MASTER

EFFECT OF SURFACE TREATMENT ON THE CORROSION RESISTANCE OF ZIRCALOY-2

APRIL 1961

CONTRACT AT-11-1-GEN-14

BETTIS ATOMIC POWER LABORATORY, PITTSBURGH, PA.,
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April 1961

Contract AT-11-1-GEN-14

Price \$.50

**Available from the Office of Technical Services,
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An experiment was performed to determine the effects of nonpickling versus pickling to depths of one and two mils on the corrosion resistance of Zircaloy-2 machined by various methods. No significant difference in corrosion rate between pickled and unpickled Zircaloy-2 was found, provided that properly sharpened and hardened tools were used.

EFFECT OF SURFACE TREATMENT ON THE CORROSION RESISTANCE OF ZIRCALOY-2

D.B.Scott

I. INTRODUCTION

A previous investigation of Goldman (Ref 1) demonstrated that the surface layers of metal on zirconium are distorted by machining operations and that the distorted metal corrodes more rapidly in high temperature water and steam than the base metal. In addition, these surface layers could be removed by bright etching in an aqueous solution of nitric and hydrofluoric acid. Although the distorted surface layers of zirconium and Zircaloy-2 were approximately equal in thickness, the corrosion behavior of the layers was markedly different—since the zirconium surfaces corroded rapidly, whereas the Zircaloy-2 surfaces exhibited good corrosion resistance (Ref 1). Later work at Bettis Laboratory (Ref 2)* provided additional qualitative data demonstrating the superior corrosion resistance of properly machined Zircaloy-2 surfaces.

The scope of the experiment reported here is an extension of the earlier investigation and defines the steam and aqueous corrosion resistance

of pickled and unpickled Zircaloy-2 as a function of the surface finish provided by various machining processes and tools.

II. EXPERIMENTAL PROCEDURE

A. Machined Surfaces

To obtain samples of different machined surfaces, Zircaloy-2 coupons, 0.1 x 0.5 x 1.5 inches in size, were machined with eight types of machine tools. Strips, previously machined to 0.120-inch thickness from one section of an atmosphere melted, reactor grade ingot, were used as the source stock. The machine tools used on the coupons were:

- 1) Shaper
- 2) Slab Mill
- 3) Straddle Mill
- 4) Shell Mill (Facing Mill)
- 5) End Mill with overlap cut
- 6) End Mill with non-overlap cut
- 7) End Mill with edge cut
- 8) Surface Grinder (wheel)

Coupons were prepared using both sharp and dull cutters with fine, medium, and coarse feeds.

*Also, J.C. Helbling and S. Kass, Bettis Laboratory Personal Communication (1958).

The surface-ground specimens were exceptions since only a new wheel with fine, medium, and coarse feeds could be used.

For the purpose of this experiment, a sharp tool is defined as one newly sharpened, a dull tool as one ready to be sharpened. In all machining operations with the exception of surface grinding no lubricant was used. Because of the nature of the cutting action, surface grinding required the liberal use of coolant. The machining record is given in Table I.

B. Corrosion Test Preparation

Coupons from each surface treatment were divided into three lots. Lot 1 was degreased by scrubbing with acetone and alcohol; lots 2 and 3 were pickled with an aqueous solution of HF and HNO_3 to remove 1 and 2 mils per surface, respectively. All coupons were corrosion tested in 680°F water or 750°F steam.

III. RESULTS

A. Unpickled Machined Coupons

The weight gains of the coupons after the first 14 days in 750°F steam and 28 days in 680°F water are shown in Tables II and III. With the exception of the straddle-milled coupons, the weight gains in both 750°F steam and 680°F water were normal, and all coupons exhibited black oxide films with white stringers—a normal condition for corrosion-tested, atmosphere-melted Zircaloy-2. One flat surface of the straddle-milled coupons was white, while the other flat surface exhibited a black temper film with stringers (see Figure 1). The white corrosion film, attributed to a defective tool, is discussed later.

