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**FACTORS AFFECTING PROPERTIES OF EXTRUDED
COMPACTS OF CRYSTAL BAR ZIRCONIUM.**

By

S. V. Arnold

**Massachusetts Inst. of Tech.,
Metallurgical Project**

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To : A. H. Kaufmann, Director, M.I.T. Metallurgical Project
 From : S. V. Arnold
 Subject: Factors Affecting Properties of Extruded Compacts of Crystal Bar Zirconium
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Six copper-clad billets of zirconium 1-1/4" D. x 3" compacted from 1/2" lengths of crystal bar under 130,000 psi. pressure at 900°C were extruded at the same temperature at 7.9:1, 14:1, and 31.6:1 reduction ratios. One extruded section at each reduction was quenched in cold water flowing through a guide tube immediately as it left the die orifice; another at each of these same reductions was allowed to cool in air.

Compacting technique followed that indicated as preferable by several experiments: Crystal bar zirconium from the Foote Mineral Company was sheared into 1/2" lengths and cold-compacted within steel cans the open ends of which were then fitted with discs and welded closed. The cans were next tapped, evacuated, refilled with argon and again sealed by welding. Hot compression at 900°C followed. The temperature and pressure promotes sufficient bonding of the crystal bar chunks to allow the compact to be stripped of its steel can and machined for recanning. Higher temperatures introduce difficulties through crumpling of the can during compacting. (It seems possible that techniques may be developed allowing higher compacting temperatures, which presently seem desirable. Low pressure, high temperature compacting within induction-heated graphite molds has been tried and appears promising. Complete bonding at 1500°C is possible but carbon diffusion into the surface metal introduces scrap loss and a machining problem in removal of contaminated metal.)

The compacted billets were canned in #16-gage copper for extrusion. (Copper possesses adequate ductility to serve as a jacket material, but extrusion temperature is limited by the 980°C copper-zirconium eutectic temperature. Steel does not have sufficient ductility for this application.)

Extrusion of the billets produced smooth, sound rounds of excellent quality. The rate of surface cooling during water quenching may have varied between the three reductions, since velocity of water flow and the speed of emission from the die were different in each instance. The mass of the different round sizes and the temperature to which they were raised by the energy expended in deformation also are factors affecting the cooling rate, both in water and in air.

It is best to discuss the microstructural characteristics of the extruded sections before their tensile properties, since the former help to explain certain peculiarities of the latter.

The interpretation of microstructures in these specimens has been difficult owing to lack of metallographic experience with zirconium, particularly after fabricating by these procedures.

Some features of the water-quenched rounds, however, seem to indicate the condition during and subsequent to extrusion. Of primary significance is the sharp demarcation between a very fine structure near the surface of these rounds and a coarser central structure (see Figure 1A). The outer zone consists mostly of very small, equiaxed grains and occasional patches of stratified metal showing the continuous varying gradations of reflectivity associated with a deformed lattice (or with grains just recrystallized from such a lattice and still retaining the parent orientation) as in Figure 1B. The outer structure showed smaller grain size nearer the surface, in some cases so fine as to be irresolvable.

The grain size inside the line of demarcation, though much coarser than that of the outer layer, is also finer nearer the outside and becomes larger toward the center of the round (see Figures 1C and 1D). Here large, irregular grains in the matrix were noted displaying continuous gradations of reflectivity suggesting that these too had not recrystallized. Acicular striations, normally straight, in certain areas appeared curved as by a continuous distortion of the lattice in further substantiation of the presence of unrecrystallized metal (see Figures 2A and 2B). The sharp demarcation between the outer and inner zones and the presence of unrecrystallized, yet dissimilar, grains in each are taken to indicate that two phases existed during extrusion. The pressure of extrusion would presumably favor maintenance of the closer-packed alpha phase. Slightly greater deformation plus surface friction would presumably raise the temperature of the outer metal above that of the inner, pointing to a beta phase condition in the outer portion of these rounds prior to cooling. The area of the outer zones comprised 70%, 72% and 76% of the extruded zirconium cross-section for the 7.9:1, 14:1 and 31.6:1 reductions respectively.

The air-cooled rounds retained very little of the core structure found in the water-quenched sections. Instead, the grain size increased gradually from edge to center in a fashion suggesting that the thermal energy of deformation had raised all but occasional, localized central areas to within the beta phase region. Recrystallization and grain growth proceeded until limited by cooling -- the outer grains which cooled first naturally being smaller than those further in.

In all microstructures, it must be stated, there seems little likelihood that any beta phase metal was retained to room temperature.

