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FOR ZIRCALOY-2

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DEVELOPMENT OF A FABRICATION PROCEDURE FOR ZIRCALOY-2

M. L. Picklesimer and G. M. Adamson
Metallurgy Division
Oak Ridge National Laboratory

Summary

The observation of stringers occurring in the plane of the plate in Zircaloy-2 fabricated according to the present fabrication practice of rolling from 1550°F (843°C) and of their disappearance upon heating into the all beta field (above 1780°F, 970°C) led to the initiation of an investigation in fabrication procedure with the objective of the elimination of the stringers. The presence of stringers affects the mechanical properties of the material and, since Zircaloy-2 is somewhat notch sensitive at and near room temperature, could cause premature failure in stressed applications such as pressure vessels.

A new fabrication procedure has been developed which eliminates the stringers usually found and which decreases the amount of preferred orientation present in the fabricated plate. The procedure consists essentially of fabricating either above 1780°F (970°C) or below 1490°F (810°C) with a finishing process consisting of heating to 1832°F (1000°C) or higher for approximately 30 minutes at temperature, water quenching if possible, air cooling rapidly if water quenching is not possible, cold or "warm" rolling (less than 500°C) with not less than 20% reduction, and annealing at 800°C or less for 15 to 30 minutes at temperature. The resulting material has no stringers, a grain size of ASTM 7 to 8, and much less preferred orientation resulting in more isotropic mechanical properties.

This new procedure was determined by rolling plate material. The process should apply equally well to swaged or rolled rod material.

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DEVELOPMENT OF A FABRICATION PROCEDURE FOR ZIRCALOY-2

M. L. Picklesimer and G. M. Adamson
Metallurgy Division
Oak Ridge National Laboratory

This memorandum is issued as a partial and preliminary report on an investigation of the morphology of Zircaloy-2 in order to provide an early release of part of the data obtained so that those interested in the engineering mechanical properties and fabrication of Zircaloy-2 can be made aware of an improved fabrication procedure. A full report on the morphology of Zircaloy-2 will be issued later.

Introduction

An investigation of the morphology of Zircaloy-2 was undertaken by the HRP Physical Metallurgy Group in order to provide a background of experience to permit the interpretation of weld structures, radiation damage effects, and mechanical property variations, and to find the optimum condition of the material for future use in reactor applications. Specimens were given a wide variety of heat treatments to give various microstructures. The data determined gave information about the rates of grain growth, the limits of the alpha and beta phase fields, the appearance of the microstructures resulting from known heat treatments, the amount of preferred orientation resulting from fabrication practice, and some mechanical properties as correlated to microstructures and heat treatment.

One result of the investigation was the observance of the disappearance of the stringers present in the "as-received" material when the material was heated into the all beta field (above 1780°F, 970°C). This indicated that the stringers were, in all probability, intermetallic compounds. The "as-received" material has been rolled from 1550°F (843°C), the present standard practice. Since the

alpha/alpha plus beta temperature had been determined during this investigation to be 810°C (1490°F), it was reasoned that holding the material at 1550°F (843°C) for a period of time before rolling would result in the presence of some beta phase in the grain boundaries and grain corners. Since Fe, Ni, and Cr, which are present as alloying elements, are beta stabilizers, they would diffuse to the beta phase present, concentrating those elements in the grain corners and grain boundaries. During fabrication, the beta phase present would be strung out in stringers or plates between the alpha grains, the major constituent present. As the material cooled below the decomposition temperature of the beta phase, the beta phase would decompose into alpha zirconium (deposited on the neighboring alpha grains) and the Fe, Ni, and Cr intermetallics with Zr which would be left in the stringer or plate pattern of the prior beta phase. This would result in the stringers and plates observed.

A micrograph of the "as-received" material is shown in Fig. 1. Broken stringers are observable since they serve as common grain boundaries for several grains. A cathodically etched specimen is shown in Fig. 2 where the stringer is shown to have metallic continuity with the matrix and is not a void as has been postulated. The presence of the beta phase in the grain corners and grain boundaries is shown in Fig. 3, the micrograph of a specimen held at 840°C and water quenched. All of the grain boundary material observed in Fig. 1 has dissolved to form the beta phase, giving the appearance of a much "cleaner" structure. The microstructure of a beta quenched specimen is shown in Fig. 4. The specimen was water quenched after 30 minutes at 1050°C in argon. The remainders of the stringers that were present can be seen as the centers of the "star" structures. The microstructure of a specimen air cooled from 1050°C is shown in Fig. 5. The dot precipitate

(a)

(b)

Fig. 1 Zircaloy-2 "As-Received", Chemically Polished, Anodized
Zr-2-1 500X
(a) Bright Field Y-18558
(b) Polarized Light Y-18559

995-005

(a)

(b)

Fig. 2 Zircaloy-2 "As-Received", Cathodically Etched, Anodized
Zr-2W-1 500X

(a) Bright Field Y-18297

(b) Polarized Light Y-18298

995-006

Fig. 3 Zircaloy-2, 840°C 30 min., Water Quenched, Chemically
Polished, Anodized. Zr-2-58 200X
Bright Field Y-17721

995-007

(a)

(b)

Fig. 4 Zircaloy-2, 1050°C 30 min., Water Quenched, Chemically Polished, Anodized.

(a) Zr-2-2 100X Y-17768
(b) Zr-2-2 500X Y-17769

995-008

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Fig. 5 Zircaloy-2, 1050°C 30 min., Air Cooled, Chemically
Polished, Anodized.
Zr-2-3 500X Polarized Light Y-17771

995-009

appearing in the boundaries between the alpha needles is probably a mixture of the Fe, Ni, Cr, and Sn intermetallics with Zr. All metallographic specimens were chemically polished and anodized.

