of the terminal would not heat sufficiently to straighten. The center of the rod thus would remain bowed. However, the portions of the rod between the terminals straightened very satisfactorily.

DISCUSSION

All eight of the coextrusions were successfully extruded with the small 0.200-in. coaxial void in the center of the rod. The 0.036-in. thick Cu-10Ni sacrificial mandrel sleeves protected the mandrel sufficiently to allow extrusion of the billets at the 60 in./min ram speed. However, examination of the center void surface of the first few extrusions indicated the sleeve and mandrel to have chilled the ID surface of the billet, especially on the lead end, sufficiently to cause some cracking of this surface during extrusion. This problem was overcome by preheating the first 2 to 3 in. of the sacrificial tube in a 800 °C block of graphite about 30 to 45 sec just prior to its use.

No noticeable difference was detected in coextruding U_3Si in Zr-2.5Nb, as compared to Zr-2. The hot extruded Zr-2.5Nb billet cladding sleeves do not provide a bright surface after etch cleaning as does Zr-2. The surface has a dull luster after the cleaning step. The attempt to solution quench the Zr-2.5Nb clad by quenching the hot extrusion immediately after the extrusion die was very detrimental to the U_3Si to cladding bond. The extrusion bowed considerably from the quench and, as can be seen in Figure 7, the U_3Si was cracked and the bond between the clad and the U_3Si was broken in many places. This result

Neg 468-5935

Neg 468-5936

Lead End of Coextrusion

Neg 468-5937

Neg 468-5938

Tail End of Coextrusion

FIGURE 7. Macros (3X) of Traverse and Longitudinal Sections from Billet No. 519-M1T2 Showing the Effect of Water Quenching Immediately After Extrusion on a Zr-2.5Nb Clad U_3Si Coextrusion

indicates the thermal shock of the water quench to be too severe for the U_3Si core and the brittle cladding bond. The photomicrograph of the bond line and the U_3Si microstructure taken from the nonfractured portions of samples from the lead and tail ends of the water quenched coextrusion can be seen in Figures 8 and 9, respectively. Bond lines shown in the samples appear dark because samples were electrolytically etched with phosphoric acid solution prior to photographing.

All the uranium-silicon alloys used in this program, consisting of 3.7%; 3.8%, and 4.15% silicon content, were successfully coextrusion-clad with both Zr-2 and Zr-2.5Nb. Producing a cladding thickness down to 0.015-in. on 0.600-in. rods appeared to involve no problem with either of these zirconium alloys. The cladding, uniform in thickness in all cases, resulted from the fairly fine grain texture within the cast and deltized U_3Si billets. The grain size is evident in Figures 8 and 9.

One billet was prepared as indicated in Figure 2 and extruded to determine the feasibility of producing integral spacer ribs on the clad U_3Si rods. A transverse section of the coextrusion is shown in Figure 10. While the results were encouraging, the cost of producing the shaped billet would no doubt exceed the cost of attaching ribs by the conventional welding techniques.

Further work will be required to determine the feasibility of the hot stretch straightening approach. While some encouraging results were obtained, any further effort would require the use of electrically insulated rod grips to allow introduction of the resistance heating current into the ends of the rods rather than into the center, as was the case in this program. Also a vertical stretch would be desirable to prevent the weight of the rod from introducing a bow.

Zr-2.5Nb

Neg 4685935 B

425X

Neg 4685935 C

200X

Lead End

Zr-2.5Nb

Neg 4685937 B

425X

Neg 4685937 C

200X

Tail End

FIGURE 8. Transverse Section from Lead End and Tail End of Coextrusion from Billet 519-M1T2 (3.7% Si) Showing Zr-2.5Nb to U_3Si Bond (left), and U_3Si Microstructure (right)

Zr-2.5Nb

Neg 4685936 B

425X Neg 4685936 C

Lead End

200X

Zr-2.5Nb

Neg 4685938 B

425X Neg 4685938 C

Tail End

200X

FIGURE 9. Longitudinal Sections from Lead End and Tail End of Coextrusion from Billet 519-M1T2 (3.7% Si) Showing Zr-2.5Nb to U₃Si Bond (left), and U₃Si Microstructure (right)

Neg 468-5940

FIGURE 10. Transverse Section of Zircaloy-2
U₃Si Coextrusion Showing Intergally
Extruded Longitudinal Spacer Ribs

An autoclave corrosion test was run on open ended samples of coextruded Zircaloy-2 clad U₃Si rod. Two alloys of uranium silicon were tested, U-3.6 wt% Si and U-4 wt% Si, by exposing the samples for 24 hr to 300 °C water. The results can be seen in Figure 11. Samples of the U-3.6 wt% Si alloy displayed split cladding, while the U-4 wt% Si samples showed no such damage. However, the corrosion rate at the bond between the U₃Si and the cladding was slightly greater than on the U₃Si surfaces. The U-3.6 wt% Si alloy core, found to have an excess of free uranium stringered from coextrusion, presented many small high rate corrosion routes into the cores. The corrosion product swelled the cores and burst the clad. The higher rate corrosion at the cladding to U₃Si bond line can be attributed to the poor corrosion resistant products, ZrSi₂ and uranium,

OPEN END CORROSION TEST OF COEXTRUDED, ZIRCALOY-2
CLAD U_3Si

(24 Hr in 300 ° Water)

3.6% Si

4.0% Si

Neg 568-5553

1X

FIGURE 11. Results from Autoclave Test Showing Corrosion Behavior of Open Ended Coextruded Fuel Rods. (Extrusions T486 and B486 were produced from billets with composite U_3Si cores giving ring effect at the ID)

formed in the bond when U_3Si and zirconium alloys are coextruded above 843 °C.* This test verified desirability of a higher silicon content to reduce the amount of free uranium and ensure a more corrosion resistant fuel.

* W. D. Wilkinson. "Uranium Corrosion and Alloys," Uranium Metallurgy. 1962, vol. II, p. 1116.

1969 U₃Si COEXTRUSION DEVELOPMENT
AND BILLET PREPARATION PROGRAM

OBJECTIVES

The purpose of the 1969 USAEC/AECL program was to evaluate the influence of several extrusion parameters and billet designs on the coextrusion of zirconium alloy clad U₃Si fuel rods with a small coaxial void. The coextrusion billets for these investigations were of 3-in. diam except those for enriched rod extrusions and the 2-1/4-in. as-cast U₃Si extrusions. All U₃Si billets (all U-3.8 wt% Si) were supplied by Eldorado Nuclear Limited with the desired heat treatment.

Current melting and casting methods limit the size of the uranium silicon alloy billets due to the unreasonably long deltizing heat treatment times required for the larger billets with their accompanying larger U₃Si₂ and free uranium particle size. Both consumable arc melting and shot casting were explored in this program as potential methods of producing an uranium silicon alloy with fine evenly dispersed U₃Si₂ and free uranium particles.

