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The Technical Feasibility of Cold Extrusion for Zircaloy-2 Tubing Production

Quarterly Report No. 3 (April - June 1961)

Advanced Technology Laboratories, A Division of American-Standard
Mountain View, California

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Quarterly Technical Progress Report #3

INVESTIGATION OF THE TECHNICAL FEASIBILITY OF COLD EXTRUSION FOR ZIRCALOY-2 TUBING PRODUCTION

ATL Job 4028

April-June 1961

for

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STATEMENT OF PROBLEM

The over-all objective of the investigation is to establish the feasibility of using cold extrusion to produce Zircaloy-2 tubular products. The first phase was concerned with determining basic feasibility of cold-extruding Zircaloy-2 and evaluating lubricants. The economic feasibility of this process also was investigated. The second phase, now in progress, is concerned with the technique and limitations of making Zircaloy-2 tubular extrusions.

SUMMARY OF PROGRESS TO DATE

Lubrication systems composed of a lubricant and a conversion coating were developed and evaluated for the cold extrusion of Zircaloy-2. A fluoride phosphate base coating and several lubricants were selected for actual extrusion tests. Bars of Zircaloy-2 wer successfully extruded at temperatures ranging from room temperature to 400 C, using reductions of 50, 65, and 80%. Excellent surface finish was obtained, and no evidence of cracks or other defects could be found. Fully annealed, extruded specimens exhibited the same tensile properties as similarly treated raw bar stock. Preferred orientation was found to be the same as for hot-extruded tubing, and corrosion resistance in 750 F, 1500-psi steam is the same as for raw bar stock.

Billets have been pierced successfully, using reductions of up to 80% at 300 C. A 140-degree conical punch profile was found to provide the best surface and to require the lowest pressure. Open-end tubes (1-inch OD) have been successfully extruded with maximum reductions of 80% and final wall thicknesses down to 0.024 inch.

Short lengths of fuel-element-size tubing (0.560" OD × 0.030" wall) have been extruded; tube quality appears to be excellent. Tools to produce this tubing, from 1-inch-diameter billets, in one piercing and two forward-extrusion steps were designed and tested.

A preliminary analysis of the economic potential of this process for Zircaloy-2 tubing production has shown that: 1) the cold extrusion of heavy-walled tube shells is economic if sufficient production volume exists, and 2) production of thin-walled fuel-containing tubes with integral end caps appears promising because a better product can be obtained at considerably lower cost than that currently associated with reactor-grade plain tubing.

PROGRESS APRIL-JUNE 1961

A. Extrusion Tests Using $1\frac{1}{4}$" Diameter Billets

Tests on extruding $1\frac{1}{4}$" diameter billets were completed during this quarter. No additional extrusions of this size are planned, except to provide specimens for mechanical testing of cold-extruded Zircaloy-2.

1. Bar Extrusion

During this quarter, 46 bar-extrusion tests were performed. Samples were extruded with 80% reduction at 400 C to complete the series of three reductions at each of five temperatures. Results are given in Table I. Lubricant breakdown occurred at 400 C in all tests and was most severe at 80% reduction. Based on these results, 300 C has been established as the maximum extrusion temperature for all future work on this program.

A series of specimens was extruded at 300 C with 65% reduction, using the 8 w/o MoS_2 + 2 w/o Sb_2S_3 in resin lubricant, to determine the variation in maximum pressure with specimen length. Although results varied significantly among the $2\frac{1}{2}$" long specimens, the averaged value obtained for peak pressure was reasonable. For $1\frac{1}{4}$" specimens, the average peak pressure was 110,000 psi; for 1-7/8" specimens, 118,000 psi; and for $2\frac{1}{2}$" specimens, 127,000 psi. When superimposed and matched at their terminal points, the three pressure-stroke curves coincide as expected. Since the initial diameter of each billet is $1\frac{1}{4}$ inch, the coefficient of friction for these conditions is easily calculated to be about 0.03. This value is somewhat lower than expected, but not unreasonable.

