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THE FORMING OF ZIRCONIUM BY IMPACT EXTRUSION

by

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Abstract

Attempts to impact extrude bare zirconium blanks at room temperature were unsuccessful. Hot impact extrusion of bare zirconium blanks at about 500°C was more successful but still unsatisfactory. Hot extrusion of zirconium blanks jacketed in copper or aluminum was much more successful, and several satisfactory cans up to 8" long and with wall thickness as thin as 1/16" have been produced. Best results were obtained with a relatively slow extrusion speed of not more than 18 strokes/minute.

I. Introduction

A study was undertaken to investigate the adaptability of impact extrusion methods for the production of zirconium cans.

Deep drawing of cylindrical cans made from zirconium sheet have been successfully tried at Argonne National Laboratory. However, this process involves a considerable number of drawing passes and necessary intermediate annealing cycles. In the process of impact extrusion, the formation of a can would be accomplished by the force of a blow shaping the product to fairly accurate dimensions in less than 1/20th of a second.

Although the impact extrusion of soft light metals, notably aluminum, is a well-known and highly successful process, deterrent factors in the extension of this forming process to other stronger and more dense metals is the high force necessary for impact extrusion and the limitation imposed by physical properties of tool materials.

Impact extrusion imparts a very rapid "kneading process" to a metal slug. The slug of a suitable diameter and height, depending on shape of the extrusion desired, is placed in a die and hit by a punch. The metal flows under this impact. The punch is smaller in diameter than the die opening, thus providing an annulus into which the extruded metal flows. The metal is forced upward forming a "jacket" around the punch; the wall thickness is determined by the size of annulus between the die and punch. The original slug size and the stroke depth of the punch determines the height and bottom thickness of the extrusion.

II. Selection of Material - General Considerations

The zirconium slugs for impact extrusion used throughout this investigation were made of crystal bar (iodide refined) material. Zirconium of the Bureau of Mines type (Kroll method) has not been investigated beyond a few exploratory extrusions. Basically, conditions to be met as to uniform, fine, randomly oriented grains, high purity and low hardness are similar to those in aluminum for impact extrusion.

Considering at first the cold impact extrusion method, crystal bar zirconium was chosen primarily because of its relative softness, at least in some of the slugs. Extrusion slugs were machined from available flat annealed stock as well as from extruded metal homogenized by vacuum diffusion at 1200° - 1400°C. Several slugs were also machined of Bureau of Mines arc-melted zirconium for a general comparison as to grain size and extrudability. Metallographic examination of slugs of all three types has indicated the presence of small equiaxed grains approximately 0.03 - 0.04 mm in size. Typical microstructures of cast end of worked and recrystallized metal are shown in Figures 1 and 2. The banded structure shown in Figure 2 is difficult to eliminate completely.

In considering impact extrusion of heated zirconium slugs, the original advantages of soft crystal bar type material are no longer a major factor, but small grain size, good homogeneity, and low oxygen and nitrogen content of the metal were still of considerable importance. Hot extrusions were made from billets of arc-melted crystal bar zirconium, rolled into rods at 660° - 675°C. Intermediate annealing was carried out in a lead bath between rolling passes. After the final annealing at 500° - 550°C, the rod was water quenched. The hardness of the rolled rod from which slugs were machined was 45 - 48 Rockwell-B. All the hot extrusions discussed in this report were made from the same billet material described above.

III. Experimental Procedure

All impact extrusions were carried out on a 750-ton crank type press at a lower speed than normally used in the impact extrusion of aluminum. Aluminum, being a softer material, is extruded at approximately 30 strokes/minute. It was expected that this speed would be too high for zirconium; consequently lower speeds of 18 - 22 strokes/minute were employed. It was learned that 18 strokes/minute was the maximum permissible extrusion speed for zirconium. The material for the dies and punches was high carbon 12% chromium steel, hardened to 60 - 65 Rockwell-C.

Extrusion test slugs were 1.430" diameter by 0.770" in height and were coated with a mixture of tallow and cylinder oil. Extrusion was performed in two steps. To investigate the range of pressures needed for these materials, a SR-4 strain gauge was cemented to the punch and wired to an oscilloscope for automatic strain recording. A sample slug made of 2S aluminum of the same dimensions as the zirconium slugs served as a standard for the recording of pressures.

The maximum pressure during the process of cold extrusion depended upon the original hardness of the material and ranged from 160 - 320 tons. Hot impact extrusion of zirconium could be carried out at much lower pressures, i.e., 70 - 75 tons, as noted in Table I.