Following are the objectives for this program:

- Evaluate the effect of higher extrusion ratios.
- Explore the use of longer coextrusion billets.
- Determine the effect of different extrusion ram speeds.
- Develop billet end designs for reducing the extrusion peak forces and preventing "rattlesnake" breaking at the ends of the U₃Si core.
- Provide zirconium alloy clad U₃Si coextrusions for an experimental 18 rod cluster BLW fuel element.
- Extrude zirconium alloy clad enriched U₃Si rods for irradiation testing by Chalk River Nuclear Laboratories.
- Explore coextrusion of as-, cast uranium silicon alloy without the deltizing heat treatment.

Vacuum consumable electrode arc melt uranium silicon alloy and evaluate the resulting cast structure.

- Produce uranium silicon alloy shot by the spinning table method and examine the resulting shot microstructure.
- Evaluate methods of shot compaction and coextrusion.

In addition to these objectives, this program provided a comparison of the extrudeability of the larger 3-in. billet to the 2-1/4-in. billet used in the 1968 program.

SUMMARY

The 3-in. diam U_3Si coextrusion billets were extruded successfully on both the 700-ton vertical hydraulic press and the higher speed 1250-ton horizontal hydropress. The extrusion force constants for these 3-in. billets were found to be consistently lower, by about 25%, than the extrusion force constants obtained for the 2-1/4-in. coextrusion billets extruded in the 1968 program.

Three extrusion ratios (billet areas to extrusion areas of 15.7/1, 18.1/1, and 21/1) were evaluated in this program. These ratios did not produce excessively high extrusion forces nor appear to contribute to defects in the coextruded rod. The coextrusions did not break at the tail end of the U_3Si core in the higher reductions, but occurred regularly in the lower reductions. Billet lengths ranging from 7-3/16 to 10-3/16 in. were successfully extruded. No detectable difference in required extrusion forces was noted, and the mandrels performed satisfactorily with the longer billets when extrusion ram speeds of 60 in./min or greater were used.

Extrusion press ram speeds of 40, 60, and 120 in./min were evaluated in this program. The extrusion mandrel pulled apart during extrusion at the 40-in./min speed, and some light die washing was noted after a few extrusions at the 120-in./min

speed. No appreciable difference in extrusion forces could be detected between these speeds. The 60-in./min speed was the same speed used for the earlier 2-1/4-in. U_3Si billet coextrusions.

Four billet lead end designs were evaluated. The double chevron shape on the U_3Si cores (designs V-V or W in Figure 12) prevented the "rattlesnake" type breaking experienced in previous extrusions at the lead end of the U_3Si core. The starting extrusion forces were within reasonable limits. The U_3Si broke through the cladding on the concave core end design (design A), and the extrusion starting force appeared to be higher for the inverse double chevron design (design AA). Neither of two attempted billet tail end designs prevented "rattlesnaking" at the tail end of the U_3Si core. A design with a thicker flat Zr-2 disk, or a concave core end design with matching Zr-2 filler piece, may prevent this breaking.

Five 3-in. billets were extruded to provide fuel stock for use in fabricating a BLW fuel element. While the coextrusion process went very satisfactorily, subsequent evaluation of the rods indicate the presence of small defects in the U_3Si core immediately under the cladding to U_3Si interface. These defects, which varied in size and number between these extrusions, were found to be prevalent through all the 3-in. billet extrusions. The cause of these defects has not been determined. However, they are believed to originate from a combination of chilling of the U_3Si billet against the relatively cold dies and small inclusions within the U_3Si billets.

Due to lack of time and sufficient program funds, the enriched U_3Si coextrusions for irradiation testing were not produced, nor were the extrusion experiments on the as-cast (nondeltized) uranium silicon alloy billets performed.

FIGURE 12. Billet Lead End Designs Tested on 3-in. Diameter U₃Si Coextrusion Billets

An attempt was made to produce an uranium silicon alloy billet by vacuum consumable electrode melting. The electrode broke and plunged into the ingot after approximately 1/3 of the electrode had been consumed. Examination of the material produced in this attempt indicated no improvement in the microstructure of this melt over the microstructure of the electrode produced by vacuum induction melting.

Approximately 60 lb of uranium silicon shot was produced by the spinning disk method. The U_3Si_2 particle size varied from approximately 2 μ in the minus 30-mesh size to 6 μ in the plus 6-mesh size. Transformation heat treatment times of less than 4 hr should be possible with this fine particle size. No attempt was made to compact or coextrude the uranium silicon alloy shot due to lack of time and program funds.

EXTRUSION BILLET

Design

Four coextrusion lead end designs shown in Figure 12 were evaluated in this program. The object of these designs was to reduce the starting extrusion force required for the U_3Si coextrusions and prevent the "rattlesnake" type breaking which seems to occur as the lead end of the U_3Si core enters the die. Previous work with the 2-1/4-in. billets indicated that both of these problems might possibly be solved by leading the U_3Si core with a thicker Zircaloy-2 filler piece shaped to gradually feed the U_3Si into the die over 4 to 6 in. of the extrusion length. As the "rattlesnaking" and high force is thought to be aggravated by chilling of the billet, the use of Zircaloy-2 with its low thermal conductivity ($0.04 \text{ cal/cm}^2/\text{cm}/^\circ\text{C}/\text{sec}$) should help reduce the end heat loss and thus help prevent this breaking.

Both the chevron lead end designs V-V and W appeared to serve their purposes of consistently preventing "rattlesnake" breaking and maintaining reasonable starting forces as verified by the extrusion constants presented in Table 3. These designs proved successful on billets extruded at three different reductions. The starting force required for end design A was comparable to the chevron designs, but did not prevent breaking of the extrusion, as can be seen in Figure 13. One billet with the inverted chevron, design A-A, was tried and required a higher starting constant but no "rattlesnake" breakage resulted.

Neither of the two billet tail end designs used on the 3-in. U_3Si coextrusion billets (Figure 14) prevented the "rattlesnake" breaking which occurred at the tail ends of all except the two billets extruded at the higher reductions of 18/1 and 21/1. A high extrusion force spike, evident in the extrusion force trace in Figure 7 accompanied these breaks. Although neither design was successful in preventing the "rattlesnake" breaking, the flat disk, design T, resulted in less "rattlesnaking" than the convexed core end, design TM. Extrusions from billets with both tail end designs are shown in Figure 15. Apparently the same problems exist at the tail end as at the lead end of the billet requiring the heat loss of the core tail end to the colder 400 °C graphite cut-off be reduced. Also a gradual tapering out of the U_3Si core at this end with a correctly shaped billet end should help.

Figure 16 shows a view of the U_3Si core, zirconium alloy cladding sleeve, and both end filler pieces. The lead end is design W and the tail end is design TM. Figure 17 shows the assembled billet, cladding sleeve, and end filler pieces along with the billet can components. A complete description of each extruded 3-in. billet, including component materials, end designs, and dimensions of the U_3Si core and zirconium alloy sleeves are given in Table 4.