The fine, medium, and coarse variations in feed were found to be insignificant in their effects on corrosion resistance, as verified by the Bettis Statistical Engineering staff (Appendix I). Since no changes in corrosion behavior were noted

throughout the corrosion tests, the results for the particular tools were averaged regardless of feed and are tabulated in Tables IV and VII for the times in test.

As the tests continued, the effect of the "sharp" and "dull" tools was noticeable on the face-milled coupons (shell mill) after 84 days in 750°F steam and 112 days in 680°F water. At these times, erratic weight gains (shown in Tables IV and VII) were indicative of poor corrosion resistance for the coupons milled with a dull cutter. The poor corrosion behavior of the one face of the straddle milled coupons (white corrosion film) continued for the duration of the test. Except for the dull cut face-milled and straddle-milled coupons, all other machined coupons including the surface-ground specimens had slightly lower than normal weight gains and black-gray temper films with stringers throughout the test (140 days in 750°F steam and 224 days in 680°F water).

B. Pickled Machined Coupons

The weight gains for the first 14 days in 750°F steam and 28 days in 680°F water are shown in Tables II and III. All machined coupons, without exception, exhibited shiny black temper films with stringers. No evidence of white corrosion product was noted on the straddle-milled (Figure 2) and slab-milled coupons.

As with the unpickled coupons of this experiment, variations in feed produced insignificant effects for the duration of test. The results are shown in Tables V, VI, VIII and IX. Comparison bar graphs for 84 and 140 days are shown in Figures 3 and 4.

The weight gains in 680°F water showed that all pickle treatments gave results within the normal experimental scatter, as shown in the bar graphs for 112 and 196 days in test (Figures 5 and 6).

