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BONDING OF URANIUM AND ZIRCONIUM ALLOYS

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BONDING OF URANIUM AND ZIRCONIUM ALLOYS

by

H. O. McIntire
H. J. Hucek
G. K. Manning

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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	7
INTRODUCTION	7
EXPERIMENTAL WORK	8
CONCLUSIONS	22

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BONDING OF URANIUM AND ZIRCONIUM ALLOYS

by

H. O. McIntire, H. J. Hucek, and G. K. Manning

Fabricating conditions for bonding zirconium and uranium alloys by roll cladding were studied.

Good bonding was obtained with a minimum reduction of 3:1 for cores of unalloyed uranium, uranium-20 w/o zirconium, or uranium-50 w/o zirconium. As measured by a shear test, the bond was not obviously improved by increasing the amount of reduction to 7:1.

With unalloyed uranium cores, the optimum temperature for rolling appeared to be 1175 F to 1250 F. Moderately higher temperatures resulted in wavy or cracked cores. Uranium-20 w/o zirconium cores cracked when rolled at temperatures of 1175 F or lower. Good results were obtained for temperatures of 1250 F to 1450 F. Cores of uranium-50 w/o zirconium did not crack and appeared to bond well at temperatures between 1100 F and 1450°F. Temperatures outside this range were not evaluated. The best core configurations resulted with the lower reduction ratios. Also, the uranium-20 w/o zirconium cores gave better results than the unalloyed or uranium-50 w/o zirconium cores.

INTRODUCTION

Pure uranium or high-uranium zirconium alloys have been considered for cores of reactor fuel elements. The poor corrosion resistance of these materials introduces the problem of cladding them with more corrosion-resistant materials, such as zirconium or zirconium-tin alloys.

The conditions necessary to produce good bonding by roll cladding should be indicative of the conditions required for any other method using heat and pressure. R. J. Beaver* concluded from his experiments that uranium can be bonded to zirconium successfully by rolling at 1175 F with a surface increase of at least 15:1

This investigation was conducted to obtain additional information about the fabricating and rolling conditions required to produce uniformly good bonds between uranium or high-uranium alloys and zirconium or zirconium-tin alloys.

* TID-5084, Section I-A, page 53.

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-8-

EXPERIMENTAL WORK

Roll cladding was performed in the conventional way for plate-type fuel elements, except that the packs were not jacketed in steel. The core was fitted into a "picture frame" of the cladding material. Cover plates of the cladding material on both sides of the core and picture frame completed the pack.

All packs were made from 0.140-in. thick hot-rolled stock. Unalloyed Fernald uranium cores were used in most of the packs, but a few were made with Derby uranium-20 w/o zirconium or Derby uranium-50 w/o zirconium cores. Picture frames and covers used with the unalloyed cores were made from unalloyed WAPD Grade 1 zirconium and zirconium of the same type alloyed with 1.5 or 2.5 w/o tin. The alloyed cores were used in conjunction with zirconium-1.5 w/o tin picture frames and covers.

The sheet stock was sheared to the approximate size desired and then the picture frames and cores were machined to fit. Surfaces were ground to make them flat and free from defects. The cores were 2 in. square and the covers and picture frames were 3 in. square.

After machining and degreasing in acetone, the unalloyed uranium was pickled in 50 per cent nitric acid solution. The uranium alloys and cladding materials were degreased and pickled in 50 per cent nitric acid saturated with ammonium bifluoride. After pickling, all materials were rinsed in water, washed in hot Alconox solution, rinsed again in water, and air dried. The packs were assembled immediately after drying, using rubber gloves to avoid contamination of the surfaces. The assembled packs were welded around the edges with a tungsten arc. Welding was done in a tank which had been pumped down to 40 to 50 microns pressure and refilled with helium. The packs were heated for rolling in an electric muffle furnace having an argon atmosphere. Rolling was done on a two-high mill having 8.25-in.-diameter rolls.

After rolling, the packs were radiographed to determine the presence of any major defects such as cracks or unevenness of the core. The quality of the bond between the cladding and the core materials was evaluated by the following methods.