TABLE 3. Conditions and Results for 3-in. U₃Si Billet Coextrusion

Billet No.	Extrusion Material	Cladding Thickness, in.	Billet Temp, °C	Extrusion Reduction*	Ram Speed, in./min	Lead End Design	Extrusion Force, tons		Extrusion Constant (K), tons/in. ² Running	Extrusion Press Capacity in Tons
							Starting	Running		
556-3	Zr-Z	0.030	850	1 : 7/1	60	V-V	420	380	21	700
556-4	Zr-Z	0.030	850	1 : 7/1	60	A	410	380	20.5	700
557-5	Zr-Z	0.030	850	1 : 7/1	60	V-V	420	380	21	700
557-6	Zr-Z	0.030	850	1 : 1/1	60	A-A	480	410	22.8	700
5044-T1	Zr-Z	0.030	850	1 : 7/1	60	V-V	430	390	21.5	700
5044-T2	Zr-Z 5Nb	0.035	845	1 : 2/1	120	W	425	390	20.2	1250
5045-T2	Zr-Z	0.033	850	1 : 2/1	120	W	No Reducing			1250
5047-T1*	Zr-Z 5Nb	0.033	850	1 : 7/1	40	V-V	420	400	21	700
5047-T2	Zr-Z	0.035	845	1 : 2/1	120	W	440	400	21	1250
5048-T1	Zr-Z 5Nb	0.035	845	1 : 2/1	120	W	450	390	21.4	1250
5048-T2	Zr-Z	0.035	850	1 : 2/1	120	W	450	410	21.4	1250
5049-T1	Zr-Z	0.030	850	2/1	60	V-V	480	420	21.7	700
5050-T2	Zr-Z 5Nb	0.035	850	1 : 2/1	120	W	395	370	18.7	1250
5051-T2	Zr-Z	0.035	850	1 : 2/1	120	W	450	420	21.4	1250

* and/or Billet apart after 2/3 of billet was extruded.

** Extrusion Reduction - Container Area Less Mandrel Area/Extrusion Area.

NOTE: Graphite cut-offs preheated to 400 °C.

FIGURE 13. Lead Ends of Two U_3Si Coextrusions Showing Effectiveness of Filler Lead End Design W (lower) in Preventing Extrusion Breakage, Compared to Design A (top) (Both end designs are shown in Figure 12)

FIGURE 14 Billet Tail End Designs Tested on 3-in. Diameter U₃Si Coextrusion Billets.

FIGURE 15. Tail Ends of Two U_3Si Coextrusions Showing Effect of Two Zr-2 Tail End Filler Designs on Breaking. (Designs T and TM are shown in Figure 14)

FIGURE 16 View Showing U_3Si_4 Pilllet, Cladding Sleeve, and End Fillers for 3-in. Pilllet.

FIGURE 17 Assembled U_3Si Core, Cladding Sleeve, and End Fillers, Along with Cu-10Ni Can Components.

TABLE 4. 3-in. U₃Si Coextrusion Billet Description

Billet No	U ₃ Si Core		Ext usion Cladding		Billet End Design		OD Can		ID Can Sleeve		OD	
	OD, in.	Length, in.	Mat'l	OD, in.	ID, in.	Lead End	Tail End	Mat'l	Mat'l	OD/ID, in.	Can Length	in.
556-3	2.645		Zr-2	2.338	2.657	V-V	T	Copper	Copper	0.500/.430	8-3/16	
556-4	2.645	6 3/4	Zr-2	2.338	2.656	A	T	Copper	Copper	0.500/.430	8-3/16	
557-5	2.644	6	Zr-2	2.330	2.655	V-V	T	Copper	Copper	0.500/.430	7-3/16	
557-6	2.643	6 3/4	Zr-2	2.330	2.655	A-A	T	Copper	Copper	0.500/.430	8-3/16	
5044-T1	2.645	8	Zr-2	2.337	2.655	V-V	T	Copper	Copper	0.500/.430	9-3/16	
5044-T2	2.584	8	Zr-2.5Nb	2.330	2.600	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
5045-T2	2.593	9	Zr-2	2.330	2.624	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
5047-T1	2.593	8	Zr-2.5Nb	2.333	2.614	V-V	T	Copper	Cu-10Ni	0.500/.400	9-3/16	
5047-T2	2.535	8	Zr-2	2.390	2.600	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
5048-T1	2.534	8	Zr-2.5Nb	2.390	2.600	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
5043-T2	2.332	8	Zr-2	2.390	2.600	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
*5043-T1	2.059	7	Zr-2	2.390	2.657	V-V	T	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	
5050-T2	2.334	8	Zr-2.5Nb	2.383	2.600	W	TM	Copper	Cu-10Ni	0.500/.400	9-3/16	
5051-T2	2.534	8	Zr-2	2.390	2.600	W	TM	Cu-10Ni	Cu-10Ni	0.500/.400	9-3/16	

*No nickel plate on can sleeve ID surface

U₃Si Billet Cores

All the U₃Si billets for the 3-in. coextrusion billets were supplied by Eldorado Nuclear Limited, heat treated and machined to 2-13/16-in. diam. The nominal silicon content of the cast billets was 3.8%. The deltizing heat treatment, performed by Eldorado Nuclear, consisted of a one-week long 800 °C treatment with the billets sealed in steel cans with a zirconium getter. Machining to the final billet sizes and shapes was performed by BNW. The 1/2-in. diam center mandrel hole, unlike those in the 2-1/4 in. billets, was drilled with a carbide tool. Table 4 gives the end shapes machined onto each billet, the diameter and the overall length. Various sized surface defects on outer diameters of many of the billets appeared to be filled with a dark brittle substance. Three of the ultrasonically tested billets, 5045-T2, 5047-T2, and 5050-T2, had subsurface defects 1/8 in. or greater in size.

The density and silicon analysis of the U₃Si billets, along with the impurity analysis of a portion of the billets, are given in Tables 5 and 6.

Cladding Sleeves

The Zircaloy-2 cladding sleeves were machined from a tube extruded in two steps from a 11-in. outer diam billet meeting Zircaloy-2 cladding Specification HWS-8721. The billet was first extruded to a 7.4-in. OD by 2-in. ID tube and then extruded to the 3.13-in. OD by 2.28-in. ID tube. Both extrusions were made at 650 °C, with the final billet extruded within a copper can.

The Zr-2.5Nb sleeves, machined from 3-in. OD by 1/4-in. wall tubular extrusions, were extruded to this size from 4-in. OD bar at 750 °C. The 4-in. bar supplied by CRNL was extruded from an 8-in. OD billet meeting AECL Specification MET-90.

