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Quarterly Technical Progress Report

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

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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 were 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 0.044 inch. Progress is being made toward obtaining uniformly good surfaces on these extrusions.

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 JANUARY-MARCH 1961

A. Extrusion Tests

1. Bar Extrusion

During the first quarter of 1961, 105 bar-extrusion tests were performed. This essentially completes bar-extrusion experiments, except those involving 80% reduction at 400 C and those needed to provide additional tensile data on the new lot of Zircaloy-2 recently obtained.

Bar-extrusion results are summarized in Table I. Good surfaces can be obtained for all conditions listed, the surface finish being about 32 to 64 microinches rms on the better samples. Some lubricant breakdown was evident at 400 C, but surfaces were still acceptable. The optimum extrusion temperature appears to be 300 C, based on such considerations as lubricant condition and extrusion pressure.

The 10 w/o MoS_2 + 10 w/o Sb_2S_3 in resin appears to hold up better under temperature than Moly-Spray-Kote. Below 300 C, specimens lubricated with Moly-Spray-Kote required lower pressures, but the resin suspension was better above that temperature. The 150-degree die angle required greater pressure than did the 120-degree angle normally used.

A series of tests was run to determine the optimum ratio of Sb_2S_3 to MoS_2 in the resin-suspension lubricants. Both bar extrusions and piercing tests were run, using a series of lubricants. The results are presented in Table II. In both cases, the 8 w/o MoS_2 + 2 w/o Sb_2S_3 combination required the lowest pressure. The lubricants with 10% total solids had better surfaces after stripping than did those with 20% solids. The 8 w/o MoS_2 + 2 w/o Sb_2S_3 in resin is being used in the remaining tests.

2. Piercing

During this report period, 145 piercing tests were run. Samples were reduced up to 80%, the better specimens having excellent surface finish. Concentricity was only fair, since coining was not used to locate the punch before piercing.

Table III presents results obtained using the same basic punch design but different punch profiles. All punches included a 3/64-inch land, relieved 45 degrees to 0.025 inch below the punch diameter. The 140-degree spherical and 140-degree conical punch profiles produced the best results. Slightly better surface finish was obtained with the conical punch profile, which also exhibited less tendency to wander. More lubricant breakdown occurred with the spherical punches, as evidenced by a double peak on the pressure-stroke curve, the second peak often being much higher than the first. This behavior was shown to be a characteristic of the punch, since it occurred with 2-inch, as well as 1-inch, billets.

New punches with the two best profiles were obtained, covering reductions of 35 to 80%. The punches were designed with a 1/32-inch land and a more gradual $2\frac{1}{2}$-degree relief angle. These changes reduced specimen adherence to the punch after piercing. Results, given in Table IV, show that the conical punch required lower pressure than did the spherical.

Theoretically, the minimum pressure for piercing occurs at 50% reduction. The results obtained tend to substantiate those calculations. It should be noted, however, that although the pressure decreases slightly or remains the same between 35 and 50% reduction, the force required increases due to the larger area of the punch.

The surface on pierced cups was somewhat variable, but changes in punch design and possibly in temperature should make it possible to obtain consistently acceptable cups.

3. Tube Extrusion

In the 46 tube-extrusion tests run to date, reductions to 80% and wall thicknesses to 0.044 inch were achieved. Surfaces ranged from badly galled to excellent. Some tool breakage has been experienced due to both defects and improper design. Consistently good results have been obtained at 65% reduction at 200 C; higher temperatures have caused lubricant breakdown. With proper tool design, 80% reduction appears to be readily attainable.

Results obtained to date, and summarized in Table V, are tentative because of current changes in tool design. The good tubes obtained appear to be free of cracks and other imperfections. The insides of a few tubes produced under poor lubrication conditions contained circumferential cracks about $\frac{1}{4}$-inch long.