TABLE I
MACHINING RECORD ON MACHINING ZIRCALOY-2 CORROSION COUPONS

Shaper		Feed (in./stroke)	Speed (strokes/min)	Finish (μ in. rms)	
Sharp Tool:	Fine Feed	0.004	12	40	
	Medium Feed	0.010	12	70	
	Coarse Feed	0.015	12	125	
Dull Tool:	Fine Feed	0.004	12	45	
	Medium Feed	0.010	12	70	
	Coarse Feed	0.020	15	125	
Slab Mill		Feed (in./min)	Speed(rpm)	Diameter of Cutter	Finish
Sharp Tool:	Fine Feed	5/8	33	6 in. x 1 in. (22 teeth)	35
	Medium Feed	1 1/4	33	6 in. x 1 in. (22 teeth)	60
	Coarse Feed	1 3/4	25	6 in. x 1 in. (22 teeth)	110
Dull Tool:	Fine Feed	5/8	33	6 in. x 1 in. (22 teeth)	30
	Medium Feed	7/8	25	6 in. x 1 in. (22 teeth)	60
	Coarse Feed	1 3/4	25	6 in. x 1 in. (22 teeth)	125
Straddle Mill		Feed (in./min)	Speed(rpm)	Diameter of Cutter	Finish
Sharp Tool:	Fine Feed	5/16	18	6 in.	250
	Medium Feed	1	18	6 in.	140
	Coarse Feed	2 1/2	18	6 in.	130
Dull Tool:	Fine Feed	5/16	18	6 in.	120
	Medium Feed	1	18	6 in.	90
	Coarse Feed	2 1/2	18	6 in.	90
Shell Mill		Feed (in./min)	Speed(rpm)	Diameter of Shell Mill	Finish
Sharp Tool:	Fine Feed	5/8	43	3 in. (12 flute)	35
	Medium Feed	1 1/4	43	3 in. (12 flute)	60
	Coarse Feed	6 1/8	43	3 in. (12 flute)	110
Dull Tool:	Fine Feed	5/8	56	3 in. (12 flute)	50
	Medium Feed	1 1/4	43	3 in. (12 flute)	70
	Coarse Feed	6 1/8	25	3 in. (12 flute)	100
End Mill Overlap Facing		Feed (in./min)	Speed(rpm)	Diameter of End Mill	Finish
Sharp Tool:	Fine Feed	7/8	168	5/16 in. (4 flute)	40
	Medium Feed	2 3/8	168	5/16 in. (4 flute)	60
	Coarse Feed	3 1/4	168	5/16 in. (4 flute)	90
Dull Tool:	Fine Feed	5/8	225	6/16 in. (4 flute)	40
	Medium Feed	1 3/4	168	5/16 in. (4 flute)	70
	Coarse Feed	3 1/4	168	5/16 in. (4 flute)	110
End Mill (No Overlap Facing)		Feed (in./min)	Speed(rpm)	Diameter of End Mill	Finish
Sharp Cutter:	Fine Feed	5/16	100	1 1/2 in. (6 flute)	35
	Medium Feed	7/16	76	1 1/2 in. (6 flute)	70
	Coarse Feed	7/8	76	1 1/2 in. (6 flute)	90
Dull Cutter:	Fine Feed	5/16	100	1 1/2 in. (6 flute)	35
	Medium Feed	7/8	100	1 1/2 in. (6 flute)	60
	Coarse Feed	1 1/4	76	1 1/2 in. (6 flute)	110
End Mill (Edge Cut)		Feed (in./min)	Speed(rpm)	Diameter of End Mill	Finish
Sharp Cutter:	Fine Feed	1 1/4	76	1 1/4 in. (6 flute)	40
	Medium Feed	1 3/4	56	1 1/4 in. (6 flute)	60
	Coarse Feed	2 3/8	56	1 1/4 in. (6 flute)	110
Dull Cutter:	Fine Feed	7/8	76	1 1/4 in. (6 flute)	35
	Medium Feed	1 3/4	76	1 1/4 in. (6 flute)	70
	Coarse Feed	1 3/4	56	1 1/4 in. (6 flute)	125
Surface Grinder		Feed (in./min)	Speed(rpm)	Diameter of Wheel	Finish
New Wheel:	Fine Feed	0.001	6700	10 in. x 60-Grain Crystolon	11
	Medium Feed	0.0015	6700	10 in. x 60-Grain Crystolon	18
	Coarse Feed	0.003	6700	10 in. x 60-Grain Crystolon	30

TABLE II
WEIGHT GAINS IN MG/DM² OF ZIRCALLOY-2 CORROSION TESTED FOR
14 DAYS IN 750°F STEAM

Shaper		No Pickle	Pickled 1 Mil per Surface	Pickled 2 Mils per Surface
Sharp Tool:	Fine Feed	25	27	26
	Medium Feed	24	27	27
	Coarse Feed	24	27	26
Dull Tool:	Fine Feed	24	27	26
	Medium Feed	24	27	26
	Coarse Feed	23	28	28
Slab Mill				
Sharp Tool:	Fine Feed	24	27	26
	Medium Feed	25	26	26
	Coarse Feed	25	27	24
Dull Tool:	Fine Feed	28	28	26
	Medium Feed	24	27	27
	Coarse Feed	26	29	28
Straddle Mill				
Sharp Tool:	Fine Feed	19	26	26
	Medium Feed	0	26	26
	Coarse Feed	2	26	25
Dull Tool:	Fine Feed	19	25	26
	Medium Feed	24	27	25
	Coarse Feed	20	28	27
Shell Mill				
Sharp Tool:	Fine Feed	25	24	26
	Medium Feed	25	27	26
	Coarse Feed	24	27	26
Dull Tool:	Fine Feed	29	27	27
	Medium Feed	31	28	27
	Coarse Feed	28	27	27
End Mill (Overlap Facing)				
Sharp Cutter:	Fine Feed	25	27	27
	Medium Feed	23	27	27
	Coarse Feed	24	28	27
Dull Cutter:	Fine Feed	24	28	28
	Medium Feed	24	28	26
	Coarse Feed	25	28	27
End Mill (No Overlap Facing)				
Sharp Cutter:	Fine Feed	25	27	25
	Medium Feed	25	26	27
	Coarse Feed	25	27	26
Dull Cutter:	Fine Feed	27	28	25
	Medium Feed	29	28	26
	Coarse Feed	25	29	28

TABLE II (Cont.)