TABLE 5. Densities and Silicon Analyses of Heat Treated and Machined U₃Si Billets

Heat and Billet No.	Diam, in.	Length, in.	Weight, kg.	Density Calculated	Immersion, (g/cm ³)	Si Top	(w/o) Bottom	MET 94 Theoretical Density
556-1	2.807	9.999	15.695	15.49		3.85	3.77	15.50
556-2	2.851	9.999	16.190	15.49		-	-	
557-1	2.825	10.009	15.930	15.50		3.87	3.72	15.50
557-2	2.823	9.990	15.889	15.50		-	-	
5044-1	2.815	9.980	15.622	15.36		3.87	3.90	15.42
5044-2	2.860	10.011	16.278	15.45	-	3.86	-	15.44
5043-1	2.819	9.974	15.623	15.32	15.35	3.89	-	15.41
5045-2	2.837	9.971	15.875	15.36	15.40	3.93	-	15.37
5047-1	2.820	10.022	15.847	15.46	15.55	-	-	
5047-2	2.783	10.014	15.442	15.48	15.56	3.81	3.82	15.48
5048-1	2.779	10.029	15.522	15.46	15.58	3.81	3.74	15.52
5048-2	2.823	10.023	16.017	15.59	15.67	3.79	-	15.52
5049-1	2.791	10.022	15.499	15.44	15.48	3.88	3.82	15.45
5050-1	2.827	10.035	15.974	15.48	15.55	3.82	3.75	15.52
5051-2	2.782	10.020	15.426	15.49	15.49	3.90	3.79	15.45

TABLE 6. Met 94 Impurity Analyses of Natural U_3Si Extrusion Billets

<u>Impurity, ppm</u>	<u>556</u>	<u>557</u>	<u>5044</u>	<u>5045</u>	<u>5047</u>	<u>5048</u>
Al	200	150	220	280	150	140
B	0.25	0.24	0.25	0.20	0.22	0.13
Ca	<5	<5	<5	<5	<5	<5
Cd	0.4	0.3	<0.2	0.3	0.3	0.5
Cr	15	12	15	15	11	12
Cu	5	2	8	5	3	4
Mg	2	2	<1	2	1	1
Mn	2	1	<1	2	1	2
Mo	<0.5	<0.5	4	1.5	<0.5	<0.5
Ni	61	55	78	125	50	55
Fe	57	200	215	240	205	230
F	<5	<5	<5	<5	<5	<5
O ₂	300 300	310 360	330 390	470 480	240 240	260 260
N ₂	70 80	35 45	100 110	60 90	85 85	50 55
H ₂	1 2	4 3	2 2	2 2	2 3	1 2
C	100 90	110 120	120 120	50 70	110 110	70 90
Zr	680	750	400	230	300	300
Hf	17	-	8	5	-	

The Zr-2 and Zr-2.5Nb sleeves used for the BLW fuel stock extrusions were tested for inclusions and surface defects by both liquid penetrant and ultrasonics. All sleeves were sound and free from surface defects. Final dimensions of each billet sleeve are given in Table 4.

Extrusion Can

The OD sleeves for the 3-in. billets were produced from both copper and Cu-10Ni seamless tubing. The copper tubing was 3-in. OD by 0.065-in. wall purchased to ASIM Specification B-75-65. The Cu-10Ni tubing, 2-7/8-in. OD by 0.085-in. wall, was purchased to ASIM Specification B-111-62. Both types of tubing were plug drawn to an ID size of 2.900 in. and then machined to a 2.980-in. OD.

All ID surfaces of both types of OD can sleeves were coated with 0.0002 to 0.0005 in. of nickel plate by the Dalic Process* except that the can sleeve used for billet 5049-T1 remained unplated. Billet 5049-T1 was extruded at a 21/1 reduction and examined to determine to what degree if any the Cu-10Ni alloy diffused into the zirconium alloy at these extrusion conditions. The plating was found to be necessary on copper extrusion cans to prevent excessive copper diffusion into the zirconium alloy cladding when extruded at temperatures of 750 °C or above.

The inner can sleeves were also produced from both copper and Cu-10Ni seamless tubing. The copper inner sleeves, commercial soft-drawn, were from 1/2-in. OD by 0.032-in. wall tubing. The Cu-10Ni inner sleeves were 1/2 in. OD by 0.050-in. wall produced by extrusion and swaging. The extrusion end caps were machined from Cu-10Ni forged bar.

COMPONENT CLEANING AND ASSEMBLY

All 3-in. U_3Si coextrusion billet components were cleaned using the same procedure described previously for the 2-1/4-in. U_3Si coextrusion billet components.

Neither the lead or tail end filler pieces were welded into the cladding sleeves. Electron beam welding was used to assemble and seal the extrusion can. Details of the welding

* A commercial plating process of Sifco Metachemical, Inc.

procedure and weld joint are given in the Appendix. All billets were checked for leaks after welding as outlined in the Appendix.

EXTRUSION EQUIPMENT AND TOOLING

The 3-in. coextrusion billets were extruded on both the 700-ton vertical hydraulic press and the 1250-ton horizontal extrusion press. The 700-ton vertical press is the same one described under Extrusion Equipment for the 2-1/4-in. billets. The 1250-ton horizontal extrusion press is a Loewy-Hydropress (accumulator-water actuated) with a 59-in. stroke, a maximum closing speed of 840 in./min, and a maximum pressing speed of 480 in./min. Extrusion dies used in both presses had a 90° included angle entry cone. Details of the throat of an extrusion die are shown in Figure 18. The dies were preheated within the extrusion container on both presses.

The extrusion mandrels were made from AISI type T-1 tool steel and were attached to the follower block as shown in detail in Figure 18. The mandrels were tapered gradually from a 0.230-in. diam at the base to a 0.210-in. diam at the tip. This taper, by helping to reduce the extrusion drag on the mandrel, will result in less heating and tensile load on the mandrel.

A photograph of a mandrel and an extrusion die used for coextrusion on the 1250-ton extrusion press is shown in Figure 19.

COEXTRUSION

The billet and die lubrication used for the 3-in. billet coextrusions operation was the same as explained previously for the 2-1/4-in. billet extrusions. The billets were preheated to 850 °C in closed stainless steel cans. The same procedure employed for the 2-1/4-in. billets was used, except that the preheat furnace was brought more slowly to temperature with the billets charge&. The purpose of the slow preheat was to prevent the development of high thermal tensile stresses on the ID can tube which could break the tube or welds.

FIGURE 18. Mandrel and Extrusion Die Throat Showing Details and Dimensions.

FIGURE 19. Cone Die and Mandrel Used to Extrude 3-in. U₃Si Billets

The 700-ton vertical press extrusion container was operated at 500 °C, and the 1250-ton extrusion press container at 400 °C (maximum operating temperature). The mandrels were not preheated prior to use to reduce the chance of their overheating during extrusion. The dies were preheated in the container.

The rods were extruded through a 15/16-in. ID guide tube placed immediately behind the dies on both presses to help remove any bow in the as-extruded rod. The rods were extruded into a transite lined V-shaped trough during use of the 1250-ton horizontal press to maintain straightness in the rods during cooling. Difficulty was encountered in maintaining the rods straight during extrusion on the 700-ton vertical press when using the vertical catch tube.