TABLE I

EXTRUSION PRESSURE AND EFFICIENCY FOR FORWARD EXTRUSION OF ZIRCALOY-2 BAR

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	-	-	-	-
	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	-	-	85,000	46	-	-	-	-
	10 w/o Graphite in Resin	-	-	83,000	47	-	-	-	-
	5 w/o Graphite + 5 w/o MoS_2 in Resin	66,000	37	-	-	-	-	-	-

TABLE II

SUMMARY OF LUBRICANT TESTS
(Performed at 300°C)

Bar Extrusion 65% Reduction	
Lubricant	Max. Pressure (psi)
8 w/o MoS_2 + 2 w/o Sb_2S_3 in Resin	110,000
10 w/o MoS_2 in Resin	114,000
10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	115,000
20 w/o MoS_2 in Resin	117,000
5 w/o MoS_2 + 5 w/o Sb_2S_3 in Resin	138,000
2 w/o MoS_2 + 8 w/o Sb_2S_3 in Resin	145,000
10 w/o Sb_2S_3 in Resin	182,000
20 w/o Sb_2S_3 in Resin	202,000

Piercing 65% Reduction 140-degree Conical Punch	
Lubricant	Max. Pressure (psi)
8 w/o MoS_2 + 2 w/o Sb_2S_3 in Resin	237,000
20 w/o MoS_2 in Resin	243,000
10 w/o MoS_2 in Resin	245,000
6.7 w/o MoS_2 + 3.3 w/o Sb_2S_3 in Resin	246,000
10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	250,000
5 w/o MoS_2 + 5 w/o Sb_2S_3 in Resin	252,000

TABLE III

PIERCING-TEST RESULTS FOR VARIOUS PUNCH PROFILES
(50% REDUCTION, 300 C, 1" BILLETS)

Punch Profile	Maximum Pressure (psi)				
	5 w/o Graphite + 5 w/o MoS_2 in Resin	10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	Moly-Spray-Kote	10 w/o Graphite in Resin	Dag 210
120° Spherical	239,000	246,000	-	-	-
140° Spherical	233,000	224,000	249,000	232,000	259,000
160° Spherical	253,000	262,000	-	-	-
180° Spherical (hemispherical)	254,000	256,000	267,000	-	-
140° Conical	243,000	238,000	244,000	-	237,000
155° Conical	236,000	252,000	-	-	-
170° Conical	263,000	266,000	-	-	-
180° Flat Face	269,000	257,000	265,000	273,000	-

Note: Punches had 3/64-inch land and 45-degree relief angle.

TABLE IV

PIERCING-TEST RESULTS FOR VARIOUS REDUCTIONS
(REVISED PUNCH DESIGN, 300 C, 1" LONG × $1\frac{1}{4}$" DIA. BILLETS)

Punch Profile	Reduction (%)	Maximum Pressure (psi): 10 w/o MoS_2 + 10 w/o Sb_2S_3 in Resin	Maximum Pressure (psi): Dag 210
140° Spherical	50	234,000	242,000
	65	263,000	273,000
140° Conical	35	224,000	228,000
	50	225,000	223,000
	65	253,000	253,000
	80	291,000	336,000+

Note: Punches had 1/32-inch land and $2\frac{1}{2}$-degree relief angle.

TABLE V

PRELIMINARY TUBE-EXTRUSION RESULTS

Reduction (%)	Temp. (°C)	Maximum Pressure (psi)	Length (in.)	OD (in.) Initial	OD (in.) Final	ID (in.)	Wall Thickness (in.) Initial	Wall Thickness (in.) Final
50	300	90,000	$1\frac{1}{4}$	1.250	1.079	0.875	0.188	0.102
"	"	104,000	$2\frac{1}{2}$	"	"	"	"	"
"	100	173,000	$1\frac{1}{4}$	"	"	"	"	"
"	"	172,000	$2\frac{1}{2}$	"	"	"	"	"
65	200	290,000	$2\frac{1}{2}$	1.250	1.023	0.875	0.188	0.074
"	300	191,000	$1\frac{1}{4}$	"	1.079	0.974	0.133	0.052
"	"	181,000	$2\frac{1}{2}$	"	"	"	"	"
80	300	217,000	$1\frac{1}{4}$	1.250	0.962	0.875	0.188	0.044
"	"	236,000	$2\frac{1}{2}$	"	"	"	"	"

Note:

Specimens lubricated with 8 w/o MoS_2 + 2 w/o Sb_2S_3 in Resin.