The second lot of Zircaloy-2 obtained for this investigation exhibited somewhat higher room-temperature tensile properties than did the first lot. However, yield and tensile strengths at 300 C were comparable. The difference in tensile properties exhibited by two lots is explained by variation in purity levels. The new lot contains 1200 ppm oxygen, while the original lot had less than 900 ppm. Extrusions were made at room temperature and at 300 C, with 65% and 80% reductions, using both lots of Zircaloy. The pressures required for the two lots were comparable, variations being within the limits of accuracy of the tests.

2. Piercing

During this quarter, 12 piercing tests were run to complete this portion of the program. Tests were conducted at room temperature and at 200 C, using 50% reduction and a 140-degree conical punch profile. Cracking occurred in many specimens, possible due to coarse grain structure. Pressures required using Moly-Spray-Kote were 376,000 psi at room temperature and 285,000 psi at 200 C. This compares with 244,000 psi required at 300 C, the normal piercing temperature employed in this investigation. Since high punch pressures can result in tool breakage, all piercing will continue to be performed at 300 C.

The 155-degree conical-profile punch was modified by grinding 0.020 inch from the diameter of the nose, leaving only part of the 1/16-inch radius on the edge of the punch. This procedure has been used successfully in cold extrusion of steel. It is theorized that the metal will follow the radius, even past the sharp corner, leaving ample clearance for removal of the punch from the extrusion. The punch exhibited less tendency to stick in the billet, and the pressure required was about the same as for the unmodified punch. This punch design was used later in the program for making small tubes.

3. Tube Extrusion

During this quarter, 35 tube-extrusion tests were run, all at 200 C with the 8 w/o $MoS_2 + Sb_2S_3$ in resin lubricant. Tubular billets, $1\frac{1}{4}$" OD, were used throughout this part of the program. Detailed dimensions are shown in Table II for each case.

Consistently good extrusions were obtained in forming 1.079" OD × 0.052" wall tubes with 65% reduction. Excellent surfaces were obtained with reasonable pressure (222,000 psi for $1\frac{1}{4}$" billets). The length of tubes produced under these conditions appears to be limited only by tooling size.

Some successful 0.962" OD × 0.044" wall tubes were extruded with 80% reduction. However, the high pressures required (about 400,000 psi on the unextruded tube wall) tended to force material between the punch and the die wall, further raising the applied force. Although the diametral clearance between the punch and the die was only about 0.002 inch, flash was sometimes as thick as 0.007 inch completely around the punch, making it extremely difficult to remove the punch from the die and causing some tool breakage. This difficulty can be eliminated to some extent by closer control of clearances and selection of appropriate follower-block material.

Considerable difficulty was experienced in the extrusion of 1.079" OD × 0.024" wall tubes with 80% reduction. Although some flash resulted from the peak pressure of about 450,000 psi on the unextruded tube wall, it was minimized by very small punch-to-die clearance. Lubrication of the mandrel presented the greatest problem. With poor lubrication, tool breakage took the form of necking of the mandrel under tension near the point of attachment to the punch. Acceptable extrusions could be produced only by extremely careful lubrication of the mandrel before each test. Use of a floating mandrel, not attached to the punch, is believed to be the solution to this problem. The mandrel can then travel with the extrusion, which moves five times faster than the punch for 80% reduction. The punch for extruding small tubing was designed to include a floating mandrel.

Tube-extrusion results are summarized in Table II. Low efficiencies as compared with those obtained for bar extrusion are due to the large surface-to-volume ratio of tubular specimens, and therefore, the large frictional losses. Thus, successful tube extrusion depends even more critically on the choice and availability of a good lubricant.

Excellent surfaces, with no indication of scratches or other defects, were exhibited by the successful extrusions produced during this portion of the program. Difficulties encountered resulted from the small wall-thickness-to-diameter ratios selected and were much more severe than those anticipated in extruding small-sized reactor tubing.