- (1) By trying to chisel the core and cladding apart.
- (2) By testing the strength of the bond in shear.
- (3) By metallographic examination of the cladding-core interface.

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-9-

The chisel test was used on all the packs rolled. In many instances it was possible to chisel the cladding from the core for packs in which shear tests showed good bond strength.

Shear tests were made in duplicate on most of the packs rolled and provided numerical values for the bond strength. A drawing of the specimen which was tested on a standard tensile testing machine is shown in Figure 1.

FIGURE 1. SHEAR-TEST SPECIMEN

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Specimens for metallographic examination were taken as follows:

- (a) across the core-picture frame interface on one end of the pack, (b) across the core-picture frame interface on one side of the pack and (c) at the middle of the pack.

The first 24 packs were given reductions of 2:1 or 3:1 at temperatures of 752 F, 932 F, 1112 F, 1292 F, 1472 F, and 1652 F. Picture frames and covers were unalloyed zirconium or zirconium-2.5 w/o tin. The cores were unalloyed uranium. This group of packs was rolled three passes, and then reheated and given two to five additional passes to obtain the final thickness desired. Much of the rolling obviously was done appreciably below the starting temperature.

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For rolling temperatures up to and including 1292 F, the cladding-to-core bond of these packs was intermittent if present at all. Packs rolled at 1472 F and 1652 F were bonded, but the cores were cracked. The cracked cores apparently resulted from rolling unalloyed uranium in the beta phase.

Except for the zone welded during assembling of the pack, there was no bonding between the cover and picture frame for packs rolled at temperatures up to 1292 F. In all instances, the cover-to-picture frame bond seemed to be good for packs rolled at 1472 F and 1652 F. There was no apparent difference in the bonding of unalloyed zirconium and of zirconium-2.5 w/o tin alloy.

Packs 25 to 59, inclusive, which also had unalloyed uranium cores, were reheated after each rolling pass. Most of these packs had zirconium-1.5 w/o tin cladding, but a few were clad with zirconium-2.5 w/o tin alloy. The rolling conditions and the results obtained for these packs are given in Table 1. The packs were given reductions ranging from 1-1/2:1 to 7:1 at rolling temperatures of 1175 F to 1472 F. Reductions in thickness of 0.005 to 0.015 in. were made on the first pass. In subsequent passes, reductions of about 0.035 in. per pass were made, until the desired total reduction had been obtained.

From Table 1, it may be seen that the chisel test indicated good bonding for all but one of the packs given reductions of 5:1 or more. With a reduction of 4:1, more than half the packs failed the chisel test, and all those rolled 3:1 failed this particular test. The shear tests, on the other hand, gave no indication that bonds obtained by a reduction of 3:1 were inferior to those obtained with higher reductions. Metallographic examination of the fractured areas showed that, in most instances, failure occurred in the cladding, rather than in the diffused layers. In a few specimens, failure occurred in the core material.

For most of the packs described in Table 1, shear specimens were also made from material which had been bent through 90 deg over 1/4-in. radius and then straightened. This was done to see whether bending this amount would break or weaken the bond between the core and the cladding. For the most part, the values obtained for the specimens which had been bent were similar to those tested as rolled. In one instance, Pack 50, which was rolled at 1250 F with a reduction of 3:1, the specimen broke during machining. This may indicate that the bond had been broken or weakened already. In the instances in Table 1 where it is indicated that there was no specimen available, the case was not one of premature failure, but that no attempt was made to make a specimen.

These data indicate that, for unalloyed uranium cores, satisfactory bonding can be obtained with rolling temperatures between 1175 F and 1300 F with reductions as low as 3:1. At lower rolling temperatures, bonding is

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- 11 -

6
less certain, and at higher temperatures, the cores become wavy or crack. Metallographic examination of the core-cladding interface showed a thin continuous diffusion zone. This diffusion zone increased somewhat in thickness with increasing rolling temperatures from 1175 F to 1300 F, but it was not altered significantly by the amount of reduction. Figures 2 and 3 show the diffusion zones obtained at 1175 F and 1250 F, respectively, with a reduction of 4:1.