The extrusion conditions, the resulting extrusion forces, and the corresponding extrusion constants are given in Table 3. The forces recorded are the starting and running forces. A typical extrusion force trace can be seen in Figure 20 for the 3-in. billets.

A Cu-10Ni sacrificial sleeve, designed to extrude with the billets was placed over the mandrels to help prevent the mandrels from overheating. The sleeves were 0.400-in. OD by 0.083-in. wall for the billets with the copper inner ID sleeves, and 0.370-in. OD by 0.068-in. wall for the billets with the Cu-10Ni inner ID sleeves. These mandrel sleeves were first preheated to 400 °C, and then about 3 in. of the lead end was preheated for 30-45 sec in a block of graphite at 800 °C. The initial attempt to preheat only the lead end of the sacrificial mandrel sleeve for 30-45 sec in the graphite block at 800 °C was found inadequate for preventing the chilling of the ID billet surface, particularly for those billets with the copper ID can sleeves. The results of such a chill can be seen in Figure 21. Protection provided by the mandrel sleeves was

FIGURE 20. Trace of Extrusion Force Versus Ram Travel for Coextrusion of 3-in. U_3Si Billet. 5044-T1.

Neg 4691038 B

4X

Cu-10Ni;
Sacrificial
TubeCu ID Can
Sleeve U_3Si

Neg 4691038 E

250X

FIGURE 21. Longitudinal Section Taken from Lead End of Coextrusion 557-5 Showing Cracking of ID Surface Caused by Excessive Chill from the Sacrificial Tube (Etchant was Murakami's reagent)

sufficient to enable all the mandrels, except the one used for Billet 5047-T1 (extruded at 40 in./min), to withstand the high billet temperatures. This mandrel pulled apart during extrusion after approximately 2/3 to 3/4 of the billet was extruded. In all cases, the T-1 tool steel mandrels were heated during extrusion to the point where a rehardening heat treatment was required before further use. Two mandrels were successfully used after rehardening.

The graphite cut-off blocks were heated to only 400 °C (less than billet temperatures) to reduce the possibility of overheating the base of the mandrels which are exposed to the hot billet for the longest time. This colder graphite block results in a high extrusion force peak at the end of the extrusion and no doubt contributes to the "rattlesnake" breaking at the tail end of the extrusion.

ALTERNATE METHODS OF U_3Si BILLET PREPARATION

Arc Melting

An arc cast ingot of U-3.8 wt.% Si was produced by vacuum consumable electrode melting. The electrode was produced by vacuum induction melting and was cast into segments approximately 18 in. long by 1.25 in. in diam. These were threaded and screwed together to form the completed 60-in. long electrode. The electrode was melted into a 3-in. diam water-cooled copper crucible using 25-26 V and 2800-3000 A. After approximately one-third of the electrode had been consumed, it broke and plunged into the ingot. Figure 22 shows photomicrographs of the as-cast ingot taken at intervals from the sidewall to the center of the ingot and the imbedded electrode.

The outer skin of the ingot showed at least one crack that could have resulted from a chilling effect of the water-cooled copper crucible. The full extent of this cracking was not investigated. The U_3Si_2 particle size, varying from 8 μ

FIGURE 22. Microstructures (250X) of As-Arc Cast U-3.8% Si Alloy
(White U_3Si_2 , Black Uranium, Swab Etched in Murakami Reagent)

or less at the ingot sidewall to over 30 μ in the ingot interior, indicated that well over 100 hr of deltizing would be required to complete transformation. The chilling effect of the imbedded electrode is noted in one photomicrograph along with the structure of the electrode. It can be concluded that, had the electrode not plunged into the ingot, larger U_3Si_2 particles would have been produced in this ingot.

Shot Casting

Approximately 60 lb of U-3.8 wt.% Si alloy shot were procured from the National Lead Company of Ohio. This material was the product of a 100-lb heat produced by vacuum induction melting in a BeO slurry-coated graphite crucible. The charge was heated to melt down in 30 min at a 250 μ furnace pressure. The furnace was backfilled at this point with helium to 1/2 atmosphere and, 35 min later, the heat was poured at 3000 °F. Just prior to bottom pouring, the heat was lanced with helium and the lance was then used as a tool to shear the 3/16-in. diam bottom pour plug. The molten metal was poured onto a disc rotating at 1000 rpm.

Figure 23 shows the chemical analysis of the major impurities, and Table 7 lists the minor impurities. All screen sizes show virtually identical chemical analyses except for increases in nitrogen from 100 ppm in the plus 6 mesh to 222 ppm in the minus 30 mesh. Approximately 50% of the particles are spherical in shape, while the balance are irregular or flat splats (Figure 24). Eighty percent of the shipped weight fell in the range of minus 60 to plus 20 mesh size. The U_3Si_2 particle size (Figure 23), which varied from approximately 2 μ in the minus 30 mesh to only 6 μ in the plus 6 mesh size, indicated transformation time requirements of less than 4 hr.

	469-1035 -30 Mesh	469-1034 -20+30 Mesh	469-1033 -10+20 Mesh	469-1031 -6+10 Mesh	469-1030 +6 Mesh
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Silico wt%	3.79	3.81	3.79	3.82	
Carbon, ppm	391	409	372	373	
Nitrogen, ppm	222	200	170	100	
Iron, ppm	266	236	229	228	
Screen Analysis %	7	5	60	20	8

FIGURE 23 Microstructures (250X) of As-Cast U-3.8 wt% Si Alloy Shot (White U_3Si_2 , Black Uranium, Swab Etched in Murakami Reagent)

TABLE 7. Minor Impurity Content (ppm) of Uranium-3.8 wt% Silicon Alloy Shot

Mesh	-6	-10	-20	-30
Size	+10	+20	+30	
Al	4	3	5	5
B	0.17	0.17	0.18	0.21
Be				
Bi	<1	<1	<1	<1
Cd	<0.2	<0.2	<0.02	<0.02
Cr	17	14	17	18
Cu	3	3	4	7
Mg	<4	<4	<4	15
Mn	10	11	12	12
Mo	<1	<1	<1	1
Ni	13	13	14	14
Pb	<1	<1	<1	<1
Sn	<1	<1	<1	<1
V	2	2	2	2
Zn	<10	<10	<10	<10
Zr	<20	<20	<20	<20

Neg 0692782

FIGURE 24 Uranium-3.8 wt% Silicon Alloy Shot Mounted for Metallography

DISCUSSION

Coextrusion Defects

A problem in the extrusion of the 3-in. billets not encountered in those of 2-1/4-in. size was the occurrence of small defects in the U_3Si core immediately under the cladding. These defects are quite evident in sections shown in Figures 25 and 26. Smaller defects below the bond line are shown in Figure 26. The defects, apparent in Figure 25, are very large and appear to be caused by chilling of the billet during coextrusion. The crack shown completely across the extrusion apparently resulted from continuation of a smaller crack by thermal stresses created during cooling of the extrusion. The defects in Figure 26 are, for the most part, below the bond line. They do not extend to the bond line and, therefore, may be caused in part by small inclusions such as UO_2 or other foreign material within the U_3Si billet core.