WEIGHT GAINS IN MG/DM² OF ZIRCALOY-2 CORROSION TESTED FOR
14 DAYS IN 750°F STEAM

End Mill (Edge Cut)		No Pickle	Pickled 1 Mil per Surface	Pickled 2 Mils per Surface
Sharp Cutter:	Fine Feed	24	26	27
	Medium Feed	24	27	26
	Coarse Feed	24	28	26
Dull Cutter:	Fine Feed	25	29	27
	Medium Feed	24	28	27
	Coarse Feed	25	29	26
Surface Grind				
New Wheel:	Fine Feed	26	27	30
	Medium Feed	27	28	28
	Coarse Feed	27	28	27

TABLE III

WEIGHT GAINS IN MG/DM² OF ZIRCALOY-2 CORROSION TESTED FOR
28 DAYS IN 680°F WATER

Shaper		No Pickle	Pickled 1 Mil per Surface	Pickled 2 Mils per Surface
Sharp Tool:	Fine Feed	18	22	22
	Medium Feed	17	22	23
	Coarse Feed	18	22	22
Dull Tool:	Fine Feed	16	19	21
	Medium Feed	15	21	22
	Coarse Feed	15	22	21
Slab Mill				
Sharp Tool:	Fine Feed	16	21	22
	Medium Feed	16	21	21
	Coarse Feed	16	21	21
Dull Tool:	Fine Feed	17	20	21
	Medium Feed	17	21	21
	Coarse Feed	18	21	21
Straddle Mill				
Sharp Tool:	Fine Feed	4	20	21
	Medium Feed	2	19	21
	Coarse Feed	-7	21	22
Dull Tool:	Fine Feed	9	21	21
	Medium Feed	13	22	21
	Coarse Feed	12	21	22
Shell Mill				
Sharp Tool:	Fine Feed	17	21	21
	Medium Feed	16	22	21
	Coarse Feed	16	21	21
Dull Tool:	Fine Feed	18	20	20
	Medium Feed	21	21	21
	Coarse Feed	18	22	21

TABLE III (Cont.)

WEIGHT GAINS IN MG/DM² OF ZIRCALOY-2 CORROSION TESTED FOR
28 DAYS IN 680°F WATER

End Mill (Overlap Facing)		No Pickle	Pickled 1 mil per Surface	Pickled 2 mils per Surface
Sharp Cutter:	Fine Feed	17	22	22
	Medium Feed	16	22	22
	Coarse Feed	16	21	21
Dull Cutter:	Fine Feed	16	22	22
	Medium Feed	17	21	22
	Coarse Feed	16	22	22
End Mill (No Overlap Facing)				
Sharp Cutter:	Fine Feed	18	22	22
	Medium Feed	18	21	21
	Coarse Feed	17	21	20
Dull Cutter:	Fine Feed	18	21	20
	Medium Feed	16	22	22
	Coarse Feed	16	21	21
End Mill (Edge Cut)				
Sharp Cutter:	Fine Feed	18	21	21
	Medium Feed	17	21	22
	Coarse Feed	17	23	23
Dull Cutter:	Fine Feed	17	23	23
	Medium Feed	17	23	22
	Coarse Feed	17	22	23
Surface Grind				
New Wheel:	Fine Feed	18	22	23
	Medium Feed	16	22	21
	Coarse Feed	15	22	22

IV. DISCUSSION

A. Unpickled Machined Coupons

Goldman (Ref 1) showed that unpickled Zircaloy-2 machined on a shaper is corrosion resistant in short-term tests in 680°F water. The enlarged scope of this experiment shows that all the surface treatments investigated produce corrosion resistant surfaces if the machining is properly performed. No real or significant differences in results for the various types of machining were noted even though cutting actions were different.

