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Quarterly Technical Progress Report

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

ATL Job 4028

July-September 1961

UNITED STATES-EURATOM JOINT RESEARCH AND DEVELOPMENT PROGRAM

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Project Agreement No. 7

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DISCLAIMER

STATEMENT OF PROBLEM

The over-all objective of the program 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 aspect 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. Bars of Zircaloy-2 successfully extruded at reductions of 50, 65, and 85% exhibited excellent surfaces and no evidence of cracks or other defects. A flouride phosphate conversion coat in connection with a lubricant containing 8 w/o MoS_2 and 2 w/o Sb_2S_3 in resin has provided the best over-all results.

Billets have been pierced successfully, with a 140-degree conical punch profile producing the best results. Open-end tubes (1-inch OD) have been successfully extruded with maximum reductions of 80% and final wall thicknesses down to 0.024 inch.

A series of bar extrusions was completed for evaluation of tensile properties of specimens given various annealing cycles. For commercial Zircaloy-2 (containing 1200 ppm oxygen), a short-time treatment (20 minutes at 660°C) provided tensile results comparable to those obtained from the more standard treatment of 6 hours at 800°C.

A precision die set for more accurate piercing of small-size specimens was designed and fabricated. Piercing anvils which will form the final-diameter bottom cap have been fabricated. These anvils should eliminate the bottom-cap separation problem.

First tube extrusions performed on as-pierced billets were only partially successful. The billets used were formed with the old tooling, which did not provide adequate concentricity. However, extrusions using machined billets were excellent. Second tube extrusions on machined billets were also successful. The punches designed to extrude full-length first-extrusion billets were improperly heat treated and failed in use; therefore, no extrusions of this type were performed.

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 JULY-SEPTEMBER 1961

A. Bar Extrusions Using $1\frac{1}{4}$" Diameter Billets

During this quarter, 80 bar-extrusion tests were performed. Samples were extruded with 50, 65, and 80% reduction at room temperature and at 300°C. Representative specimens from each reduction were machined into tensile bars and heat treated prior to tensile testing. The conditions of heat treatment were 1) vacuum annealing 6 hours at 800°C and 2) annealing 20 minutes at 660°C in a chloride salt bath (Houghton liquid heat 980). Several tensile tests were also conducted on as-extruded bars for comparison. The tensile-test results are shown in Table I.

The purpose of the salt-bath anneal was to minimize grain growth during the annealing treatment that is necessary after large reductions, as well as to decrease the time required for annealing between extrusion operations. Figure 1 shows the effect of the heat treatments on grain size, the salt-bath anneal giving a grain size of 10 to 20 microns. The only problem that might arise because of this heat treatment would be a higher tensile strength and consequently an increase in extrusion pressure over those samples annealed 6 hours at 800°C. Table I shows that this is true for the low-oxygen-containing Zircaloy-2, lot A; however, for lot B, containing 1200 ppm O_2, the physical properties are essentially the same for the two heat treatments. Since commercial Zircaloy contains up to 1500 ppm O_2, the more advantageous salt-bath heat treatment should be satisfactory for the extrusion annealing.

B. Extrusion of Small Tubes

The extrusion of small tubing (0.560" OD × 0.030" wall) was continued during this quarter. Because of tool-design difficulties, individual specimens have not been subjected to all three successive reductions. Tooling for the three steps (piercing and two extrusions) was described in a previous report.

1. Piercing

Although no specimens were pierced during this report period, several specimens were already available for subsequent extrusion operations. These specimens had been pierced using the old tooling when the concentricity was not adequate. New tooling, utilizing a precision die set, has been designed and is 80% fabricated.

2. First Tube-Extrusion Step

Thirty first extrusions were completed on pierced specimens, using either a 6-hour at 800°C vacuum anneal or a 30-minute at 660°C vacuum anneal prior to extrusion.* For both heat treatments, the maximum extrusion pressure required was 206,000 psi. This pressure is somewhat higher than normally required because the pierced specimens used were not concentric.