The under-cladding defects shown in all microphotographs are from rods extruded in copper billet cans with the exception of Extrusion 5094-T1. This extrusion in a Cu-10Ni billet can, had smaller defects in spite of its higher extrusion ratio. Table 8 shows inspection results of the BLW fuel extrusions indicating that, while the billets with Cu-10Ni billet can were not entirely free from defects, the billet with the copper can had more defects.

A gross break in coextrusion 5044-T1, is shown in Figure 27. The belief that this crack was caused by a crack existing in the U_3Si billet core prior to coextrusion was verified by the determination that surfaces of the break had parted and deformed during the extrusion process. This billet core was not checked by ultrasonics as the coextrusion was not considered for BLW fuel stock. A view of the U_3Si microstructure close to the break is shown in Figure 26.

Neg 4691037 B 4X
Coextrusion Section

Neg 4691037 C 75X Neg 4691037 E 250X

FIGURE 25. Longitudinal Section Near Tail End of Coextrusion 556-4 Showing Gross Cracking in U_3Si Beneath the Cladding

Neg 4691036 E

250X

Near Cladding Interface

Neg 4691036 F

250X

Near ID Surface

FIGURE 26. Longitudinal Section Showing U_3Si Structure in Extrusion 5044-T1 Near Break in Figure 27 (Etchant was Murakami's Reagent)

TABLE 8. U₃Si Under Cladding Defects in BLW Fuel Extrusion (From Inspection of 3/4-in. long longitudinal sections taken from the lead and tail ends of the extrusion)

	<u>Location</u>	<u>Number of Subinterface Defects in Section</u>	<u>Number of Interface Defects in Section</u>
5044 T-2	Lead	few	none
	Tail	few	few
5045 T-2	Lead	No samples examined	No samples examined
	Tail		
5047 T-2	Lead	none	one
	Tail	two	none
5048 T-1	Lead	several	few
	Tail	one	two
5048 T-2	Lead	few	none
	Tail	three	none
5050 T-2	Lead	few	few
	Tail	several	several
5051 T-2	Lead	none	none
	Tail	four	none

Few = less than 12

Several = more than 12

Subinterface Defects = those defects appearing immediately under the cladding but not extending into the U₃Si cladding interface.

Interface Defects = those defects under the cladding extending into U₃Si cladding interface

Neg 4691036 B 4X
Coextrusion Section

Neg 4691036 D 250X
Center of Break

FIGURE 27. Longitudinal Section of Coextrusion 5044-T1 Showing Gross Break Near Center of Coextrusion (Etchant was Murakami's reagent)

Figures 28, 29, and 30 are transverse sections from the lead end of billet 556-3 and the resulting extrusion. Inclusions are evident in both, and those as shown beneath the cladding in Figure 30 may be responsible for some of the under-cladding defects. No radial cracks were associated with these inclusions, probably because of the circumferential compressive action during extrusion.

Further study and testing will be required to determine and verify the cause of the extrusion defects and establish the necessary steps for their prevention.

Extrusion Reduction and Speed Variations

Three extrusion ratios were evaluated in this program, 15.7/1 (16.2/1 when using the 1250-ton press with the slightly larger container size), 18/1, and 21/1. These ratios did not appear to contribute to any defects within the extruded rod, to adversely affect the U_3Si -to-cladding bond, or to produce excessively high extrusion forces, as can be seen from an inspection of Table 3. A comparison of the U_3Si -to-cladding bond and U_3Si microstructure for the three extrusion ratios is given in Figures 31, 32, and 33. The under-cladding defects do not appear larger for the higher reduction and, in fact, may be smaller. Also, no "rattlesnake" breakage occurred at the tail ends of the rods extruded at 18/1 and 21/1.

Three speeds of 40 in./min, 60 in./min, and 120 in./min were investigated. No difficulties were encountered at the two higher speeds. However, pulling apart of the mandrel during extrusion at the 40-in./min speed indicates the slower speed to be so slow that the mandrel was allowed to overheat excessively during the extrusion. Effects of the different extrusion speeds on the under-cladding defect problem could not be compared because the slower speed extrusions were performed in a hotter extrusion container than the higher speed

Neg 4691042 A

250X

Near OD of Billet

Neg 4691042 C

250X

Center Wall of Billet

Neg 4691042 D

250X

Near ID of Billet

FIGURE 28. Transverse Section of U_3Si Billet 556-3 Taken from Lead End of Billet (Etchant was Murakami's reagent)

Neg 4691040 B

4X

Coextrusion Section

Neg 4691040 C

500X

Neg 4691040 D

500X

FIGURE 29. Transverse Section of Coextrusion 556-3 Near Lead End Showing U_3Si to Zr-2 Bond Layer

FIGURE 30. Transverse Section of Coextrusion 556-3 Taken from Lead End Showing U_3Si Structure Under Cladding and Center of U_3Si core (Etchant was Murakami's reagent)

FIGURE 31. Longitudinal Sections Showing Zr-2 to U₃Si Bonds for Three Coextrusions Extruded at Area Reduction Ratios of 15/1, 18/1, and 21/1

FIGURE 32. Longitudinal Sections of Three Zr-2 Clad U₃Si Coextrusions Showing U₃Si Microstructure Immediately Under Cladding (Extrusion area reductions are given above)

Extrusion
556-3
15/1
Reduction

Neg 4691039 C

250X

Extrusion
557-6
18/1
Reduction

Neg 4691041 D

250X

Extrusion
5049-T1
21/1
Reduction

Neg 4691089 D

250X

FIGURE 33. Longitudinal Sections from Three U₃Si Coextrusions Showing Microstructures Near Center of U₃Si Core (Extrusion area reductions are given above)

(120 in./min) extrusions. This temperature difference would tend to compensate for the increased billet chilling resulting from the slower extrusion speeds.

Extrusion Billets

The chevron shaped lead end designs (V-V and W) on the billets were found to function satisfactorily in preventing "rattlesnake" breakage and a high extrusion force peak when the U_3Si core enters the extrusion die. An effort should be made to simplify the designs for economic purposes. Figure 34 shows one design which is less involved and should result in lower cost fabrication. This design would require testing to determine the effect of the square end. The basic design used on the 2-1/4-in. billets, except with a thicker Zr-2 filler piece and a larger chamfer, is regarded as another possibility.

The suggested billet tail end design, shown in Figure 34, is merely a flat, disk-shaped Zr-2 filler piece of greater thickness. A U_3Si core with a concave shaped end and a matching Zr-2 filler piece also might be used. This design would help to gradually feed the U_3Si core out of the die as it complements the concavity produced as the billet passes through the conical die. Figure 35 illustrates the concave bowing produced in the extrusion when extruding a billet through a conical die.

