HEAT TREATMENT OF THE ALLOY

Zr-2.5 wt% Nb

T. E. Michaels

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By

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TABLE OF CONTENTS

LIST OF FIGURES.	iv
LIST OF TABLES	v
ABSTRACT	1
INTRODUCTION.	1
SUMMARY AND CONCLUSIONS	1
DISCUSSION	2
Background Material	2
Microconstituent Survey	5
Recrystallization Effects	9
Isothermal Transformation Characteristics	11
Aging Characteristics.	14
Tensile Properties.	17
ACKNOWLEDGEMENT.	19
BIBLIOGRAPHY.	19
DISTRIBUTION.	20

LIST OF FIGURES

1	<i>Equilibrium Phase Diagram for the Zirconium-Niobium Alloy System</i>	3
2	<i>Knoop Hardness of Zr-2.5 wt% Nb as a Function of Solution Heat Treatment Temperature</i>	6
3	<i>Zr-2.5 wt% Nb Heat Treated 1000 °C for 6 hr, Water Quenched.</i>	6
4	<i>Zr-2.5 wt% Nb Heat Treated 750 °C for 6 hr, Water Quenched.</i>	6
5	<i>Zr-2.5 wt% Nb Furnace Cooled from 1000 °C to 600 °C, Water Quenched.</i>	7
6	<i>Zr-2.5 wt% Nb Furnace Cooled from 1000 °C to 800 °C, Water Quenched.</i>	7
7	<i>Zr-2.5 wt% Nb Solution Heat Treated at 850 °C for 6 hr, Water Quenched.</i>	8
8	<i>Partial Equilibrium Phase Diagram for Zr-Nb Alloy System</i>	8
9	<i>Specimen Orientation Nomenclature</i>	10
10	<i>Recrystallization Results Showing Grain Size versus Solution Heat Treatment Temperature for 60% Cold Worked Zr-2.5 wt% Nb</i>	11
11	<i>850 °C Heat Treatments on 60% Cold Worked Zr-Nb Pressure Tubing to Study the Recrystallization Characteristics</i>	12
12	<i>Plot for the Determination of the Activation Energy for Secondary Recrystallization</i>	13
13	<i>Hardness Data for Isothermal Transformation Heat Treatments</i>	15
14	<i>Isothermal Transformation Diagram for Zr-2.5 wt% Nb</i>	15
15	<i>Isothermal Heat Treatments Performed at 750 °C.</i>	16
16	<i>Aging Characteristics of Zr-2.5 wt% Nb</i>	18
17	<i>Tensile Properties of Zr-2.5 wt% Nb as a Function of Solution Heat Treatment Temperature</i>	18

LIST OF TABLES

I	<i>Chemical Composition of Zr-2.5 wt% Nb Pressure Tubing</i>	4
II	<i>Heat Treatments Performed for Microconstituent Survey on Zr-2.5 wt% Nb Alloy</i>	5
III	<i>Knoop Hardness of Zr-2.5 wt% Nb for Different Heat Treatments</i>	5
IV	<i>Heat Treatments Performed for Recrystallization Study of 60% Cold Worked Zr-2.5 wt% Nb</i>	9
V	<i>Results of Recrystallization Study Performed on Zr-2.5 wt% Nb, 60% Cold Worked</i>	10
VI	<i>Heat Treatments Performed on Zr-2.5 wt% Nb to Study Isothermal Transformation Characteristics</i>	13
VII	<i>Knoop Hardness of Zr-2.5 wt% Nb as a Function of Isothermal Heat Treatment Temperature and Time</i>	14
VIII	<i>Aging Treatments Performed on Heat Treated Zr-2.5 wt% Nb Specimens</i>	17
IX	<i>Results of Aging Study</i>	17
X	<i>Heat Treatments Performed on Tensile Samples of Zr-2.5 wt% Nb</i>	19

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ABSTRACT

Heat treatment effects on Zr-2.5 wt% Nb are described. Five areas were studied qualitatively: (1) Microconstituent survey, (2) Recrystallization, (3) Isothermal Transformation Characteristics, (4) Aging Characteristics, and (5) Tensile properties as a function of solution heat treatment temperature.