B. Extrusion of Small Tubes

Tooling was designed and fabricated to produce, in three steps (piercing and two extrusions), 12-inch lengths of 0.560" OD × 0.030" wall tubing from 0.955" diameter billets. The tool dimensions and extrusion pressures required are given in Table III. Final tube length is limited by length of the available die holder. To date, each extrusion step has been tested separately, but individual specimens have not been subjected, as yet, to all three successive reductions.

1. Piercing

The piercing punch contained a 140-degree conical profile and the partial-edge radius previously described. The assembly was similar to that used for large billets. Pierced surfaces were excellent, and the punch was easily removed from the specimen without sticking. The 120-degree chamfer is formed on the specimen during the piercing step by the use of a shaped rather than a flat anvil. The main problem encountered in this step has been obtaining concentricity of the pierced hole in the billet. Moving the die in relation to the punch has only partly corrected this misalignment. The difficulty is probably related to changes in press alignment with stroke and pressure. It would appear that the best solution is to use a precision die set or sub-press of some type for the piercing operation. In production, a small press is required because of the small load (25 to 30 tons), so that alignment should be easy to maintain. Methods of improving press alignment are being investigated.

Some improvement probably can be made by coining the specimen before piercing, i.e., compressing the billet with a short rigid punch. This would make the specimen completely fill the die and also provide an accurately located center indentation to guide the piercing punch. Simple tooling is currently being manufactured to coin the billets.

2. First Tube-Extrusion Step

Tooling for the first small-tube extrusion step is similar to that used for producing larger tubes. Because of the short length of the mandrel, only small tensile loads would be impressed on it. Therefore, it was considered safe to have the mandrel attached to the punch. The die angle is 120 degrees.

Open-end tubes were extruded with no difficulty, using graphite follower blocks to eliminate the unextruded butt. Approximately 3/16 inch of tapered wall was found at the trailing end of the extrusion, and the extruded surface was excellent both internally and externally. No tool breakage was experienced.

A number of capped tubes were extruded, but cracking normally occurred at the bottom of the cap. The entire bottom often separated from the tube wall. The contact point of the mandrel with the bottom was adjusted by changing the length of the graphite follower, but bottom imperfections were still obtained. If the mandrel hits bottom before the punch contacts the top of the specimen, the bottom is cracked or severed. If the punch hits before the mandrel, some of the tube extrudes around the bottom of the mandrel, forming an irregular tube.

This cracking, which has been found in the extrusion of other materials when the bottom thickness is greater than the thickness of the extruded tube wall, is due to a) the difference in reduction in area given to the solid bottom and the tube wall and b) the high stresses set up at the junction of the solid bottom and the tube. One solution is to form the final diameter of the solid bar during the piercing or coining operation. Only the tube will then be extruded in the subsequent extrusion steps. This technique is used in the cold extrusion of certain steel parts. Piercing anvils are being designed so that the final 0.560" diameter bottom cap will be formed when the billet is coined.

3. Second Tube-Extrusion Step

The die for this extrusion is similar to the other tube-extrusion dies, except that it will accept a billet 4 inches long rather than one only 3 inches long. The punch, however, is designed differently. The mandrel, a 12-inch length of hardened and ground high-speed steel drill rod, is separate from the punch. The punch is hollow and has an accurately ground length to serve as a guide for the mandrel. The punch backing plate also has a clearance hole for the mandrel. No ejector is used due to the length of the extruded tube, but graphite follower blocks are used, allowing the tube and mandrel to fall out of the die after extrusion. The mandrel is carefully lubricated so that it can be removed from the extruded tube.

An excellent tube was produced using this tooling. Surfaces were good, and the mandrel was easily removed. Additional work is in progress on this type of small-tubing production, and punches of slightly revised design are being manufactured for further experiments.