The inner diameter surface of the Cu-10Ni outer diameter can sleeve was not plated with nickel on the billet extruded at the highest reduction. The Cu-10Ni can was etched off of 1-in. long samples taken from both the lead and tail end of the extrusion, and no diffusion of the can material into the Zr-2 cladding was evident. Thus, it would appear that the practice of nickel plating the inner diameter of the Cu-10Ni can sleeves is unnecessary. Further testing should be done to verify this.

FIGURE 34. Suggested U_3Si Billet Design
Based on Results of Design Tests.

FIGURE 35. Extrusion Effect on Straight Transverse Planes Within Billet Extruded Through Conical Die.

Extrusion Straightening

The use of the straightening guide tube behind the die and the V-shaped cooling trough used when extruding on the 1250-ton horizontal press was found to produce reasonably straight coextrusions. The resulting bow in the BLW fuel extrusions is given in Table 9. The bow was checked on successive 20-in. lengths along each BLW extrusion. As can be seen, the bow varies considerably along the extrusion and between extrusions. This bow could probably be improved by using a guide tube with an ID closer to the size of the extrusion, and by rotating the extrusion while it cools in the V-trough. It is doubtful that better than a 0.015 to 0.020-in. bow per 20-in. length could be obtained consistently in as-extruded rod.

TABLE 9. Bow in BLW U_3Si Fuel Rod Extrusions

<u>Billet Number</u>	<u>Single Throw Warp in 20-in. Lengths</u> <u>Quarters of the Extrusion</u>			
	<u>1st</u>	<u>2nd</u>	<u>3rd</u>	<u>4th</u>
5044 T-2	0.021	0.026	0.018	0.007
5045 T-2	0.013	0.010	0.018	0.013
5047 T-2	0.025	0.018	0.007	0.012
5048 T-1	0.022	0.010	0.015	0.014
5048 T-2	0.007	0.022	0.014	0.006
5050 T-2	0.005	0.008	0.008	0.017
5051 T-2	0.011	0.003	0.012	0.013

Shot Preparation

Previous work by Battelle-Northwest has shown that uranium shot can be canned and successfully hot extruded into bar with densities greater than 99% of theoretical. It is recommended that this method of consolidating the U-3.8 wt.% Si alloy be given serious thought. Deltizing of the shot can be performed

during the extrusion preheat. Efficient hot compaction of the shot is achieved during the early stages of extrusion through the use of protruding end plugs whose welds shear upon initial compaction (Figure 36). Clad fuel elements would then be fabricated by a subsequent coextrusion.

Arc Melting

Vacuum consumable electrode melting does not appear to produce an improved cast microstructure. Some improvement might be achieved by arc melting at from one-third to one atmosphere higher furnace pressures. At the higher furnace pressures, arc stability can be maintained with lower current densities, thus producing shallower molten pools and more rapid solidification. The lower limit of current density is then fixed by that required to move the molten metal to the crucible wall.

ACKNOWLEDGEMENT

The author wishes to acknowledge the assistance of R. G. Nelson for his evaluation of the alternate U_3Si billet preparation approaches described within this report.

FIGURE 36. Proposed Method of Consolidation and Extrusion of 3.8 wt% Si Alloy Shot

APPENDIX

U₃Si COEXTRUSION BILLET INSPECTION, CLEANING AND ASSEMBLY
PROCEDURESINTRODUCTION

Following are the requirements and procedures for non-destructive testing, cleaning, component inspection, welding, and weld testing of the U₃Si coextrusion billets. These requirements and procedures are taken from the program Process and Quality Control Specification A-1, Rev. 0.

NONDESTRUCTIVE TESTING OF ZIRCONIUM ALLOY SLEEVES

The zirconium alloy cladding sleeves used in the coextrusion billets to be extruded for the BLW fuel irradiation tests will be examined nondestructively by ultrasonic and liquid penetrant methods. The components shall be inspected for internal and surface defects using pulse-reflective ultrasonic techniques. The feed helix of the transducer shall be small enough to permit the signal from the reference notches to appear at least twice on successive revolutions of the tube. Any component producing an indication equal to or larger than the standard reference notches shall be considered for rejection.

The exterior and interior surfaces of the components shall be examined for cracks, laps, stringers, and linear indications by liquid penetrant. Any such indications on the surface of the cladding components shall be rejected.

Ultrasonic inspection shall be in accordance with ASIM Standard E 213-63T. Equipment will be calibrated as per paragraph 5 of ASIM Standard E 213-63T, using an artificial standard having the following defect sizes:

<u>Depth, in.</u>	<u>Length, in.</u>	<u>Width, in.</u>
0.007 ± 0.001	0.250 ± 0.010	Not to exceed 0.010

The liquid penetrant inspection will be in accordance with BNW-PR-1 (Liquid Penetrant Test Procedures).

ULTRASONIC TESTING OF U_3Si BILLETS

The billets shall be inspected for internal defects using a pulse-echo ultrasonic technique. The billet OD surface shall be scanned 100%, both in a longitudinal and circumferential direction. Any component producing an indication equal to or larger than the standard reference notch shall be considered for rejection.

The equipment shall be calibrated with an artificial standard having a defect size of 1/8-in. diam by 1/4-in. deep.

COEXTRUSION BILLET COMPONENT CLEANING

U_3Si Billet Cores

The U_3Si billet cores shall be cleaned prior to final component inspection and assembly as follows:

1. Remove any visible dirt and grease by washing the component in acetone.
2. Vapor degrease in trichloroethylene containing no more than 10% by volume mineral oil as determined by a boiling point above 190 °F.
3. Abrasive dry-blast clean, using glass bead if surface shows any discoloration or evidence of oxidation or foreign materials.
4. Rinse in deionized water (minimum quality of one megohm resistivity).
5. Rinse in absolute ethyl alcohol.
6. Air dry.
7. Visually inspect, and repeat all previous steps for any dirt, grease, scale, or oxidation found present.
8. Seal in plastic bag.

Zircaloy Component Cleaning

All Zr-2 components shall be cleaned prior to final inspection and assembly as follows:

1. Remove any visible dirt and grease by washing the component in acetone.
2. Vapor degrease in trichloroethylene containing no more than 10% by volume mineral oil as determined by a boiling point above 190 °F.
3. Abrasive dry-blast clean, using glass beads, if surface shows any discoloration or evidence of oxidation or foreign materials.
4. Rinse in deionized water (minimum quality of one megohm resistivity).
5. Immerse for 2 min (± 30 sec) in the following etch solution at room temperature:
 - 4 parts by volume 48% hydrofluoric acid,
 - 39 parts by volume 70% nitric acid, 57 parts by volume deionized water (resistivity one megohm or better).
6. Rinse in flowing deionized water for 5 min or more.
7. Rinse in absolute ethyl alcohol.
8. Air Dry.
9. Visually inspect, and repeat all previous steps for any dirt, grease, scale or oxidation found present.
10. Seal in plastic bag.