INTRODUCTION

As part of the USAEC-AECL Cooperative Program the heat treatment characteristics of the Zr-2.5 wt% Nb alloy were studied to determine the feasibility of heat treating this alloy to improve its mechanical properties over the 30% cold worked condition. The topics studied included a microconstituent survey, recrystallization, isothermal transformation characteristics, aging, and tensile properties. The results are presented in this report. From the data, conclusions will be made relating to the heat treatment feasibility of this alloy.

The use of Zircaloy-2 by the United States and Zr-2.5 wt% Nb by Canada as reactor pressure tube material has led to a useful exchange of information and cooperative development efforts related to these two alloys. One example is the USAEC-AECL Coopera-

tive Program on the Zr-2.5 wt% Nb alloy evaluation. It is under this program that much of the Zircaloy-2 technology accumulated in the U.S. is being related to the Zr-Nb alloy.

SUMMARY AND CONCLUSIONS

Heat treatments were performed on the Zr-2.5 wt% Nb alloy to survey the microconstituents, the recrystallization behavior of the 60% cold worked material, isothermal transformation characteristics, aging response, and tensile properties as a function of heat treatment.

The microconstituent survey and the recrystallization study showed that it is possible to establish equilibrium within a few hours for the range of solution heat treatment temperatures (700 to 1000 °C) studied. The activation energy for recrystallization was calculated as approximately 37 kcal

per mole for a 60% cold worked condition. The recrystallized grain size was estimated as 3.1 μ .

The isothermal heat treatments show evidence of a coherent omega precipitate formation, which strengthens the alloy substantially. The formation time is too rapid for this strengthening mechanism to be applicable to the heat treatment of large samples. The isothermal diagram shows that, due to the rapid transformation kinetics on isothermal holding, fast quenching rates are required to produce the martensitic component α' from β .

The aging study showed only possible evidence of a secondary hardening effect. This was observed after 100 hr of aging on a sample initially solution-heat-treated at 700 °C followed by a water quench.

The tensile properties of samples of various solution heat treatment conditions showed the 850 °C solution heat treatment as having the largest factor of yield strength times ductility. This solution heat treatment is near the top of the $\alpha + \beta$ field for the Zr-2.5 wt% Nb alloy.

In conclusion, the mechanical properties of the Zr-2.5 wt% Nb alloy change notably with heat treatment. The optimum heat treatment appeared to be 850 °C for 6 hr followed by a water quench. For this heat treatment, aging produced no secondary hardening effect for aging at 500 °C for durations up to 100 hr, but aging is useful in stabilizing the mechanical properties of the specimen after solution heat treatment.

DISCUSSION

BACKGROUND MATERIAL

Niobium stabilizes the β (body centered cubic) phase of zirconium. A phase diagram for the Zr-Nb alloy system, Figure 1, shows a monoeutectoid reaction occurring at 20.5 wt% niobium. This reaction is sluggish to the extent that the monoeutectoid microconstituent is rarely observed in heat treated sections. This is due to the slowing down effect that niobium in solid solution has on the β to α reaction in zirconium. Due to very slow reaction rates, it is not feasible to heat-treat alloys of high niobium content. The 2.5 wt% Nb alloy is of low enough niobium concentration that equilibrium can be established in a few hr at solution heat treatment temperatures.

Slow cooling from the β to $\alpha + \beta$ field promotes the transformation of β to α in a mass diffusion type of process. A transition phase commonly called omega, ω , is formed as an intermediate step. During its formation this ω phase maintains coherency with the body centered cubic β phase. This coherency results in strengthening the alloy. As the transformation process continues, ω undergoes a transition to α , the low temperature hexagonal close packed structure of zirconium. The exact nature of the formation and decomposition of ω is not completely understood by investigators. The Bibliography illustrates some investigations.

FIGURE 1. Equilibrium Phase Diagram for the Zirconium-Niobium Alloy System

A martensitic product α' is formed by a shear type reaction as β transforms to α' for specimens quenched from the β or $\alpha + \beta$ field to below the martensite start temperature, m_s . Alpha prime has a hexagonal close

packed structure like α but is richer in niobium. The solid solution strengthening effect of niobium in zirconium is responsible for α' having higher strength than α .