Metallographic examination indicated that these rolling conditions produced only intermittent bonding between the cover plates and picture frames, except at the edges, which were welded prior to rolling. Bonding between the cover and the picture frame as the result of rolling was more complete on the ends of the packs than on the sides. The core-picture frame interface was only partially bonded in the best packs produced. The two thin, parallel, lines in the upper portion of Figure 4 indicate the poor bonding of cover to picture frame at the sides of the pack. The absence of these lines in the lower portion of Figure 4 indicates the improved bonding at the end of the pack.

In all instances, the ends of the unalloyed cores tended to thin down. This thinning was more pronounced as the rolling reduction increased, but a rolling temperature between 1175 F and 1300 F had little influence on the amount of thinning. Figure 5 shows the core end for Pack 50, which was rolled at 1250 F with 3:1 reduction. The end of this core was very slightly thinned. Figure 6 shows the more pronounced thinning which resulted with a 6:1 reduction. The sides of the cores in all instances were more uniform than the ends. The amount of convexity between the straight portions of the sides and the extreme tip of the end was determined from the radiographs. For the unalloyed cores, this convexity was not affected significantly by rolling temperature or amount of reduction within the range employed. The average convexity was about 0.1 in. The general uniformity of the cores was fairly good for rolling temperatures up to 1250 F. When rolled at 1300 F, a general wavy condition became apparent on the radiographs. It is probable that the unalloyed uranium core was slightly softer than the cladding material at these rolling temperatures.

Rolling conditions and results for packs having uranium-20 w/o zirconium alloy cores or uranium-50 w/o zirconium alloy cores are given in Table 2. The cladding for these packs was zirconium-1.5 w/o tin alloy. Temperatures of rolling represent the core structures (Table 3) as indicated by the uranium-zirconium constitutional diagram determined by Rough and Bauer (BMI-803).

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TABLE 1. ROLLING CONDITIONS AND RESULTS FOR

Pack	Cladding	Rolling Temp, F	Total Reduction	Number of Passes	Special Heat Treatment	Chisel Test, Core-To-Cover Bond
26	Zr-2.5 Sn	1175	7:1	11	None	Good
28	Ditto	1175	7:1	12	1700 F, 20 min - after first pass	-
34	Zr-1.5 Sn	1175	7:1	11	None	Good
47	Ditto	1300	7:1	11	"	-
33	Zr-1.5 Sn	1175	6:1	10	None	Good
38	Ditto	1175	6:1	10	"	Good
53	"	1250	6:1	11	"	Good
46	"	1300	6:1	10	"	Good
32	"	1175	5:1	9	"	Good
37	"	1175	5:1	9	"	Failed
52	"	1250	5:1	9	"	Good
45	"	1300	5:1	9	"	Good
31	"	1175	4:1	8	"	Failed
39	"	1200	4:1	8	"	Failed
59	"	1200	4:1	10	"	Good
40	"	1225	4:1	9	"	Failed
41	"	1250	4:1	8	"	Failed
51	"	1250	4:1	8	"	Good
58	"	1250	4:1	10	1700 F, 2 hr - after first pass	Good
42	"	1275	4:1	8	None	Failed
25	Zr-2.5 Sn	1175	3:1	7	None	Failed
27	Ditto	1175	3:1	8	1700 F, 20 min - after first pass	Failed
50	Zr-1.5 Sn	1250	3:1	7	None	Failed
57	Ditto	1250	3:1	8	1700 F, 2 hr - after first pass	Failed
43	"	1300	3:1	7	None	Failed
35	"	1475	3:1	6	"	Core cracked during rolling
49	"	1250	2:1	6	"	Failed
56	"	1250	2:1	6	1700 F, 2 hr - after first pass	Failed
36	"	1472	2:1	4	None	Core cracked during rolling
55	"	1250	1-1/2:1	5	1700 F, 2 hr - after first pass	Failed

(a) All specimens broke in zirconium, unless otherwise noted.