The effect of the sharpness or dullness of tools was significant, however. The peculiar behavior of the straddle-milled samples was traced directly

to a cutter improperly heat treated to $R_c 55$ instead of the desired $R_c 63$. Since the cutter was soft, a smearing action rather than a cutting action was encountered. This adversely affected the corrosion resistance of the Zircaloy-2 coupons involved. Except for the coupons machined with this tool, all coupons machined with sharp cutters exhibited good corrosion resistance. The corrosion behavior of the coupons machined with dull tools depended upon the characteristics of the cutting tool. The rotating dull cutter of the shell mill caused poor corrosion behavior, apparently because of the vigorous smearing action inherent with this type of cutter combined with the dullness of the tool. The corrosion behavior of unpickled Zircaloy-2 machined with a sharp face

Figure 1 *As-Machined Zircaloy-2 Coupons Machined on a Horizontal Straddle Mill and Corrosion Tested for 14 Days in 750°F Steam.*

mill (shell mill) tool was comparable to that of the other coupons machined with sharp cutters.

The effect of surface grinding differed from previously reported results. The coupons in this experiment were black-gray and produced normal weight gains; coupons from earlier experiments were white and flaked within two weeks. The type of wheel and quantity of lubricant used are believed to be important factors in enhancing the corrosion resistance of these surfaces. Dry grinding with silicon carbide, and wet or dry grinding with alumina wheels produced distorted and burned surfaces. Although no direct data are available on the effect of grinding Zircaloy-2 with and without a lubricant, previous work on 18/8 chrome nickel stainless steel revealed that dry grinding causes an oxide film and smeared metal surface (Ref 3). Wet grinding eliminates the temperature effect causing the oxide film and allows a clean cutting action rather than the smearing action mentioned.

Since zirconium is a "getter" and well known for its ability to absorb oxygen, it is reasonable to assume that dry grinding will cause the Zircaloy-2 surface to oxidize and will impair its corrosion resistance.* Apparently, a silicon carbide wheel with the lubricant flooding the workpiece is a necessary condition to prevent oxidation and optimize the grinding operation. In the current experiment, a water coolant was used; however, further experimental work is necessary to determine the effects of a mineral oil coolant.

No statistical difference was noted in the effects of the feeds on corrosion behavior.

B. Pickled Machined Coupons

No visual difference was noted between machined coupons pickled 1 mil per surface and coupons

*L. S. Rubenstein, J.G. Goodwin, and F. L. Shubert, from work on the effect of oxygen on properties of Zircaloy-2, Bettis Laboratory, (October 1959).

TABLE IV
CORROSION RESULTS FOR UNPICKLED ZIRCALOY-2 TESTED IN 750°F STEAM

	Weight Changes in mg/dm ² for the Following Days in Test					
	<u>14</u>	<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>
<u>Shaper</u>						
Sharp Tool	24	29	45	59	76	85
Dull Tool	24	30	46	59	81	86
<u>Slab Mill</u>						
Sharp Tool	25	30	51	63	87	94
Dull Tool	26	32	53	67	85	95
<u>Straddle Mill</u>						
Sharp Tool	7	9	23	34	45	55
Dull Tool	21	25	42	55	69	79
<u>Shell Mill</u>						
Sharp Tool	25	30	50	63	78	93
Dull Tool	29	36	52	59	68	76
<u>End Mill (Overlap Face Cut)</u>						
Sharp Tool	24	30	52	64	80	95
Dull Tool	24	29	50	62	78	92
<u>End Mill (Non-overlap Face Cut)</u>						
Sharp Tool	25	30	53	63	79	93
Dull Tool	27	33	49	63	78	91
<u>End Mill (Edge Cut)</u>						
Sharp Tool	24	30	52	64	80	95
Dull Tool	25	30	52	65	82	96
<u>Surface Grind</u>						
New Wheel	27	34	52	67	82	97
<u>Standards</u>	29	36	61	83	105	134

**Figure 2 Zircaloy-2 Coupons Machined on a Horizontal Straddle Mill and Pickled 0.002 Inch per Surface.
Coupons Shown after Corrosion Test in 750°F Steam for 140 Days.**

pickled 2 mils per surface. The pickling operation removed the adverse effect of the defective tool on the straddle-milled samples as well as any effect of the dull shell-mill cutter.