Unbonded interfaces were found over the entire cross section of the 7.9:1 reduced rounds, but for higher reductions lack of bonding was closely relegated to surface or near-surface interfaces, (which apparently incurred greater contamination, probably during hot compacting). Metal adjacent to unbonded interfaces sometimes showed marked differences in grain size from that of the surrounding matrix (see Figure 2C). Grain size varied between chunks of crystal bar to an extent suggesting variation in composition rather than other possible causes (see Figure 2D).

The properties of tensile specimens fashioned from the rounds are listed in Table I and presented graphically in Figure 3. Before an analysis of the results is attempted, it should be stated that specimens of 0.250" gage diameter were made from the 7.9:1 reduced rounds and specimens of 0.100" gage diameter from rounds of the two higher reductions. Circumstantially, in specimens from the water-quenched rounds these diameters included only the coarse-grained core structure. Metallographic examination suggested that this was alpha phase metal which largely recrystallized after extrusion. The air-cooled rounds are considered to have transformed almost completely to beta phase during and after extrusion, leaving only a small amount of centrally disposed metal possibly in the alpha condition prior to cooling. The tensile data therefore compare two different microstructures which may account in large part for the observed differences. Specimens from water-quenched rounds gave consistently higher ultimate strength and ductility than those from air-cooled rounds. The low reductions in area of specimens from 7.9:1 reduction ratio are attributable to the presence of unbonded interfaces which, since they were longitudinally

oriented, did not affect as adversely the elongation values.

There are scant data with which to compare these subject values, but Bureau of Mines metal similarly extruded at reductions of 14:1 and 31.6:1 and air cooled possessed approximately 68,000 psi. ultimate strength, 15% elongation and 45% reduction of area. This would indicate that the extruded compact is considerably softer and more ductile in elongation. However, the reduction of area is lower than that of the Bureau of Mines metal, a reflection perhaps on the extent of bonding between longitudinal interfaces of adjoining crystal bar chunks in the extruded compacts.

The most interesting aspect of the data is the decrease in properties which, if microstructural interpretation is valid, appears associated with transformation into the beta phase during extrusion. Owing to our rudimentary knowledge of zirconium behavior in fabrication and debatable possibilities suggested by the microstructures of the subject round, this phenomenon deserves further investigation before attempting any conclusions.

Table 1

Effect of Cooling Rate from Extrusion at 900°C on
Tensile Properties of Zirconium Crystal Bar Compact*

Specimen Number	Reduction ratio	Cooling Method	Ultimate Strength (psi.)	Elongation (%)	Reduction in Area (%)
942-1	7.89:1	Air	43,300	15.53	18.8
942-3	"	"	47,100	12.31	16.25
943-1	"	W.Q.	49,600	23.47	28.6
943-3	"	"	51,900	21.66	24.1
944-1	14.05:1	Air	42,800	22.5	41.8
944-3	"	"	46,400	20.2	41.6
945-1	"	W.Q.	55,300	26.0	46.5
945-3	"	"	53,300	26.5	50.6
946-1	31.6:1	Air	45,100	18.3	33.3
946-3	"	"	48,300	19.0	38.8
947-1	"	W.Q.	55,000	23.8	56.2
947-3	"	"	55,000	18.8	56.4

- * Tensile specimens from material reduced 7.89:1 were machined to Dwg. 1461-1 with gage 0.250" D. x 1". Tensile specimens from material reduced 14.05:1 and 31.6:1 were machined to Dwg. 1671-1 with gage 0.100" D. x 1".

(A) Demarcation between Outer (Above) and Inner Zones. 14:1 Reduced Round, Water Quenched. Longitudinal Section.

(B) Continuous Gradations of Reflectivity in Stratified Metal of Outer Zone, Presumably Unrecrystallized.

(C) Grain Size at Outer Portion of Inner Zone in 14:1 Reduced Round, Water Quenched. Longitudinal Section.

(D) Grain Size at Center of Inner Zone. Same Round as in (C). Longitudinal Section.

Figure 1. Photomicrographs of Zirconium Crystal Bar Compacts Extruded at 900°C and Water Quenched. 250X Magnification Under Polarized Light.

(A) Coarse Grains at Center of Inner Zone Showing Continuous Gradations in Reflectivity and Curved Striae Suggestive of Unrecrystallized Metal. 14:1 Reduced Round, Water Quenched.
Transverse Section 250X

(B) Curved Striae, Same Section as in (A).
Bright Light 1500X

(C) Variations in Structure Adjacent to Unbonded Interface, 14:1 Reduced Round, Water Quenched.
Transverse Section 250X

(D) Difference in Grain Size between Adjacent Chunks of Crystal Bar in 31.6:1 Reduced Round, Air Cooled.
Transverse Section 250X

Figure 2. Photomicrographs of Extruded Zirconium Crystal Bar Compacts, Most Under Polarized Light.

FIG. 3

END