(b) Bent 90 deg over 1/4-inch radius, then straightened, machined, and tested; test area machined in center of bend.

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- 13 -

PACKS WITH UNALLOYED URANIUM CORES

Shear Strength of Core-To-Cover Bond, psi ^(a)		Metallographic Examination	
As Rolled	Bent 90 Deg ^(b)	Core-Cover Interface	Cladding-to- Cladding Interface
47,100	42,900	Thin diffusion zone	Bonded intermittently
54,700	50,500	Rather wide diffusion zone	Almost completely bonded
53,800	No specimen	Thin diffusion zone	Bonded intermittently
38,200	30,600	Thin diffusion zone	Bonded intermittently
44,300	46,800	Thin diffusion zone	Bonded intermittently
36,000	42,800	Thin diffusion zone	Bonded intermittently
53,800	44,700	Thin diffusion zone	Bonded intermittently
47,800	50,400	Rather wide diffusion zone	Almost completely bonded
53,150	55,900	Thin diffusion zone	Bonded intermittently
30,200	32,900	Very thin diffusion zone	Bonded intermittently
53,900	43,500	Thin diffusion zone	Bonded intermittently
48,400	57,400	Rather wide diffusion zone	Bonded intermittently
50,700	21,800 ^(c)	Very thin diffusion zone	No bonding
66,600 ^(d)	44,800	Thin diffusion zone	Bonded intermittently
42,500	No specimen	Thin diffusion zone	-
48,800	29,500	Thin diffusion zone	Bonded in spots
45,500	31,700	Wider diffusion zone than Pack 40	Bonded intermittently
44,600	42,500	Thin diffusion zone	Ditto
44,200	No specimen	Rather wide diffusion zone	-
53,800	31,400	Wide diffusion zone	Bonded intermittently
51,700	No specimen	Thin diffusion zone	No bond
65,500 ^(d)	47,900	Thin diffusion zone	Bonded intermittently
38,400 ^(c)	Broke in machining	Thin diffusion zone	No bond
50,900	No specimen	Thin diffusion zone	-
47,500	75,800	Rather wide diffusion zone	Bonded intermittently
No specimen	No specimen	-	Ditto
44,000	Broke in machining	No diffusion zone	No bond
Broke in machining	No specimen	Wide diffusion zone	-
No specimen	No specimen	-	Bonded intermittently
Broke in machining	No specimen	Intermittent spots of diffusion zone	-

(c) Broke at the interface.

(d) Broke in uranium.

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- 14 -

9

1000X

N6378

FIGURE 2. DIFFUSION ZONE BETWEEN ZIRCONIUM-1.5 W/O TIN (TOP) AND UNALLOYED URANIUM (BOTTOM), ROLLED AT 1175 F WITH 4:1 REDUCTION

Nitric acid + lactic acid + hydrofluoric acid etch

1000X

N6379

FIGURE 3. DIFFUSION ZONE BETWEEN ZIRCONIUM-1.5 W/O TIN (TOP) AND UNALLOYED URANIUM (BOTTOM), ROLLED AT 1250 F WITH 4:1 REDUCTION

Nitric acid + lactic acid + hydrochloric acid etch

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10

10X

N5886

FIGURE 4. CLADDING-TO-PICTURE FRAME BOND OBTAINED
AT 1250 F WITH 4:1 REDUCTION

Side of pack in upper view and end of pack in
bottom view; nitric acid + hydrochloric acid
+ hydrofluoric acid etch

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- 16 -

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TABLE 2. ROLLING CONDITIONS AND RESULTS FOR