1. Cold Impact Extrusion - Cold impact extrusion of zirconium requires prodigious pressures which increase with increasing hardness of the blank (see Table I). It was possible to indent a slug (Figures 3 and 4) to some extent, but tool breakage was very high and circumferential cracks (Christmas-tree pattern) were very common. Severe discoloration of the outside surface of the extrusion indicated that high local temperatures were produced during the impact. Successive cold impacts deformed the metal to total rupture, the extruded can wedging itself between punch and die. It quickly became evident that even rather soft zirconium (R-B 25 to 38) could not be cold impact extruded satisfactorily and emphasis was shifted to hot impact extrusion.

2. Hot Impact Extrusion - In order to facilitate the flow of metal and to reduce impact pressures, slugs prepared from crystal bar zirconium were heated in a muffle furnace to 500° - 550°C and extruded at between 14 - 16 strokes/minute. Lubricant, applied to the die and punch, was a mixture of tallow and cylinder oil. Slug dimensions for hot impact extrusion were similar to the one used in cold extrusions. Impacting of the slug was done in one operation, resulting in the production of a can of 2" in height and approximately 1/8" in wall thickness. Although a general improvement over cold impact extrusion has been noted, a great deal of tearing and "Christmas-tree" patterning on the outside wall was noticeable. Slugs machined from Bureau of Mines type metal and initially of the same grain size showed the same effects.

The difficulty of tearing was not overcome despite small grain size and very slow extrusion speeds. Other lubricants (such as molybdenum sulfide, which was applied to the inside surface of the die and punch) did not alleviate the rippling effect on the outer surface of the extruded cans nor the cracking. An additional contributing factor to this defect is the increase in temperature of the metal by internal and external friction in the course of impact extrusion. This additional rise in temperature cannot be dissipated successfully, that is, speedily enough, to the surrounding mass of the die or the punch block assembly. It was localized apparently around

the bearing surface of the die; this action resulted in metal pickup from the hot slugs. Rippling and tearing of the outside surface was in almost all instances intergranular, i.e., the path of rupture was intergranular. The defects in bare slug extrusions could also originate in the method of heating the zirconium slugs. The slugs were heated in air, building up a surface scale of zirconia and probably hardening the surface metal by diffusion of oxygen and nitrogen to form solid solutions. Further, zirconium has a hexagonal close packed crystal structure and hence is anisotropic. Extruding zirconium in the beta phase, that is, in the body centered cubic structure, would have to be carried out above 860°C. This would necessitate heating the zirconium slug to 950 - 1000°C to cope with the heat loss in transferring the slug to the press.

An alternate and preferable method would be to enclose the zirconium blank with a copper or aluminum jacket and to extrude at moderate temperatures. The jacket would prevent oxidation of the zirconium during heating of the blank and would provide metal film lubrication of the tools during extrusion.

The slug blanks were encased completely in thin copper or aluminum jackets. Heating of the slug was carried out at 500°C, allowing for about 30 seconds to transfer the slug to the die. Extrusion was carried out at 18 strokes/minute. These extrusions resulted in smooth-walled cans which could be removed from the punch by a mechanical stripper. By using the jacketing technique for the impact extrusion of zirconium, ordinary cylinder oil served very successfully for lubrication of the die cavity and the punch. Copper or aluminum appeared to be equally satisfactory for the jacketing materials. The thickness of the jacket, however, had a considerable influence on the surface finish of the extruded can. Copper or aluminum jackets of 1/16" thickness were tried initially. These caused a bad patterning effect on the outside surface of the cans and folds at the bottom of the cans (see Figure 5). Thin jackets (1/32" thick) of either copper or aluminum were much more successful (Figures 6 and 7). Too high extrusion speeds causes tearing of the metal jacket (see Figure 6) and it is felt that the rate should be held below 18 strokes/minute for successful hot extrusion of even jacketed zirconium blanks.

Jackets of zirconium impact extrusion can be removed by conventional pickling methods, resulting in fairly smooth outside and inside surfaces. Successfully impact extruded zirconium cans have been produced in lengths up to 8" and in diameters up to 1.15" approximately. The initial experiments in hot extruding jacketed zirconium slugs produced wall thicknesses of 1/8"; by reducing the impacting stroke to 14 strokes/minute, it was possible to reduce the wall thickness to 1/16". If very careful aligning of the punch is carried out, a still further reduction in wall thickness might be possible.