Age hardening is complicated in the Zr-Nb alloy system, and many theories exist. Williams and Gilbert have advanced a theory of age hardening based on the formation of a niobium rich precipitate from the martensitic microconstituent α' . Their experimental evidence shows coherency of such a precipitate is directly related to the maximum hardness of age hardening curves. The fact that a slow cooled

microstructure will not respond to aging heat treatment also supports the theory of Williams and Gilbert since slow cooling produces no α' microconstituent.

Increasing the oxygen interstitial content tends to raise the $\beta/\alpha + \beta$ phase boundary and raises the martensite start temperature. The impurity concentration of the material studied is listed in Table I.

TABLE I. Chemical Composition of Zr-2.5 wt% Nb Pressure Tubing

Impurity	Impurity Content, ppm		
	Outside Surface	Center	Inside Surface
H	13	-	13
O	860	-	795
N	21	30	29
C	105	125	115
Fe	500	670	460
Cr	<50	<50	<50
Ni	<50	<50	<50
Sn	<50	<50	<50
Al	25	-	26
B	< 0.25	-	< 0.25
Cd	< 0.25	-	< 0.25
Co	<10	-	<10
Cu	<20	<20	<20
Hf	54	-	64
Pb	<20	-	<20
Mg	<20	-	<10
Mn	<25	-	<25
Mo	<20	-	<20
Si	11	-	12
Na	<10	-	<10
Ti	<20	-	<20
W	<20	<20	<20
U	< 0.3	-	< 0.3
V	<10	-	<10

MICROCONSTITUENT SURVEY

Samples from as-received Zr-2.5 wt% Nb pressure tubing were heat treated according to the schedule shown in Table II. Before heat treatments the samples were encapsulated in quartz tubing which was evacuated to 1×10^{-4} mm Hg and backfilled with helium. Photomicrographs were taken at various magnifications to exhibit the different microconstituents obtainable by different heat treatments. Knoop hardness measurements were made to determine the relative hardness of each microstructure studied.

Samples solution-heat-treated in the β or $\alpha + \beta$ field followed by a water quench undergo a martensitic transformation as β transforms to α' by a shear type reaction. Table III summarizes the Knoop hardness measurements taken on specimens with this type of heat treatment. The hardness of the alloy decreased from 240 to 200

as the solution heat treatment temperature was lowered from 850 °C to 675 °C. A plot of the Knoop hardness versus solution heat treatment temperature is shown in Figure 2.

Quenching from the β field produces microconstituents α' and retained β . Figure 3 is a photomicrograph at 500X from a specimen that was solution heat treated at 1000 °C in the β field for 6 hr and then water quenched. Quenching from the $\alpha + \beta$ field produces microconstituents α , α' , and retained β . The microstructure of a specimen solution heat treated for 6 hr at 750 °C in the $\alpha + \beta$ field is shown in Figure 4.

The transformation of β to α during a slow furnace cool involves the formation of a Widmannstätten α precipitate. The microstructure of a specimen cooled from 1000 °C to 600 °C in approximately 30 min was composed almost entirely of Widmannstätten α , Figure 5.

TABLE II. Heat Treatments Performed for Microconstituent Survey on Zr-2.5 wt% Nb Alloy

Temperature, °C	Time, hr	Quench
1000	8	WQ ^(a)
850	8	WQ
1000	8	FC ^(b)
1000	16	FC ^(c)
1000	16	FC ^(d)

(a) WQ - water quenched

(b) FC - furnace cooled

(c) Furnace cooled to 800 °C then water quenched

(d) Furnace cooled to 750 °C then water quenched

TABLE III. Knoop Hardness of Zr-2.5 wt% Nb for Different Heat Treatments

Temperature, ^(a) °C	Knoop Hardness ^(b)
850	242
825	226
800	221
775	226
750	206
725	209
700	210
675	201

(a) Solution heat treated 6 hr followed by a water quench

(b) Average of 30 indents; 10 on each of the three principle surfaces, longitudinal, transverse, and tangential

FIGURE 2. Knoop Hardness of Zr-2.5 wt% Nb as a Function of Solution Heat Treatment Temperature

465-7016

400X

FIGURE 3. Zr-2.5 wt% Nb Heat Treated 1000 °C for 6 hr, Water Quenched. 400X Polarized Light

465-6522B

1200X

FIGURE 4. Zr-2.5 wt% Nb Heat Treated 750 °C for 6 hr, Water Quenched. 1200X

465-6828B

200X

FIGURE 5. Zr-2.5 wt% Nb Furnace Cooled from 1000 °C to 600 °C, Water Quenched. 200X Polarized Light.