Experimental Procedure and Results

It was reasoned that, since the stringers dissolved on heating into the beta field, it would be possible to eliminate the stringers by beta quenching or beta treating and air cooling, and annealing the resultant alpha needle structure with recrystallization and grain growth giving a new equi-axed alpha grain structure. The structures resulting from beta quenching and alpha annealing are shown in Figs. 6 and 7 for specimens annealed 2 hours at 700°C and 15 minutes at 800°C, respectively. The procedure eliminated the stringers but resulted in a very coarse final grain size. Beta quenched and beta air cooled specimens were then cold rolled 10, 15, and 20% and annealed at 800 to 830°C for 15 minutes in the furnace. The structures are shown in Figs. 8 and 9. The degree of preferred orientation for the specimen beta quenched, cold rolled 20%, and annealed at 800°C for 15 minutes was determined and compared to that of the "as-received" material. The experimental results showed that beta quenching, rolling 20%, and annealing resulted in a very fine grained structure with much less preferred orientation than the starting material. Air cooling resulted, always, in a coarser grain size than water quenching, and cold reductions of 10 and 15% resulted in very large grain sizes. Thus it was concluded that a minimum of 20% cold reduction was necessary, that "warm" rolling from 500°C was satisfactory, that beta quenching was preferable but that air cooling rapidly from the beta field was acceptable, that the temperature of annealing was not too critical but should be 800°C or less, that annealing for 15 minutes at 800°C was sufficient for complete recrystalli-

Fig. 6 Zircaloy-2, 1000°C 30 min., Water Quenched, 700°C 2 hr.,
Water Quenched, Chemically Polished, Anodized.
Zr-2-45 100X Polarized Light Y-18568

995-011

Fig. 7 Zircaloy-2, 1000°C 30 min., Water Quenched, 800°C 15 min.,
Water Quenched, Chemically Polished, Anodized.
Zr-2-51 100X Polarized Light T-9355.

995-012

(a)

(b)

Fig. 8 Zircaloy-2, 1000°C 30 min., Water Quenched, Cold Rolled
Annealed 800°C 15 min., Water Quenched, Chemically
Polished, Anodized. 100X Polarized Light
(a) Zr-2-88 Cold Rolled 10% Y-18570
(b) Zr-2-84 Cold Rolled 20% Y-18569

995-013

(a)

(b)

Fig. 9 Zircaloy-2, 1000°C 30 min., Air Cooled, Cold Rolled,
Annealed 800°C 15 min., Water Quenched, Chemically
Polished, Anodized. 100X Polarized Light
(a) Zr-2-92 Cold Rolled 10% Y-18573
(b) Zr-2-93 Cold Rolled 20% Y-18575

995-014

zation, that the amount of preferred orientation could be decreased markedly by beta quenching, rolling, and annealing, and that the stringers observed could be removed by this treatment.

Subsize Izod notched impact specimens of Zircaloy-2 in the "as-received" condition (see Fig. 1) were held in evacuated quartz capsules at 800, 810, 820, and 830°C for 15 minutes and at 830°C for 5 minutes and air cooled. The impact transition curves were determined and the resulting data are shown in Fig. 10. The specimens held above 810°C had much lower impact strengths, higher transition temperatures, and were embrittled by the heat treatment. Microstructural examination showed the presence of a grain boundary constituent in the specimens held above 810°C. Comparative microstructures for the 810 and 830°C specimens are shown in Fig. 11.

Several Zircaloy-2 plates have been fabricated to the new procedure by commercial fabricators, but the finished plates have not been received. Tensile stress-strain curves and impact curves will be determined on this material shortly after it is received.

No fabrication work has been done with forged, swaged, or rolled rod material but the new fabrication procedure would be expected to apply equally well to these processes. Morphological studies made on Zircaloy 3a, 3b, and 3c indicate that the procedure reported for Zircaloy-2 would apply to these alloys as well with no change in the recommended temperatures.

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Y-20400

Fig. 10 Impact Energy Curves for Heat Treated Zircaloy-2

910-566

(a)

(b)

Fig. 11 Zircaloy-2 750X Bright Field Chemically Polished, Anodized

(a) Zr-2B-11 810°C 15 min., Air Cooled Y-19966

(b) Zr-2B-17 830°C 15 min., Air Cooled Y-19972

995-017

Conclusions

It was concluded that a fabrication procedure for Zircaloy-2 which would result in a stringer-free, fine grained, and less oriented material was as follows:

1. All soaking for forging or rolling, all forging, and all rolling should be done either above 970°C or below 810°C to eliminate the presence of an alpha plus beta structure during fabrication and that no soaking for any period of time should be done between 810 and 970°C.
2. After the major reduction in size is made, the material should be heated to not less than 970°C for approximately 30 minutes at temperature and water quenched by immersion or spray, or air cooled rapidly to below 700°C.
3. Cold or "warm" (from 500°C or less) reduction of not less than 20% in thickness or area should be made, on the quenched material, to finished size.
4. The worked material should be annealed at 780 to 800°C for approximately 15 minutes at temperature.
5. The annealed material may be air cooled or water quenched to room temperature.

Recommendations

It is recommended that, henceforth, all Zircaloy-2 be fabricated using the above procedure. It is believed that this procedure should also be used for fabrication of Zircaloy 3a, 3b, and 3c with no changes in the temperature recommended for Zircaloy-2.

A complete report on the morphological study of Zircaloy-2 will be issued in the near future.

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