C. Evaluation of Extrusions

Extruded samples of the new lot of Zircaloy-2 were tensile tested before and after annealing. In all cases, the tensile and yield strengths at room temperature were about 10% higher than for the original lot of Zircaloy-2.

Annealing treatments for as-received bar stock have been investigated. One hour at 800 C yields approximately the same tensile properties as 6 hours and 24 hours at the same temperature. Annealing 6 hours at 800 C, with samples extruded to 80% reduction, sometimes causes very large grain size. This can be partially eliminated by annealing for 1 hour or less. Annealing extruded Zircaloy for 15 to 30 minutes in a salt bath at 650 to 675 C was tried. Resulting grain size was about 10 to 20 microns, and contaminated skin appeared to be less than 0.001 inch deep. This annealing treatment will be investigated during the production of small tubes in an attempt to minimize grain growth, especially after large reductions. Tensile tests will be conducted on various samples subjected to this treatment.

Annealed samples of the new lot of Zircaloy exhibited elliptical fracture surfaces after tensile testing. Wah Chang, the producer of the material, believes that this is due to "memory" of the material from the forging operation. Since the lot was produced, the production scheme has been modified to eliminate this unsymmetrical preferred orientation. It is possible that some of the cracking observed only at 90-degree intervals in certain extrusion tests was due to this preferred orientation.

During this quarter, Wah Chang was contacted to discuss Zircaloy production and fabrication techniques and to evaluate the market for cold-extruded tubing. In addition, Hanford Laboratories were visited to discuss Zircaloy tubing and mechanical properties.

An interim progress report* was published in May, along with a request for extension of the project through fiscal year 1962.

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* ATL-D-598, 5 May 1961.

TABLE I

EXTRUSION PRESSURE AND EFFICIENCY FOR
FORWARD EXTRUSION OF ZIRCALOY-2 BAR AT ELEVATED TEMPERATURE

Temp.	Lubricant	50% Reduction 120° Die Angle		65% Reduction 120° Die Angle		65% Reduction 150° Die Angle		80% Reduction 120° Die Angle	
		Max. Pressure (psi)	Deform. Eff., η (%)	Max. Pressure (psi)	Deform. Eff., η (%)	Max. Pressure (psi)	Deform. Eff., η (%)	Max. Pressure (psi)	Deform. Eff., η (%)
Room Temp.	Moly-Spray-Kote	159,000	37	174,000	55	-	-	226,000	69
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	153,000	62	-	-	257,000	61
100 C	Moly-Spray-Kote	138,000	39	149,000	58	-	-	214,000	67
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	173,000	50	-	-	229,000	62
	5 w/o Graphite + 5 w/o MoS_2 in Resin	126,000	43	-	-	-	-	-	-
200 C	Moly-Spray-Kote	96,000	39	131,000	48	142,000	43	176,000	57
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	156,000	39	142,000	43	203,000	50
	5 w/o Graphite + 5 w/o MoS_2 in Resin	90,000	42	-	-	-	-	-	-

TABLE I
(concl.)

Temp.	Lubricant	50% Reduction 120° Die Angle Max. Pressure (psi)	50% Reduction 120° Die Angle Deform. Eff., η (%)	65% Reduction 120° Die Angle Max. Pressure (psi)	65% Reduction 120° Die Angle Deform. Eff., η (%)	65% Reduction 150° Die Angle Max. Pressure (psi)	65% Reduction 150° Die Angle Deform. Eff., η (%)	80% Reduction 120° Die Angle Max. Pressure (psi)	80% Reduction 120° Die Angle Deform. Eff., η (%)
300 C	Moly-Spray-Kote	78,000	38	108,000	44	122,000	39	140,000	56
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	100,000	48	112,000	43	135,000	58
	5 w/o Graphite + 5 w/o MoS_2 in Resin	72,000	41	-	-	-	-	-	-
400 C	Moly-Spray-Kote	93,000	36	93,000	42	-	-	162,000	40
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	85,000	46	-	-	151,000	43
	10 w/o Graphite in Resin	-	-	83,000	47	-	-	-	-
	5 w/o Graphite + 5 w/o MoS_2 in Resin	66,000	37	-	-	-	-	-	-

NOTES:
1. All specimens used the high-temperature fluoride phosphate conversion coating under the indicated lubricant.
2. The specimens used in the 50% reduction tests were $2\frac{1}{2}$ inches long. All others were $1\frac{1}{4}$ inches long.