465-6827E

200X

FIGURE 6. Zr-2.5 wt% Nb Furnace Cooled from 1000 °C to 800 °C, Water Quenched. 200X Polarized Light.

If a specimen undergoing a slow furnace cool is interrupted within the $\alpha + \beta$ field by a water quench, the remaining untransformed β will transform to α' upon quenching. The resultant microstructure will contain microconstituents α' , Widmannstätten α , and retained β . Such a microstructure, of a specimen that was cooled from 1000 °C to 800 °C over a time interval of 14 min and then water quenched, is shown in Figure 6.

A specimen solution heat treated at 850 °C for 24 hr was composed of approximately 60% retained β and α' and 40% α , Figure 7. Applying the lever rule, this depicts the $\beta/\alpha + \beta$ phase boundary to be at 4.0 wt% niobium at a temperature of 850 °C.

The $\alpha + \beta/\beta$ phase boundary for this alloy occurs at 850 °C based on the phase diagram of Lundin and Cox. The high interstitial content apparently shifted this phase boundary to 870 °C. This is based on the results of applying the lever rule to 850 °C heat treatment, for 24 hr. This is in general agreement with the work of Williams and Gilbert who reported a phase boundary at 920 °C for 1100 ppm oxygen. Figure 8 is an approximate partial equilibrium phase diagram for the Zr-Nb alloy system. Over a temperature range of 850 °C to 650 °C the equilibrium concentration of niobium in α varies from less than 0.1 wt% to 0.6 wt%. Over the same temperature range the equilibrium niobium concentration in β varies from

465-6518B

1200X

465-6518A
200X with Polarized Light

FIGURE 7. Zr-2.5 wt% Nb Solution Heat
Treated at 850 °C for 6 hr, Water
Quenched 1200X

FIGURE 8. Partial Equilibrium Phase
Diagram for Zr-Nb Alloy System

approximately 4 to 20.5 wt%, and the relative amount of β varies from 60% to 10%. The strength of specimens solution-heat-treated in the $\alpha + \beta$ field is directly affected by the amount of niobium in β and the relative amount of β . The decrease in the relative amount of β dominates to the extent that the hardness decreases as the solution heat treatment temperature is lowered. Thus the 850 °C heat treatment has the greatest hardness for specimens quenched from the $\alpha + \beta$ field, Figure 2.

Widmannstätten α and α' are microconstituents often confused in microconstituent surveys. It is instructive that both of these microconstituents occur in the same microstructure, Figure 6. The Widmannstätten α nucleates primarily at the grain boundaries and impurity sites throughout the grains; it grows on habit planes to a "leafy" type appearance under slow cooling conditions. Upon water quenching, the remaining β transforms to a much finer structure of α' and retained β , which constitutes the continuous phase.

RECRYSTALLIZATION EFFECTS

Specimens were heat-treated according to the schedule in Table IV for a study of the recrystallization characteristics of the as-received 60% cold worked, Zr-2.5 wt% Nb pressure tubing. These heat treatments were performed in a vacuum chamber that was evacuated to 1×10^{-4} mm Hg and backfilled to atmospheric pressure with helium. Figure 9 is a schematic showing the specimen size and orientation nomenclature with respect to the Zr-2.5 wt% Nb pressure tubing. Longitudinal sections of the

TABLE IV. Heat Treatments Performed for Recrystallization Study of 60% Cold Worked Zr-2.5 wt% Nb

Temperature, (a) °C	Time, hr
850	3
850	6
850	12
750	3
750	6
750	12
650	3
650	6
650	12

(a) *Followed by a water quench*

samples were studied at 500X to show the primary recrystallization effects. Transverse sample sections were mounted for microscopic examination at 500X to verify the measurements made on the longitudinal sections and to study the grain growth results.