B. Evaluation of Extruded Bar

1. Tensile Tests

Bar samples extruded at 300 C were tensile-tested before and after annealing. As shown in Table VI, the properties after annealing were essentially the same as in samples extruded at room temperature and annealed, except that the elongation and reduction in area were somewhat greater for the elevated-temperature extrusions. The tensile and yield strengths for samples as-extruded at 300 C were about 8% lower than for samples as-extruded at room temperature, but the elongation and reduction in area were greater for the elevated-temperature extrusions.

2. Metallographic Examination

No evidence of cracks or other discontinuities could be found, except that when severe lubricant breakdown had occurred some slight surface cracking resulted. Microhardness surveys of extruded bar showed no significant diametral variation. The microstructure was typical of cold-worked material and was fine grained after annealing.

3. Preferred-Orientation Studies

Qualitative pole figures were prepared for an as-extruded, 80% reduction sample. A strong fiber texture was found, with the basal planes (0001) parallel to the extrusion axis. The $[10\bar{1}0]$ direction is the fiber axis. The orientation is most pronounced on the outside and at the center of the sample and is less pronounced halfway to the center. The results were similar to those obtained at Hanford* on hot-extruded Zircaloy-2 tubing. It is planned to examine annealed samples as well as the starting bar stock for preferred orientation.

4. Corrosion Testing

Samples of bar stock and extruded specimens were tested in 1550-psi, 780 F steam for 14 days. Samples extruded to 80% reductions and samples extruded 80% and annealed exhibited weight gains of 36.0 and 26.6 mg/dm^2, respectively, as compared with 37.5 and 38.6 mg/dm^2 for as-received and annealed bar stock, respectively. In all cases, the corrosion product was adherent, shiny black oxide.

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* J. J. Laidler, "Preferred Orientations in Extruded Zircaloy-2 Tubing," HW-64815, April 15, 1960.

TABLE VI

TENSILE-TEST RESULTS, EXTRUDED ZIRCALOY-2 BARS

Condition	Tensile Strength (psi)	Yield Strength (0.2% Offset) (psi)	Elongation in 2 Inches (%)	Reduction in Area (%)
As-received, Annealed	63,800	36,300	22	33
Extruded at Room Temperature, Annealed				
Reduced 50%	66,100	40,300	16	26
Reduced 65%	68,800	41,600	17	30
Reduced 80%	67,400	41,200	22	30
Extruded at 300 C, Annealed				
Reduced 50%	67,600	39,600	23	36
Reduced 65%	68,000	39,800	24	38
Reduced 80%	68,200	40,200	24	41
As-extruded at Room Temperature				
Reduced 50%	86,000	76,500	4.5	14
Reduced 65%	92,600	78,500	7.2	20
Reduced 80%	99,100	78,000	7.8	30
As-extruded at 300 C				
Reduced 50%	79,500	68,000	11	32
Reduced 65%	84,600	70,800	11	36
Reduced 80%	91,000	75,200	12	42

Notes: Annealing treatment: 6 hours, 1500 F, <0.1 micron.
Strain rate: 0.001 inch/inch/second.
All tests on 0.251-inch-diameter by 2-inch gage-length specimens.

CONCLUSIONS

The production of cold-extruded Zircaloy-2 fuel-containing tubes appears to be quite promising. Large-diameter tubes have been produced, and it should be possible to extrapolate the results to smaller sizes. Corrosion resistance appears adequate, and the preferred orientation is similar to that found in tubing produced by conventional means. Final judgement as to the suitability of this process for producing tubes for nuclear applications cannot be made until more tubes have been extruded and fully evaluated.

PLANS FOR FUTURE WORK

Tube extrusion will be continued using other reductions, temperatures, and tool configurations. Closed-end tubes will be extruded and limitations determined. Small-size tubing (about 0.560-inch OD) will be extruded.

Piercing tests will be conducted at temperatures below 300 C. Other extrusion variables, such as surface finish and annealing cycle, will be investigated.

PRINCIPAL INVESTIGATORS

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

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