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The height of the original blank for the intended extrusion is of course dependent on the ultimate length and wall thickness of the can desired; in the case of an 8" long can, 1/16" wall and 1" O.D., a blank about 3-1/2" long was required.

Typical microstructures of various parts of impact extruded cans are shown in Figure 8.

Table I

**HARDNESS AND PRESSURE REQUIREMENTS FOR THE IMPACT
EXTRUSION OF ZIRCONIUM**

Material	Hardness Rockwell-B	Impact Extrusion Pressure (Tons)	
		Cold	Hot (500 - 550°C)
Crystal bar (slugs machined from flat stock, annealed)	68 - 75 68 - 75	320	-
Crystal bar (slugs machined from extruded and homogenized rod)	25 - 38 ⁽²⁾	160 - 175	-
Bureau of Mines Metal, Arc Melted at ANL (slugs machined from rolled rod)	75 - 80	300 - 320	-
Arc-Melted Crystal Bar (slugs machined from hot rolled rod)	45 - 48	-	70 - 75 ⁽¹⁾
For "Standard" purpose: Grade 2S Aluminum (slugs machined from wrought stock) (same dimension size as Zr slugs)	35 - 45 ⁽³⁾	Ca. 45	-

(1) No noticeable variation in pressure requirements for extrusion of slugs of different heights.

(2) An increase in hardness to R_B 95-100 was observed in this Zr grade after cold impact extrusion. In the other grades only slight hardness increases were observed due to cold extrusion.

(3) Brinell, 500 kg load.

Figure 1 - Cast Structure of Crystal Bar Zirconium
Arc Melted at ANL for Impact Extrusion

Micro 12324

50X

Etchant: 5% HF/2% AgNO₃, Aqueous
Polarized Light

Coarse jagged-edge grains containing subgrains.

Figure 2 - Hot-Rolled Rod of Above Cast Billet

Micro 12325

50X

Etchant: 5% HF/2% AgNO₃, Aqueous
Polarized Light

Fine equiaxed grains, approximately 0.040 mm
(at 75X ASTM). Twinning and a nonrecrystallized
area may be seen parallel to the direction of
rolling and in the center of the rod.

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Figure 3 - Impact Extrusion Zirconium Blank

Macro 11714 1X
 Typical zirconium slug (1.430" dia. x 0.770")
 before impact extrusion.

Figure 4 - Cold Impact Extruded Zirconium Cans

Macro 11719

(1) Single impact, 3/8" in depth, (2) Slug No. 1 after second impact,
 (3) and (4) Can produced in one impact.

1X

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Figure 5 - Hot Impact Extruded Zirconium Cans
(Blanks jacketed in 1/16" Copper or Aluminum)

1
Macro 14940

2

3
Approx. 1X

Hot impact extruded zirconium cans. (1) Aluminum jacket partly removed, (2) Cross section of same can, and (3) Copper-jacketed zirconium can.

In this case the copper jacket was removed longitudinally to show bottom area more clearly. The thick apparent ring around bottom is a surface fold caused by a too heavy (1/16") jacket on the blank used for these extrusions.

Figure 6 - Hot Impact Extruded Zirconium Cans

1
Macro 14941

2
Approx. 3/4X

(1) Impact extruded zirconium can with copper jacket, and (2) Aluminum-jacketed can extruded at 22 strokes/minute causing torn wall. Slow extrusion speeds (up to 18 strokes/minute) are essential for impact extrusion of zirconium.

Figure 7 - Hot Impact Extruded Zirconium Cans

Macro 14942

Approx. 1/2X

Typical zirconium cans produced by impact extrusion. Second picture shows can with copper jacket. Third picture shows rippled surface due to looseness of copper jacket around the original slug; jacket was pickled off. First and fourth pictures show cans after jacket removal; here slugs were tightly jacketed.

Figure 8 - Microstructures in Impact Extruded Zirconium Cans

Figure 8-a - Top of Can Wall

Micro 12498

50X

Polarized Light ;
Etchant: 5% HF/2% AgNO₃

Figure 8-b - Can Bottom

Micro 12496

50X

Polarized Light
Etchant: 5% HF/2% AgNO₃

Figure 8-c - Junction of Can Wall and
Can Bottom.

Micro 12497

50X

Polarized Light
Etchant: 5% HF/2% AgNO₃