The results of the recrystallization study are summarized in Table V and Figure 10 for 60% cold worked samples. The Hern's intercept method was used to obtain the approximate grain size from photographs of longitudinal sections. Analysis of the grain size was attempted from photomicrographs of the transverse sections. However, a satisfactory etch could not be obtained on all the samples because of the very fine grain size and high degree of cold work. The sequence of photomicrographs for the 850 °C heat treatments is shown in Figure 11.

The activation energy is given by an Arrhenius type equation:

$$\frac{1}{\tau} = AL^{-Q/RT} \quad (1)$$

FIGURE 9. Specimen Orientation
Nomenclature

TABLE V. Results of Recrystallization Study Performed on
Zr-2.5 wt% Nb, 60% Cold Worked

Temperature, °C	Time, hr	Grain per inch at 500X,		Avg Grain Diam, μ
		Transverse	Longitudinal	
650	3	60	18	1.55
650	6	56	20	1.53
650	12	52	17	1.69
750	3	48	16	1.83
750	6	40	16	2.00
750	12	34	15	2.33
850	3	23	15	2.33
850	6	19	13	3.26
850	12	17	12	3.56

where

τ = half time for recrystallization

A = constant

Q = activation energy

R = ideal gas constant = 1.987 cal
per °k mole

T = absolute temperature in °k

By taking the logarithm of Equation
1, we obtain the following:

$$\ln \left(\frac{1}{\tau} \right) = \ln A - Q/RT \quad (2)$$

$$\ln (\tau) = \ln A + Q/RT \quad (3)$$

FIGURE 10. Recrystallization Results Showing Grain Size Versus Solution Heat Treatment Temperature for 60% Cold Worked Zr-2.5 wt% Nb

Then, Equation 4 and 5 are obtained by differentiating with respect to $1/T$.

$$\frac{d \ln(\tau)}{d\left(\frac{1}{T}\right)} = \frac{Q}{R} \quad (4)$$

$$Q = R \frac{d \ln(\tau)}{d\left(\frac{1}{T}\right)} \quad (5)$$

Thus, from a plot of $\ln(\tau)$ versus $\frac{1}{T}$, the activation energy can be obtained by:

$$Q = R (\text{Slope}) \quad (6)$$

From Figure 10 the half time for recrystallization for the 750 °C heat treatment temperature is 13 ± 2 hr. The half time for recrystallization for the 850 °C heat treatment was 2.1 ± 0.25 hr (Figure 12). The corresponding

activation energy was calculated to be 37 ± 8 kcal per mole. The recrystallized grain size is estimated as 3.1μ .

The tendency is for increased cold work to lower the activation energy for recrystallization. It is therefore expected that the 30% cold worked Zr-2.5 wt% Nb pressure tubing will have a higher activation energy for recrystallization than the 60% cold worked material.

ISOTHERMAL TRANSFORMATION CHARACTERISTICS

The isothermal transformation characteristics were studied to establish

466-5783

400X

3 hr, 400X Polarized Light

466-5789

400X

6 hr, 400X Polarized Light

466-5814

400X

12 hr, 400X Polarized Light

FIGURE 11. 850 °C Heat Treatments on
60% Cold Worked Zr-Nb Pressure Tubing
to Study the Recrystallization
Characteristics

FIGURE 12. Plot for the Determination of the Activation Energy for Secondary Recrystallization

an isothermal transformation diagram. The isothermal heat treatments were performed using a salt bath and are summarized in Table VI. The samples were encapsulated in quartz tubing which was evacuated to 1×10^{-4} mm Hg and backfilled with helium. All the samples were held at 1000 °C for 24 hr before entering the salt bath. They were submerged in the salt bath for the specific time and temperature, then immediately water quenched. The time lapse from the 1000 °C furnace to the salt bath was less than 10 sec. The time in transferring the samples from

the salt bath to the water quench was less than 5 sec for all the samples.

TABLE VI. Heat Treatments Performed on Zr-2.5 wt% Nb to Study Isothermal Transformation Characteristics

Salt Temperature ^(a)	Times, min
850	0.5, 1, 2, 4
800	0.25, 0.5, 0.75, 2
750	0.5, 1, 2, 4, 8
700	4, 8, 16

(a) All samples were held at 1000 °C for 24 hr before entrance into the salt bath.

