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SUBJECT: SUGGESTED FABRICATION PROCEDURES FOR ZIRCALOY-2 MILL PRODUCTS IN INGOT QUANTITIES

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ABSTRACT

Suggested fabrication procedures for Zircaloy-2 sheet, plate, rod, and bar are presented. These procedures are based upon the best present knowledge of the physical and mechanical metallurgy of Zircaloy-2 and are designed to produce material with a minimum amount of preferred orientation, anisotropy of mechanical properties, and intermetallic stringers. The recommended procedures cover ingot soaking, fabrication, heat treatment, finish, workmanship, identification, and inspection.

A brief discussion of the physical and mechanical metallurgy of Zircaloy-2 is presented in order to explain the particulars of the working and heat treatment schedule chosen.

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SUGGESTED FABRICATION PROCEDURES FOR ZIRCALOY-2
MILL PRODUCTS IN INGOT QUANTITIES

INTRODUCTION

The following is intended only as a statement of the recommended fabrication procedure for mill products made from ingot quantities of Zircaloy-2. It does not constitute a specification. Specifications: ASTM B349 – 60 T, B350 – 60 T, B351 – 60 T, and B352 – 60 T should be referred to for specifications of size and straightness tolerances, finish, ingot preparation and conditioning, etc., where appropriate. It should be pointed out that manufacturers will not normally allow a specification of both procedure and product.

Studies of the effects of fabrication variables have shown that the preferred orientation and anisotropy of mechanical properties and strain behavior of Zircaloy-2 vary greatly with the exact fabrication procedure.[1,2] Although a particular degree and type of anisotropy of plastic properties is desirable for some applications, more generally an isotropic material is needed. A fabrication procedure which yields Zircaloy-2 with properties approaching those of an isotropic material has been described and discussed previously.[1–3] It is essentially this fabrication procedure which will be detailed as a suggested fabrication procedure for Zircaloy-2.

In order to follow intelligently the working and heat treating particulars of the fabrication procedure, it is worthwhile to review some of the facets of the physical metallurgy of the alloy. Zircaloy-2 is a sponge zirconium-base alloy with nominal additions of 1.5 wt % Sn, 0.1 wt % Fe, 0.1 wt % Cr, 0.05 wt % Ni. Although the tin tends to stabilize the low temperature hexagonal close-packed α phase, the minor alloying additions lower the $\alpha/\alpha+\beta$ temperature to 1490–1500°F for material containing 40 to 50 ppm H_2.[(ref 3)] Thus the range of the $\alpha + \beta$ phase field is from 1490 to 1780°F. Because equilibrium partitioning of the alloying elements occurs fairly rapidly at temperatures in the two-phase field, the time that the material is held between the temperatures of 1490 and 1780°C should be limited to a minimum. Holding and fabricating Zircaloy-2 in the temperature range of 1490 to 1580°F will result in the migration of iron, nickel, and chromium to the β phase in the grain boundaries and the consequent formation of intermetallic stringers there on decomposition of the β phase at or below 1490°F. The stringers of the Zr-Sn-Fe-Ni-Cr intermetallic phase are objectionable as they: (1) add to the directionality of mechanical properties;[3,4] (2) are associated with stringer-type corrosion;[5] and (3) serve as nuclei for void formation in the heat-affected zone of weldments.[6] Holding Zircaloy-2 in the temperature range of 1725 to 1780°F results, in time, in the partitioning of the major portion of the tin and oxygen present to the α phase, and, therefore, in the production of hard particles (α grains high in oxygen content) which are not eliminated during the fabrication which follows. Thus it is critical that all soaking be performed at temperatures above 1780°F or below 1490°F.

The major mode of deformation of α-phase Zircaloy-2, slip on the system $\{10\bar{1}0\}$ $\langle\bar{1}2\bar{1}0\rangle$, forces the development of a high degree of preferred orientation in α-worked material. Even though $\alpha/\alpha+\beta$ transformation is part of the usual fabrication schedule for Zircaloy-2, the transformation orientation relationships between the α and β phases are such that an α grain transformed to the β phase and then back to α tends to retain its original crystallographic orientation. The preferred orientation of the finished plate is such that there should be little ductility in compression in the direction normal to the plane of the plate, but good ductility for both tension and compression in directions in the rolling plane. Round tensile specimens of this material show elliptical cross sections after straining. The major axis of the ellipse occurs in the thickness direction of the plate for both the longitudinal and transverse direction specimens, confirming that little ductility is exhibited across the thickness of the plate in compression.