The effects of pickling depth on weight gain were uncertain in both 680°F water and 750°F steam. An examination of weight gains of the standard Zircaloy-2 coupons placed in each autoclave indicated that at least part of the difference in results was caused by the autoclaves. Based on the statistical analysis of this particular study (Appendix I), no definite comparisons can be made as to which depth of pickling gave better results. Figures 3 through 6 show comparisons among the different treatments.

V. CONCLUSIONS

Machined, unpickled Zircaloy-2 exhibits good corrosion resistance if properly machined. The sharpness and hardness of the tools used had

a definite effect on the corrosion resistance of machined, unpickled Zircaloy-2. Variations in the feed and type of machine used had no effect on the corrosion behavior when a sharp cutter was used. Surface grinding with a silicon carbide wheel and ample lubricant does not adversely affect the corrosion resistance of Zircaloy-2. Corrosion behavior differed more as a result of the difference in the autoclaves used for testing than as a result of the difference between coupons pickled 1 and 2 mils per surface.

APPENDIX I: STATISTICAL ANALYSIS*

An experimental design for the present study included a complete factorial design for each test condition (680°F water and 750°F steam) and involved seven machines, three feeds, sharp and

*W. L. Davis, Bettis Laboratory, now with the Atlantic Refining Company, Philadelphia, Pa.

TABLE V
CORROSION RESULTS FOR ZIRCALOY-2 PICKLED 1 MIL/SURFACE AND
TESTED IN 750°F STEAM

Weight Changes in mg/dm ² for the Following Days in Test						
	<u>14</u>	<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>
<u>Sharper</u>						
Sharp Tool	27	35	60	76	93	105
Dull Tool	27	35	60	78	96	107
<u>Slab Mill</u>						
Sharp Tool	27	34	58	74	92	103
Dull Tool	28	34	57	75	91	101
<u>Straddle Mill</u>						
Sharp Tool	26	33	57	72	89	101
Dull Tool	27	35	58	75	92	102
<u>Shell Mill</u>						
Sharp Tool	26	34	59	74	91	102
Dull Tool	27	34	57	78	94	108
<u>End Mill (Overlap Face Cut)</u>						
Sharp Tool	27	32	58	76	93	104
Dull Tool	28	34	59	82	97	113
<u>End Mill (Non-overlap Face Cut)</u>						
Sharp Tool	27	33	58	76	91	102
Dull Tool	28	35	60	79	95	107
<u>End Mill (Edge Cut)</u>						
Sharp Tool	27	34	59	77	92	104
Dull Tool	29	36	60	81	97	109
<u>Surface Grind</u>						
New Wheel	28	35	59	75	92	101
<u>Standards</u>						
	26	42	68	99	124	151