The specimens were studied for microstructural effects, and Knoop hardness indents were taken using a 1 kg load.

The Knoop hardness values are summarized in Table VII for the isothermal heat treatments. A graph of hardness versus time for each temperature was constructed (Figure 13). The 50% transformation point was taken as the inflexion point of each curve. The 100% transformation point was taken as the minimum hardness condition and the 0% transformation point as the maximum hardness condition. An isothermal transformation diagram was constructed from these data. This diagram is shown in Figure 14.

TABLE VII. Knoop Hardness of Zr-2.5 wt% Nb as a Function of Isothermal Heat Treatment Temperature and Time

Temperature, °C	Time, min	Knoop Hardness	
		1 kg	2 kg
700	1	229	227
	4	207	206
	8	199	204
	16	203	201
750	9.5	243	237
	1	249	242
	2	236	233
	4	218	213
800	0.25	238	232
	0.5	241	234
	0.75	236	231
	1.0	236	232
	2	234	232
850	0.5	239	236
	1	250	248
	2	253	243
	4	235	231

In the graph of hardness versus time for the different isothermal heat treatments, there was an initial increase in hardness observed in the 800 °C and 750 °C curves, followed by a reduction in hardness for longer times at each temperature. This initial hardness increase is possibly due to the formation of the coherent ω precipitate as β transforms to α . For longer transformation times ω loses its coherency and the hardness decreases.

Photomicrographs were not useful for the determination of the isothermal transformation diagram because of the difficulty in distinguishing between the Widmannstätten α and the martensitic α' microconstituents. Figure 15 shows the sequence of photomicrographs taken for the 750 °C isothermal heat treatments.

The isothermal transformation diagram for Zr-2.5 wt% Nb is presented in Figure 14. The speed of the formation of Widmannstätten α from β suggests that rapid quenching must be performed to produce α' from β .

AGING CHARACTERISTICS

Specimens were heat-treated and aging treatments were performed at 500 °C according to the schedule of Table VIII. Transverse sample sections were then mounted, and 10 Knoop hardness indents were taken across each sample to show the effect of the aging heat treatments. These hardness data are summarized in Table IX.

A decrease in hardness with aging time was observed for all three of the solution heat treatments studied

FIGURE 13. Hardness Data for Isothermal Transformation Heat Treatments

FIGURE 14. Isothermal Transformation Diagram for Zr-2.5 wt% Nb

465-7006

200X

30 sec at 750 °C

465-7007

200X

1 min at 750 °C

465-7008

200X

2 min at 750 °C

465-7010

200X

8 min at 750 °C

FIGURE 15. Isothermal Heat Treatments
Performed at 750 °C. 200X Polarized
Light

TABLE VIII. Aging Treatments Performed on Heat Treated^(a) Zr-2.5 wt% Nb Specimens

<u>Specimen</u>	<u>Age Time</u>	<u>Age Temperature, °C</u>
1	10 min	500
2	30 min	500
3	1 hr	500
4	5 hr	500
5	24 hr	500

(a) Heat treated at 850 °C for 24 hr followed by a water quench

TABLE IX. Results of Aging Study

<u>Solution Heat Treatment^(a) Temperature, °C</u>	<u>Aging Time at 500 °C in hr</u>	<u>Knoop^(b) Hardness Number</u>
850	0.25	228
850	0.5	226
850	1.0	220
850	5.0	216
850	24.0	212
750	0.25	220
750	1.0	212
750	5.0	179
750	24	176
750	48	179
750	100	177
700	0.25	205
700	1	196
700	5.0	177
700	24	177
700	48	177
700	100	187

(a) Followed by water quench

(b) Average of 10 indents across a transverse surface with respect to orientation nomenclature.

(Figure 16). This decrease is explained as solute depletion in α' . There was a slight secondary hardening effect observed upon aging the 700 °C solution heat treatment. This was noticed after 100 hr of aging at 500 °C and resulted in a Knoop hardness change from 177 to 187. The driving force for the formation of a precipitate is the high niobium concentration in α' as compared to equilibrium α . The higher the niobium concentration in α' the sooner the coherent precipitate ω will form. This is one possible explanation for the secondary hardening effect observed.