Zirconium-2.5 wt.% Niobium Alloy

All Zr-2.5 wt.% Nb alloy cladding sleeves will be cleaned prior to final inspection and assembly as follows:

1. Remove any visible dirt and grease by washing the component in acetone.
2. Vapor degrease in trichloroethylene containing no more than 10% by volume mineral oil as determined by a boiling point above 190 °F.
3. Abrasive dry-blast clean, using glass beads, if surface shows any discoloration or evidence of oxidation or foreign materials.

4. Rinse in deionized water (minimum quality of one megohm resistivity).
5. Immerse in the following etch solution at 50-55 °C for 15 to 20 seconds while stirring the bath continuously:
30% by volume deionized water, 30% by volume
95%-98% H_2SO_4 , 30% by volume 71% HNO_3 , 10% by volume
48% HF.
6. Immediately transfer into a 50% volume 71% HNO_3 solution at 40 °C for 3 to 5 sec.
7. Rinse in deionized running water for a minimum of 5 min.
8. Rinse in absolute ethyl alcohol
9. Air dry
10. Visually inspect, and repeat all previous steps for any oxide, grease, dirt, or scale found present (hot extruded or annealed Zr-2.5Nb material will display a dull smooth surface after cleaning).
11. Seal in a plastic bag.

Copper and Copper-Nickel Component Cleaning

The copper and copper-nickel billet can components, except those plated with nickel, will be cleaned prior to final inspection and assembly as follows:

1. Remove any visible dirt and grease by washing the components in acetone.
2. Vapor degrease in trichloroethylene containing no more than 10% by volume mineral oil as determined by a boiling point above 190 °F.
3. Abrasive dry-blast clean, using glass beads if surface shows any discoloration or evidence of oxidation or foreign materials.
4. Rinse in deionized water (minimum quality of one megohm resistivity).
5. Etch in Zinctone (a commercial bath by Turco Products, Inc.) at room temperature for 60 sec.

6. Rinse in process water for 5 min or more.
7. Rinse in absolute ethyl alcohol.
8. Air dry.
9. Visually inspect, and repeat all previous steps for any dirt, grease, scale, or oxidation found present.
10. Seal in plastic bag.

Nickel Plated Copper and Copper-Nickel Component Cleaning

The nickel plated copper and copper-nickel components will be plated and cleaned prior to final inspection and assembly as follows:

1. All components will be cleaned by the method outlined for copper and copper-nickel component cleaning prior to the plating step.
2. The nickel plate will be applied by the appropriate Dalic plating instructions.
3. Perform steps 1, 2, and 4 as prescribed for U_3Si billet core cleaning.
4. Rinse in absolute ethyl alcohol.
5. Air dry.
6. Visually inspect, and repeat steps 3 through 6 for any dirt, grease, scale, or oxidation found present.
7. Seal in plastic bag.

COMPONENT INSPECTION

All components will be handled with clean laundered gloves during inspection and resealed in plastic bags at the completion.

Visual Inspection

All components will be visually inspected to ensure that the components are clean and free of oxide, scale, grease, or dirt. The presence of any of these will require recleaning of the component in accordance with the appropriate cleaning procedure.

The cladding sleeves and U_3Si billets will be examined for cracks, scratches, pits, surface porosity, or other defects equal to or greater than the appropriate ultrasonic calibration standard defects. The can sleeves will be examined for all of the foregoing defects greater than 0.015 in. deep. Any of these defects will be recorded and will be cause for rejection.

Dimensional Inspection

All components will be inspected to ensure that the components are within the tolerances of the appropriate component drawings. The OD, ID, and length measurements for the U_3Si billet cores, cladding sleeves, and billet can sleeves will be recorded on appropriate inspection forms. Diameter and wall thicknesses shall be recorded to the nearest 0.001 in. and the lengths to the nearest 1/32 in.

The inspection shall be made at room temperature (65-75 °F) and the measurements shall be made as follows:

1. All OD measurements will be made with the appropriate size micrometer.
2. All ID measurements will be made with the appropriate size three-point plug micrometer.
3. Can wall thickness will be measured with a ball micrometer.
4. Length measurements will be made with a vernier caliper or scale.

BILLET ASSEMBLY

Billet Welding

The extrusion billet cans will be assembled and sealed by electron beam welding. The final seal weld shall be in a vacuum of at least 3×10^{-4} mm Hg. Component subassemblies and the final sealed billets shall be cooled to room temperature while still in the vacuum. The welding chamber may be back-filled with helium or argon to speed the cooling rate. The billet

must be in a vacuum of at least 3×10^{-4} mm Hg for a time of 30 min or more before starting the final closure weld. Following is a description of the weld joint design:

All components will be handled with clean laundered gloves during assembly. Should any components become soiled prior to or during assembly, the components must be recleaned according to the applicable cleaning procedure.

Weld Repairs

Defective or questionable welds may be repaired by electron beam welding. The welding will consist of a complete refusing of the entire weld joint. Any foreign materials in the weld joint must be removed by filing prior to the reweld. If the final closure weld has been made, the billet must remain in a vacuum of greater than 3×10^{-4} mm Hg for two hours prior to starting the repair weld. A maximum of three weld repairs will be allowed on a billet before requiring a complete billet disassembly and reassembly with new billet can components.

Weld Inspection

Visual Inspection

Each weld shall be visually examined for lack of fusion, cracks, voids, craters, porosity, and weld contamination. Each weld shall be first examined without magnification and then with 5X or 10X magnification. Any of the foregoing defects shall be repaired as described for weld repairs reexamined as described in this paragraph.

Helium Leak Check

Following the completion of a successful visual examination, the billet shall be leak-checked using the following procedure:

1. Place billet in a vacuum of at least 3×10^{-4} mm Hg for a minimum of 15 min.
2. Back-fill vacuum chamber with He and allow the billet to remain in the helium for at least 15 min.
3. Remove from chamber and place billet in leak detector chamber. The leak detector must be in a room separate from the helium back fill chamber.
4. Check leak detector with a sensitivity calibration standard having a calibrated helium leak rate of no more than $0.0036 \pm 10\% \mu$ per ft³/hr.
5. Perform leak check according to appropriate operating instructions for helium leak test machine. The billet shall be exposed to the vacuum, including pump down, for no more than one minute prior to commencing with the helium check. Should this time be exceeded in allowing the machine to reduce pressure to the prescribed leak check starting level, the billet shall be removed and the steps 1 through 5 repeated. The billets shall remain in the test for at least 15 min.
6. Recheck the machine sensitivity with the calibrated leak standard before removing the billets. Should the test indicate the billet to be leaking, an attempt shall be made to find the leak. Defective welds shall be repaired in accordance with instructions prescribed for weld repairs. Should the leak be through the billet can components, or cannot be located, disassemble and reassemble the billet using new billet can components. All welding repairs will be visually examined as specified for visual weld inspection and followed by the billet helium leak test as outlined in this paragraph.

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