Pack	Cladding	Cores	Rolling Temp, F	Total Reduction	Number of Passes
102	Zr-1.5 w/o Sn	U-20 w/o Zr	1100	7:1	11
104	Ditto	Ditto	1175	7:1	13
106	"	"	1250	7:1	10
108	"	"	1300	7:1	10
110	"	"	1450	7:1	11
111	"	"	1450	7:1	11
112	"	"	1450	7:1	11
114	"	U-50 w/o Zr	1100	7:1	14
116	"	Ditto	1150	7:1	13
118	"	"	1250	7:1	14
120	"	"	1350	7:1	12
122	"	"	1450	7:1	12
123	"	"	1450	7:1	12
101	"	U-20 w/o Zr	1100	3:1	8
103	"	Ditto	1175	3:1	7
105	"	"	1250	3:1	7
107	"	"	1300	3:1	9
109	"	"	1450	3:1	8
113	"	U-50 w/o Zr	1100	3:1	10
115	"	Ditto	1150	3:1	9
117	"	"	1250	3:1	9
119	"	"	1350	3:1	8
121	"	"	1450	3:1	8

(a) Broke specimens in machining.

(b) Specimens broke outside test area.

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- 17 -

12

PACKS WITH URANIUM-ZIRCONIUM ALLOY CORES

Chisel Test, Core-To-Cover Bond	Shear Strength Of Core-To-Cover Bond As Rolled, psi	Metallographic Examination, Cover-Core Interface
Core cracked during rolling		-
Ditto		-
Failed	46,800	Thin diffusion zone
Failed	49,800	Wider diffusion zone than Pack 106
Failed	45,600	Wider diffusion zone than Pack 108
Good	- (a)	Same diffusion zone as Pack 110
Failed	- (b)	Ditto
Failed	48,400	No visible diffusion zone
Good	43,200	Ditto
Failed	38,900	"
Good	46,800	"
Good	48,300	"
Failed	57,400	"
Core cracked during rolling		-
Failed	29,300	No visible diffusion zone
Failed	- (b)	Thin diffusion zone
Failed	- (b)	Wider diffusion zone than Pack 105
Failed	41,000	Wider diffusion zone than Pack 107
Failed	45,500	No diffusion zone
Failed	48,400	Ditto
Failed	62,200	"
Failed	46,800	"
Failed	51,100	"

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- 18 -

13

30X

N6444

FIGURE 5. END OF UNALLOYED URANIUM CORE ROLLED AT 1250 F WITH 3:1 REDUCTION (PACK 50)

End convexity, 0.12 in.

30X

N6445

FIGURE 6. END OF UNALLOYED URANIUM CORE ROLLED AT 1250 F WITH 6:1 REDUCTION (PACK 53)

End convexity, 0.06 in.

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TABLE 3. CORE STRUCTURE OF URANIUM-ZIRCONIUM ALLOYS

Temperature, F	Structure	
	Uranium-20 w/o Zirconium	Uranium-50 w/o Zirconium
1100	High-alpha + epsilon region	High-epsilon + sigma region
1150	-	Low-gamma + sigma region
1175	Low-alpha + gamma region	-
1250	High-alpha + gamma region	High-gamma + sigma region
1300	Gamma + gamma-prime region	-
1350	-	Low-gamma region
1450	Gamma region	High-gamma region

At each rolling temperature employed, one pack was reduced 3:1 and another pack was reduced 7:1. From Table 2, it is seen that the cores of Packs 101, 102, and 104 cracked during rolling. These packs had uranium-20 w/o zirconium cores. Cores in both packs rolled at 1100 F cracked, whereas, of the two packs rolled at 1175 F, only the core reduced 7:1 cracked.

Of the packs having uranium-20 w/o zirconium cores, only one could not be chiseled apart. The shear tests, however, indicated that the bond was at least as strong as the cladding material for the rolling conditions used. Where the specimens broke in machining, it is believed that they were mishandled, rather than that the bond was particularly weak. Thus, it appears that reductions as low as 3:1 and temperatures of 1175 F to 1450 F give satisfactory bonding for cores of uranium-20 w/o zirconium alloy.