TENSILE PROPERTIES

Tensile properties of this alloy were determined; see Table X for a summary of the tests conducted. These specimens were machined from the as-received pressure tubing so that the longitudinal axis of the specimen was aligned with the axial direction of the tube. The tests were performed at room temperature using a strain rate of 0.0002 in./min. Figure 17 is a plot of the tensile properties as a function of solution heat treatment temperature for a 6 hr heat treatment followed by a water quench.

The 850 °C heat treatment for 24 hr followed by a water quench showed the optimum tensile properties for the heat treatments studied. The 1000 °C for 6 hr heat treatment showed brittle properties in the water quenched condition and very ductile properties in the furnace cooled condition.

FIGURE 16. Aging Characteristics of Zr-2.5 wt% Nb

FIGURE 17. Tensile Properties of Zr-2.5 wt% Nb as a Function of Solution Heat Treatment Temperature

TABLE X. Heat Treatments Performed on Tensile Samples of Zr-2.5 wt% Nb (a)

Temperature, °C	Time, hr	Quench	Sample Number
1000	6	FC ^(b)	1
1000	6	WQ ^(c)	2
850	6	WQ	3
800	6	WQ	4

(a) Room temperature, 0.002 in./min

(b) FC - furnace cooled

(c) WQ - water quenched

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BIBLIOGRAPHY

A. E. Austin and J. R. Doig. "Structure of the Transition Phase Omega In Ti-Cr Alloys," *Journal of Metals*, January 1957.

W. F. Bourgeois and J. N. Chirigos. *Development of Zirconium-Base Alloys* WAPD-TM-546, Westinghouse Atomic Power Development, Pittsburgh, Pennsylvania. November 1965.

C. E. Ells, S. B. Dalgaard, W. Evans and W. R. Thomas. *Development of Zirconium-Niobium Alloys*, A/Conf. 28, p. 22, Canada, May 1964. Third United Nations International Conference on the Peaceful Uses of Atomic Energy.

V. S. Emelyanov, Y. G. Godin and A. I. Evstyukhin. *Mechanical Properties of Zirconium Binary and Ternary Alloys with Tantalum and Niobium at Room and Elevated Temperatures*, p. 2055, USSR.

P. D. Frost, W. M. Parris, L. L. Hirsch, J. R. Doig and C. M. Schwartz. *Isothermal Transformation of Titanium-Manganese Alloys*, presented before the Thirty-fifth Annual Convention of the Society held in Cleveland, Oct. 17 to 23, 1953.

P. D. Frost, W. M. Parris, L. L. Hirsch, J. R. Doig and C. M. Schwartz. *Isothermal Transformation of Titanium-Chromium Alloys*, paper presented before the Eighth Western Metal Congress of the Society, held in Los Angeles, March 23 to 27, 1953.

G. R. Love and M. L. Picklesimer. "The Kinetics of Beta-Phase Decomposition in Niobium (Columbium)-Zirconium," *Transaction of the Metallurgical Society of AIME* 430, vol. 236, April 1966.

C. E. Lundin, et al. *The Determination of the Equilibrium Phase Diagram, Zirconium Niobium*, AEC Technical Report TID 11919, Denver Research Institute. 1964.

R. J. McClintick, G. William Bauer, Lee S. Busch. *Physical Metallurgy and Heat Treatment of Titanium Alloys*, Mallory-Sharon Titanium Corp., Niles, Ohio.

The Metallurgy of Zirconium, Edited by Benjamin Lustman, Westinghouse Electric Corp, Atomic Power Division, Pittsburgh, Pennsylvania and Frank Kerze, Jr., Reactor Development Division, Atomic Energy Commission, Washington, D.C. McGraw-Hill Book Company, Inc.

Robert E. Reed-Hill. *Physical Metallurgy Principles*, Van Nostrand Company, Inc., Princeton, New Jersey. University Series in Basic Engineering.

C. D. Williams and R. W. Gilbert. *Tempered Structures of A Zr-2.5 Wt% Nb Alloy*, Chalk River Nuclear Laboratories, Atomic Energy of Canada Limited, Chalk River, Ontario, Canada.

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