Very small needles of α phase are formed when Zircaloy-2 is water quenched from temperatures in the β-phase field. Cold working by 25% reduction in thickness or area provides a sufficient number of randomly oriented nuclei to allow recrystallization into an almost randomly oriented fine-grained α structure on annealing at 1375–1450°F. The properties of Zircaloy-2 plate having received this treatment approach those of an isotropic material. In commercial practice, one must generally settle for no more rapid a quench than that afforded by a water spray or forced air draft. The needles formed on the quench will then be considerably larger than those obtained by water quenching, the needle size increasing appreciably with increasing section thickness and decreasing quenching rate. Thus, a greater reduction is necessary to give the minimum number of nuclei which will yield the desired structure on annealing. For material 1/4-in. thick and greater, reductions of 30 to 40% are needed, while 25 to 30% reduction is still sufficient for material less than 1/4-in. thick. Final reduction of greater than 40% is undesirable as it tends to nullify the randomization of texture accomplished by the β heat treatment, quench, and cold working.

The following fabrication procedures have been written in such a manner as to take advantage of the above knowledge of the physical and mechanical metallurgy of Zircaloy-2. Allowances have been made, in so far as possible, for the limited flexibility and availability of production equipment suitable for fabrication of the sizes of material desired.

Fabrication Procedures for Zircaloy-2 Sheet, Plate, Rod, and Bar

Vacuum melted ingots prepared according to ASTM Specification B350 – 60 T shall be used.

1. Ingot Soaking

   1.1 The ingot shall be charged into a hot furnace having an atmosphere that is neutral to oxidizing but never reducing. The ingot shall be held at 1900 to 1950°F for not less than 30 min nor more than 60 min after the time it is estimated that the core of the ingot has reached temperature.

   1.2 In no case shall the work piece be held or soaked at temperatures between 1475 and 1750°F and passage of the work piece through this temperature range shall be as rapid as possible since partitioning of the alloying elements occurs in the two-phase field defined by these temperatures.

1.3 At no time shall the work piece be permitted to remain in contact for more than a very few minutes with copper and copper alloys above 1600°F, or with iron, nickel, and their alloys above 1700°F because of the eutectics formed with these materials at temperatures greater than those stated.

2. Fabrication

2.1 Ingot breakdown: The ingot breakdown may be by either forging or rolling. In either case, the maximum starting temperature shall be 1950°F and the minimum starting temperature shall be 1900°F.

2.1a The minimum forging temperature for any major amount of working shall be 1750°F, with reheating to 1900–1950°F required should the surface temperature fall below 1750°F. Surface patting to smooth the surface of the slab or billet and to provide final sizing shall not be construed as major working, and may occur at temperatures below 1750°F provided the work piece cools during such working at approximately the rate it would in still air with no working.

2.1b If the ingot breakdown is by rolling and the entire operation can be completed within a period of five minutes, no minimum temperature during fabrication shall be specified but the amount of working at temperatures below 1700°F shall be as small as possible. The minimum temperature for working shall be 1750°F if the breakdown cannot be completed within five minutes, with reheating to 1900°F required if the surface temperature should fall below 1750°F.

2.2 Major Reduction

2.2a Sheet and plate: The major reduction in size after the ingot breakdown shall be accomplished at temperatures of the work piece between 1750 and 1950°F or between 1200 and 1450°F, depending on the type and amount of reduction required. Part of the major reduction may be made in the higher temperature range and the remainder of the reduction in the lower temperature range. Any cross rolling required to obtain the necessary widths shall be made during the early stages of the major reduction or during the ingot breakdown. The major reduction step shall end at a thickness of not less than 125% and not more than 140% of the finished thickness specified in the order, with proper allowance being made for surface conditioning and pickling losses.