The ends of the uranium-20 w/o zirconium cores were slightly thickened for rolling temperatures of 1250 F and 1350 F. Figures 7 and 8 show the ends of the cores for Packs 105 and Packs 106. These packs were rolled at 1250 F with reductions of 3:1 and 7:1, respectively. The cores of packs rolled at 1450 F had ends which were very slightly thinned. Apparently, thinning of the ends occurs when the hardness of the core is less than that of the cladding. Figures 9 and 10 show the ends of cores rolled at 1450 F with 3:1 and 7:1 reductions, respectively. A crack is apparent in the core rolled 7:1. The amount of convexity at the ends of the cores was appreciably less for the uranium-20 w/o zirconium cores than for the unalloyed cores. With 7:1 reduction, it was about 0.04 in., and for the three packs given a reduction of 3:1, it was about 0.03 in.

With uranium-50 w/o zirconium cores, 50 per cent of the packs rolled 7:1 could not be chiseled apart. Of those rolled 3:1, all of the packs could be chiseled apart. Shear-test values for packs rolled 3:1 were about the same as for those rolled 7:1.

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- 20 -

15

30X

N6441

FIGURE 7. URANIUM-20 W/O ZIRCONIUM CORE ROLLED AT 1250 F WITH 3:1 REDUCTION (PACK 105)

End convexity, 0.06 in.

30X

N6435

FIGURE 8. URANIUM-20 W/O ZIRCONIUM CORE ROLLED AT 1250 F WITH 7:1 REDUCTION (PACK 106)

End convexity, 0.06 in.

30X

N6436

FIGURE 9. URANIUM-20 W/O ZIRCONIUM CORE ROLLED AT 1450 F WITH 3:1 REDUCTION (PACK 109)

End convexity, 0.015 in.

30X

N6437

FIGURE 10. URANIUM-20 W/O ZIRCONIUM CORE ROLLED AT 1450 F WITH 7:1 REDUCTION (PACK 110)

End convexity, 0.008 in.

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16
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30X

N6443

FIGURE 11. URANIUM-50 W/O ZIRCONIUM CORE ROLLED AT 1100 F WITH 3:1 REDUCTION (PACK 113)
End convexity 0.05 in.

30X

N6439

FIGURE 12. URANIUM-50 W/O ZIRCONIUM CORE ROLLED AT 1450 F WITH 3:1 REDUCTION (PACK 121)
End convexity, 0.04 in.

30X

N6438

FIGURE 13. URANIUM-50 W/O ZIRCONIUM CORE ROLLED AT 1450 F WITH 7:1 REDUCTION
End convexity, 0.11 in.

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For the uranium-50 w/o zirconium cores, it appears that reductions as low as 3:1 at rolling temperatures of 1100 F to 1450 F will give satisfactory bonding between the cover and core.

Cores of uranium-50 w/o zirconium had thickened ends at rolling temperatures of 1100 F and 1150 F. Figure 11 shows the thickened core end for Pack 113, which was rolled 3:1 at 1100 F. Slightly thinned edges resulted at 1250 F, 1350 F, and 1450 F. Figures 12 and 13 represent the core end obtained with 3:1 and 7:1 reductions, respectively, at 1450 F. Convexity at the ends of cores rolled 7:1 was about 0.09 in. and, for those rolled 3:1, it was about 0.04 in. Here, also, the temperature of rolling had no significant influence on the amount of convexity.

CONCLUSIONS

From the data presented in this report, the following conclusions are evident.

1. For unalloyed uranium cores, the best rolling temperatures are between 1175 F and 1250 F. Lower temperatures give inadequate bonding, and moderately higher temperatures result in cracked cores. Apparently good core-cladding bonding results with reductions as low as 3:1 when rolled in this temperature range.
2. For uranium-20 w/o zirconium cores, rolling temperatures between 1175 F and 1450 F with a minimum reduction of 3:1 appear to give good bonding. Cracking of the core may occur at lower rolling temperatures.
3. For uranium-50 w/o zirconium cores, apparently good bonding is obtained for rolling temperatures between 1100 F and 1450 F with a minimum reduction of 3:1.
4. The best core geometry is obtained with low reductions. Uranium-20 w/o zirconium alloy is better in this respect than unalloyed uranium or uranium-50 w/o zirconium alloy.

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