TABLE VI
CORROSION RESULTS FOR ZIRCALOY-2 PICKLED 2 MILS/SURFACE AND
TESTED IN 750°F STEAM

	Weight Changes in mg/dm ² for the Following Days in Test					
	<u>14</u>	<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>
<u>Shaper</u>						
Sharp Tool	26	33	53	67	84	108
Dull Tool	27	34	55	69	88	113
<u>Slab Mill</u>						
Sharp Tool	25	34	52	64	80	107
Dull Tool	27	34	53	66	81	109
<u>Straddle Mill</u>						
Sharp Tool	26	33	52	63	79	105
Dull Tool	25	34	51	64	79	105
<u>Shell Mill</u>						
Sharp Tool	26	34	52	65	80	107
Dull Tool	22	34	55	69	87	114
<u>End Mill (Overlap Face Cut)</u>						
Sharp Tool	27	33	52	65	82	107
Dull Tool	26	33	56	71	90	117
<u>End Mill (Non-overlap Face Cut)</u>						
Sharp Tool	26	34	51	64	79	103
Dull Tool	26	35	54	66	84	109
<u>End Mill (Edge Cut)</u>						
Sharp Tool	26	33	51	63	78	104
Dull Tool	27	34	53	66	81	106
<u>Surface Grind</u>						
New Wheel	28	36	54	66	79	105
<u>Standards</u>	26	42	60	78	98	131

TABLE VII
CORROSION RESULTS FOR UNPICKLED ZIRCALOY-2 TESTED IN 680°F WATER

		Weight Changes in mg/dm ² for the Following Days in Test							
		<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>	<u>168</u>	<u>196</u>	<u>224</u>
<u>Shaper</u>									
Sharp Tool		18	22	26	29	34	40	44	50
Dull Tool		15	21	25	28	32	38	43	49
<u>Slab Mill</u>									
Sharp Tool		16	21	27	36	44	49	53	63
Dull Tool		17	23	32	40	46	51	59	66
<u>Straddle Mill</u>									
Sharp Tool		-1	2	2	2	14	5	8	11
Dull Tool		11	15	21	25	30	35	39	45
<u>Shell Mill</u>									
Sharp Tool		16	21	27	33	40	46	52	58
Dull Tool		19	23	25	26	27	28	30	32
<u>End Mill (Overlap Face Cut)</u>									
Sharp Tool		16	21	27	36	43	49	55	62
Dull Tool		16	20	23	28	35	41	45	51
<u>End Mill (Non-overlap Face Cut)</u>									
Sharp Tool		18	24	30	39	46	52	59	67
Dull Tool		17	22	26	31	38	44	49	57
<u>End Mill (Edge Cut)</u>									
Sharp Tool		17	23	28	39	46	50	56	66
Dull Tool		17	23	29	38	45	51	58	65
<u>Surface Grind</u>									
New Wheel		16	22	27	33	42	49	53	58
Standards		21	26	30	38	48	56	62	74

TABLE VIII
CORROSION RESULTS OF ZIRCALOY-2 PICKLED 1 MIL/SURFACE AND
TESTED IN 680°F WATER

		Weight Changes in mg/dm ² for the Following Days in Test						
		<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>	<u>168</u>	<u>196</u>
<u>Shaper</u>								
	Sharp Tool	22	26	29	33	41	48	54
	Dull Tool	21	25	28	32	41	47	52
<u>Slab Mill</u>								
	Sharp Tool	21	25	28	31	36	42	48
	Dull Tool	21	26	28	31	38	44	51
<u>Straddle Mill</u>								
	Sharp Tool	20	25	28	32	38	45	50
	Dull Tool	21	25	28	31	40	47	53
<u>Shell Mill</u>								
	Sharp Tool	21	26	28	30	37	46	53
	Dull Tool	21	25	28	32	39	47	53
<u>End Mill (Overlap Face Cut)</u>								
	Sharp Tool	22	26	29	32	36	46	51
	Dull Tool	22	25	28	32	38	47	52
<u>End Mill (Non-overlap Face Cut)</u>								
	Sharp Tool	21	25	28	32	38	46	52
	Dull Tool	21	26	28	32	42	48	54
<u>End Mill (Edge Cut)</u>								
	Sharp Tool	22	26	29	32	38	45	52
	Dull Tool	23	26	29	32	39	47	52
<u>Surface Grind</u>								
	New Wheel	22	26	29	32	37	45	50
<u>Standards</u>		22	27	30	42	51	57	63