Cracking of the capped tubes continued to occur; in some cases they separated from the tube wall. It is hoped, however, that new anvils which will form the final diameter during the piercing operation will rectify this situation. It might also be possible to use the floating-mandrel principle followed in the second tube-extrusion step.

3. Second Tube-Extrusion Step

A series of 19 second tube extrusions was performed on machined billets during this quarter. Excellent results and smooth surfaces were obtained, and there was no evidence of end-cap separation. Attempts at extruding a full-length billet (4-1/8 inches) resulted in a broken punch, caused by the punch not being sufficiently guided in the die. New tooling now being fabricated should solve this problem.

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* The 30-minute anneal at 660°C is comparable to the salt-bath treatment previously described. Salt was not used because at this time the resultant oxide film could not be easily removed from the specimens.

The extrusion pressure varied with billet length, being about the same for cups as for tubes in each case. For 4-1/8" billets, the maximum pressure was 314,000 psi; for 3-1/16" billets, 247,000 psi; for 2-5/32" billets, 180,000 psi.

Several final tubes were tensile tested to determine the amount of specimen preparation required for an accurate test. Results comparable to known data were obtained on specimens which were not machined and had no reduced-area gage length. This preliminary work should facilitate mechanical testing of final extruded tubes.

No extrusions were performed on as-pierced and as-first-extruded billets because of punch breakage. New long punches and dies were ordered to handle these 4-1/8" billets, but the punch material was improperly heat treated, breaking during use. It was therefore necessary to re-order punches before this part of the program could be performed.

CONCLUSIONS

The production of cold-extruded Zircaloy-2 fuel-containing tubes appears to be feasible, although some tooling difficulties remain to be solved. Corrosion resistance appears to be adequate, and the preferred orientation is similar to that found in tubing produced by conventional means.

PLANS FOR FUTURE WORK

The extrusion of small-sized tubes will be continued, with emphasis on improving the concentricity of the piercing operation. Tubes will be extruded, using various annealing cycles and end-cap configurations. The short-time annealing cycle will be investigated with respect to the use of a salt bath. 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, Section Supervisor and Project Leader; J. G. Hill, Associate Metallurgist.