2.2b Rod and bar: The major reduction in size after the ingot breakdown shall be accomplished at temperatures of the work piece of above 1750°F and below 1950°F or above 1200°F and below 1450°F, depending on the method of reduction. The major reduction may be accomplished by press forging, hammer forging, drawing, rolling, or any combination of these. All hammer forging <u>must</u> be done at temperatures above 1750°F, reheating at any time the surface temperature of the work piece falls to 1750°F, except for finishing pats in sizing or surface smoothing. All drawing must be accomplished at or below 1450°F, and all jacketing material must be <u>completely</u> removed before heat treatment at any temperature above 1450°F. Press forging and rolling shall be done at temperatures above 1750°F as much as possible. If the temperature of the material cannot be maintained within the limits without excessive amounts of reheating, reduction at 1200 to 1450°F shall be permissible. Thorough working of the center of the work piece is a requirement.

The major reduction shall end at a cross-sectional size such that not less than a 25% nor more than a 40% reduction in cross-sectional area need be made in the finishing fabrication.

2.3 Surface and Edge Conditioning: Following the major reduction and before β heat treatment, all surfaces of the work piece shall be examined for defects. Edge cracking shall be removed by shearing; pits and inclusions by chiseling and grinding to fair or by grinding and pickling to insure that the defect has been removed.

2.4 Finishing fabrication: Following the β-quench heat treatment described in 3.1, the work piece shall be reheated to a temperature of not less than 700°F and not more than 1200°F. For sheet material less than 1/4-in. thick and for rod and bar of less than 1/2 in., a reduction of not less than 25% nor more than 30% is required. For heavier sections, a final reduction of not less than 30% nor more than 40% is required.

3. Heat Treatment

3.1 β-quench heat treatment: After completion of the major reduction step and conditioning, the work piece shall be placed in a preheated furnace with neutral to slightly oxidized atmosphere at 1800 to 1850°F. The work piece shall be held at temperature for not less than 20 min nor more than 45 min and cooled very rapidly to a temperature below 1400°F, preferably by water quenching. The work piece must reach the water quench bath at a minimum temperature of 1650°F. If water quenching facilities are not available, forced air cooling may be used. The forced air draft must reach both sides of the work piece. The cooling rate between the temperatures of 1800 and 1400°F must produce a reasonably fine acicular α structure in the material.

3.2 Anneal: Following the final working operations, the work piece shall be annealed at a temperature of not less than 1375°F nor more than 1450°F for not less than 30 min and not more than 45 min at temperature. The work piece shall then be cooled rapidly to room temperature by water quenching if practical.

4. Surface Finish and Edge Condition

4.1 The final product shall be sand, grit, or vapor blasted to remove the oxide scale and then pickled. The pickling operations at all times shall be performed in such a manner as to minimize hydrogen pickup.

4.2 Shearing to final sizes may be done at room temperature provided a test shearing is made to insure that there will be no edge cracking. Should the test shear show edge cracking, shearing at or above 500°F shall be required.

5. Workmanship and Fabrication History

5.1 It shall be the responsibility of the fabricator to follow the exact procedure outlined and to report in detail any departure from the procedure permitted in this specification and authorized by a company observer present. The fabricator will exercise the best mill practices in endeavoring to supply material free from defects of any nature.

5.2 A complete history of the fabrication process from ingot breakdown to finished products, including temperatures, atmospheres, reheating cycles, reduction schedules, and descaling and pickling temperatures shall be supplied to the purchaser.

6. Identification

6.1 The identity of all materials with respect to ingot melt number and location on ingot, top or bottom, shall be maintained at all stages of fabrication of material.

6.2 All plate, sheet, bar, and rounds greater than 1/2 in. in diam shall be marked with purchase order number and ingot melt number and location. Rounds smaller than 1/2 in. in diam shall be bundled and tagged.

6.3 All scrap shall be identified as to its stage of fabrication and returned to the purchaser.

6.4 Indentation stamping of finished material is prohibited.

7. Inspection

7.1 The company shall have the right to have a qualified observer follow all phases of fabrication. The observer shall have access to any portion of the seller's plant in which the work is being conducted. It shall be the responsibility of the seller to notify the purchaser sufficiently in advance (one week if possible) of fabrication of each separate portion of an order so that the observer may be present.

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