TABLE IX
CORROSION RESULTS OF ZIRCALOY-2 PICKLED 2 MILS/SURFACE AND
TESTED IN 680°F WATER

Weight Changes in mg/dm ² for the Following Days in Test							
	<u>28</u>	<u>56</u>	<u>84</u>	<u>112</u>	<u>140</u>	<u>168</u>	<u>196</u>
<u>Shaper</u>							
Sharp Tool	23	27	31	34	46	53	57
Dull Tool	21	26	30	34	47	53	56
<u>Slab Mill</u>							
Sharp Tool	21	25	29	33	43	51	55
Dull Tool	21	25	28	33	44	51	55
<u>Straddle Mill</u>							
Sharp Tool	21	26	29	34	47	52	56
Dull Tool	21	25	28	33	46	52	56
<u>Shell Mill</u>							
Sharp Tool	21	26	28	33	46	53	56
Dull Tool	21	25	29	33	47	53	57
<u>End Mill (Overlap Face Cut)</u>							
Sharp Tool	22	26	30	32	44	51	56
Dull Tool	22	26	31	34	48	53	57
<u>End Mill (Non-overlap Face Cut)</u>							
Sharp Tool	21	25	30	33	45	53	56
Dull Tool	21	26	30	33	46	53	56
<u>End Mill (Edge Cut)</u>							
Sharp Tool	22	26	31	33	45	53	56
Dull Tool	23	26	30	34	45	53	57
<u>Surface Grind</u>							
New Wheel	22	26	30	34	43	52	56
<u>Standards</u>							
	22	29	34	50	55	63	72

Figure 3 Average Weight Gains for Machined Coupons after 84 Days in 750°F Steam.

Figure 4 Average Weight Gains for Machined Coupons after 140 Days in 750°F Steam.

dull cutters, three pickling depths, two autoclaves, and two replications.

Delays in coupon preparation necessitated the placement of coupons with different pickling depths in different autoclaves. Two standard Zircaloy-2

specimens were placed in each autoclave; with this information, it was possible to derive a statistic for testing the effect of pickling depth after adjusting for differences between autoclaves. However, this statistic was relatively inadequate because of limited information. Because of financial

Figure 5 Average Weight Gains for Machined Coupons after 112 Days in 680°F Water.

Figure 6 Average Weight Gains for Machined Coupons after 196 Days in 680°F Water.

limitations, one autoclave was eliminated after 28 days of testing for all but the unpickled specimens.

A. Method of Analysis

The results caused by the difference in the autoclaves interacted with the other experimental results. This was verified by tests of significance. As a consequence, whenever applicable, all interactions involving the autoclaves were pooled with duplicate measurements for testing the significance of the remaining factors. All conclusions were based on a probability level of 0.95.

B. Conclusions

The variability attributable to the different autoclaves was high; as a result the magnitude of some of the other effects reported as significant may have been too small to be of practical importance. This was particularly true for the machined coupons tested in 680°F water.

The over-all effects of pickling depth and machine feeds were judged too small to be considered as other than random variations. Some particular combinations of variables were important for the unpickled specimens but were not large enough to be considered important for the pickled specimens.

The straddle-milled, unpickled coupons showed lower weight gains than the unpickled coupons processed by the other machines. However, this effect was not noticed for the pickled specimens.

The coupons processed by the dull cutter on the shell mill showed erratic corrosion resistance in both the steam and water tests.

The unpickled specimens machined with a dull tool showed higher initial weight gains as well as a higher rate of gain than those machined with a sharp tool.

The pickled coupons machined with a dull tool showed higher weight gains than those machined with a sharp tool, but the rate of gain was the same for both groups.

ACKNOWLEDGMENTS

The author thanks J. Maringo of the Development Machine Shop for his aid in preparation of the coupons, the Corrosion Test Laboratories of the Engineering Laboratories for the corrosion tests; A. C. Nelson and W. L. Davis for their help in statistically analyzing the data; and S. Kass for his guidance in performing and reporting the experiments.

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