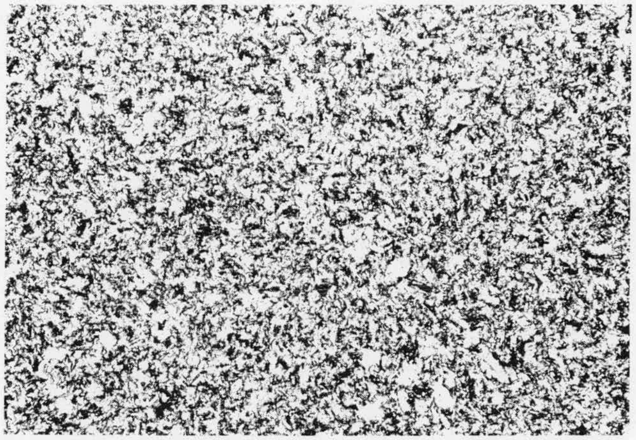
TABLE I

TENSILE-TEST RESULTS

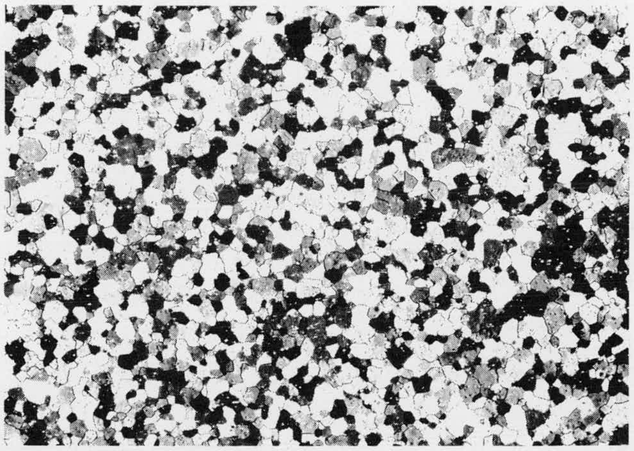
Condition	Extruded at Room Temperature								Extruded at 300°C							
	Lot A*				Lot B**				Lot A*				Lot B**			
	Ultimate	Yield	Elong.	R/A	Ultimate	Yield	Elong.	R/A	Ultimate	Yield	Elong.	R/A	Ultimate	Yield	Elong.	R/A
As extruded 50%	86,000	76,500	5	14	98,750	92,000	9	20	79,500	68,000	11	32	94,000	83,400	12.5	30
Extruded 50%, 6 hours 800°C†	66,100	40,300	16	26	76,000	52,000	21	38	67,600	39,600	23	36	75,500	52,000	22	41
Extruded 50%, 20 min 650 or 660°C††	75,500	54,500	20	37	74,000	55,000	19	33	73,000	50,000	21	38	76,000	55,000	23	45
As extruded 65%	92,600	78,500	7	20	101,900	87,000	10	23	84,600	70,800	11	36	95,500	80,200	12	37
Extruded 65%, 6 hours 800°C†	68,800	41,600	17	30	74,800	46,800	20	65	68,000	39,800	24	38	76,000	51,500	21	40
Extruded 65%, 20 min 650 or 660°C††	68,300	44,100	19	29	76,000	53,500	20	33	76,000	53,500	23.5	49	76,800	54,200	26	48
As extruded 80%	99,100	78,000	8	30	106,500	91,100	10	39	91,000	75,200	12	42	102,300	79,500	14	44
Extruded 80%, 6 hours 800°C†	67,400‡	41,200	22	30	76,000	49,300	22	40	68,200	40,200	24	41	72,600‡	48,200	20	28
Extruded 80%, 20 min 650 or 660°C††	78,000	54,500	25	46	77,000	53,500	24	45	76,800	54,000	25	50	75,750	53,700	24	45
As received	67,900‡‡	60,900	26		97,600	92,200	5	22								
As received, 6 hours 800°C†	63,800	36,300	22	32	75,600	53,700	20	42								
As received, 20 min 650 or 660°C††					75,200	56,400	21	39								

* Lot A contains <900 ppm O_2.
** Lot B contains 1200 ppm O_2.
† Less than 0.1-micron pressure.
†† In salt bath.
‡ Very large grain size.
‡‡ Wah Chang test.

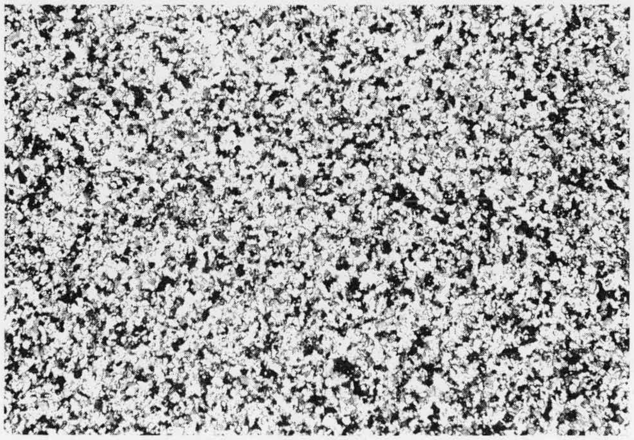
Ultimate = ultimate strength, psi
Yield = 0.2% offset yield strength, psi
Elong. = elongation in 2 in., %
R/A = reduction in area, %



As Extruded.



Annealed 6 hours at 800°C in Vacuum.



Annealed 20 minutes at 660°C in Salt.

Magnification: 100×

Etch: 20 ml HNO_3, 20 ml H_2O_2, 5 ml HF

EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE OF ZIRCALOY-2 AFTER 80% REDUCTION BY EXTRUSION AT 300°C

FIGURE 1

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