TABLE II

TUBE-EXTRUSION RESULTS

Reduction (%)	Temp. (°C)	Maximum Pressure (psi)	Length (in.)	η (%)	OD (in.) Initial	OD (in.) Final	ID (in.)	Wall Thickness (in.) Initial	Wall Thickness (in.) Final
50	300	90,000	$1\frac{1}{4}$	33	1.250	1.079	0.875	0.188	0.102
"	"	104,000	$2\frac{1}{2}$	28	"	"	"	"	"
"	100	173,000	$1\frac{1}{4}$	31	"	"	"	"	"
"	"	172,000	$2\frac{1}{2}$	31	"	"	"	"	"
65	200	290,000	$2\frac{1}{2}$	21	1.250	1.023	0.875	0.188	0.074
"	300	191,000	$1\frac{1}{4}$	25	"	1.079	0.974	0.133	0.052
"	"	181,000	$2\frac{1}{2}$	26	"	"	"	"	"
"	200	222,000	$1\frac{1}{4}$	27	"	"	"	"	"
"	"	256,000	$2\frac{1}{2}$	24	"	"	"	"	"
80	300	217,000	$1\frac{1}{4}$	36	1.250	0.962	0.875	0.188	0.044
"	"	236,000	$2\frac{1}{2}$	32	"	"	"	"	"
"	200	399,000	$1\frac{1}{4}$	25	"	"	"	"	"
"	"	451,000	$1\frac{1}{4}$	22	"	1.079	1.032	0.109	0.024

NOTE: Specimens lubricated with 8 w/o MoS_2 + 2 w/o Sb_2S_3 in resin over the high-temperature fluoride phosphate conversion coating.

TABLE III

EXTRUSION OF SMALL TUBES

Step	Reduction	Max. Load	Max. Pressure	Initial Dimensions			Final Dimensions		
				ID	OD	Length	ID	OD	Length
	(%)	(lb)	(psi)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)
Piercing	30	56,000	265,000	Solid	0.945	15/16	0.520	0.955	1-1/4
1st Tube Extrusion	75	127,000	241,000	0.520	0.955	1-3/16	0.510	0.654	4-3/8
2nd Tube Extrusion	67	47,000	314,000	0.510	0.654	1-1/8	0.500	0.560	12

NOTES: 1. Piercing pressure figured on punch area, tube-extrusion pressure on the area of unextruded tube wall.

2. All specimens used the 8 w/o MoS_2 + 2 w/o Sb_2S_3 in resin lubricant over the high-temperature fluoride phosphate conversion coating.

CONCLUSIONS

The production of cold-extruded Zircaloy-2 fuel-containing tubes appears to be feasible. Both large- and small-diameter tubes have been produced successfully. Corrosion resistance appears to be adequate, and the preferred orientation is similar to that found in tubing produced by conventioanl means.

PLANS FOR FUTURE WORK

The extrusion of small-sized tubes will be continued, with emphasis on perfecting tool design. Various annealing cycles will be investigated. Finished tubes will be evaluated with respect to mechanical properties and defects to determine suitability for reactor use.

PRINCIPAL INVESTIGATORS

Investigators on this project include: F. E. Weil, Metallurgist and Project Leader; J. G. Hill, Associate Metallurgist. Over-all supervision is exercised by Dr. Donald R. Mash, Manager, Materials Laboratory.

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