MASTER

LOAD RELAXATION STUDIES OF ZIRCALOY-4

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1. INTRODUCTION

This paper reports load relaxation studies of Zircaloy-4 as a function of temperature. The experimental data are analyzed by using the approach of a plastic equation of state as proposed by Hart.¹ The effects of strain aging and deformation twinning are discussed based on the experimental results.

The mechanical properties of zirconium and the Zircaloys have been extensively reported in the literature.²⁻⁹ The effects of strain aging, deformation twinning and anisotropy were shown to play an important role in governing their mechanical properties. In the previous investigations, the test methods included tensile, creep, temperature change, and strain rate change tests. Load relaxation tests on zirconium were reported by Lee and Hart⁷ but such tests have not been used for the investigation of the Zircaloys from the viewpoint of a plastic equation of state.

Recently a phenomenological model for non-elastic deformation in a crystalline solid has been proposed by Hart and tested experimentally by using the load relaxation test as a major experimental tool.¹⁰⁻¹² This model has been found to describe the deformation properties of a variety of metals. In fact in the absence of strain aging and deformation twinning all the materials tested showed very similar behaviors from the viewpoint of Hart's plastic equation of state. A purpose of the present work is to test the applicability of the same phenomenological model for the description of the deformation properties of Zircaloy-4.

The effects of strain aging in the Zircaloys have been identified from tensile and creep data.^{3,9} The present knowledge of this phenomenon is not sufficient however for the prediction of its in-

fluence on the creep behavior. In the present work attempts were made to use the load relaxation test to obtain additional information on strain aging.

The approach of Hart's plastic equation of state was developed recently. In the next section a brief review of this approach will be given as a background for the convenience of the reader. It will be followed by a description and discussion of the experimental results. In the discussion the present experimental results will be compared with existing mechanical property data reported for the Zircalloys.

2. PHENOMENOLOGICAL DEFORMATION FOR MODEL

The concept of a plastic equation of state adopted in the present work was first introduced by Hart¹ to describe deformation controlled by grain matrix processes. Recent experimental and theoretical progress on this subject has been summarized in reference 1. Hart suggests that a plastic equation of state can be formulated in terms of stress, plastic strain rate, temperature and a hardness parameter. The existence of this type of plastic equation of state can be tested by performing load relaxation experiments. At a given temperature the load relaxation experiments generate stress-plastic strain rate relationships at essentially constant hardness. It has been demonstrated in a variety of metals and commercial alloys that the stress-plastic strain rate relationships of different hardnesses obtained by deforming the material to various plastic strain levels prior to load relaxation, are related by scaling.¹ This type of behavior justifies the concept introduced by Hart and suggests that the constant hardness stress-plastic strain rate relationship uniquely specifies the hardness state of the material.

Recently the phenomenological theory of plastic equation of state has been extended by Hart.¹⁰ He has proposed a deformation model consisting of essentially two parallel branches. One of the branches, representing dislocation glide controlled processes, is important at lower homologous temperatures. The other branch contains no contribution from dislocation glide controlled processes and predominates at higher homologous temperatures. The stresses in these two branches are σ_f and σ_a respectively and their sum equals the applied stress σ .

We shall review this deformation model in some detail as it will be the basis for the data analysis and discussion.

It has been shown experimentally^{1,10} at high homologous temperatures that the constant hardness $\log_{10}\sigma - \log_{10}\dot{\epsilon}$ curves can be represented by a flow law,

$$\ln (\sigma^*/\sigma_a) = (\dot{\epsilon}^*/\dot{\alpha})^\lambda \quad (1)$$

where σ^* is a hardness parameter, λ is a temperature independent parameter which determines the shape of the $\log_{10}\sigma - \log_{10}\dot{\epsilon}$ curve and has been found to have a value close to 0.15 for several materials and $\dot{\epsilon}^*$ is a parameter which depends on temperature, heat treatment and deformation.

In the region of stress and temperature where Equation (1) is applicable, σ_a approaches σ , $\dot{\alpha}$ approaches $\dot{\epsilon}$, and the measured $\log_{10}\sigma - \log_{10}\dot{\epsilon}$ curves are concave downward. The curves for different hardnesses at a given temperature in the same region, are related through translation along a straight line in $\log_{10}\sigma - \log_{10}\dot{\epsilon}$ space. The curves for different temperatures at a given hardness are related through translation along the $\log_{10}\dot{\epsilon}$ axis such that $d \ln \dot{\epsilon} = d(Q \cdot 1/RT)$.

Analytically these dependences can be represented as

$$\dot{\epsilon}^* = (\sigma^*/G)^m f \exp \{-Q/RT\} \quad (2)$$

where G is shear modulus, m is the inverse of the slope of the translation path and is temperature independent, f is a frequency parameter, Q is the activation energy for deformation and RT has the usual meaning.

The rate of deformation in the region where Equation (1) applies is assumed to be controlled by diffusive processes. In the

present work the activation energy Q will be taken to be that for diffusion.

At lower homologous temperatures, the constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curves have been shown^{1,10} to be represented by

$$\dot{\epsilon} = \dot{a}^* (\sigma_f / M)^M \quad (3)$$

where $\dot{a}^*$ is a rate parameter, σ_f is an effective stress and becomes $(\sigma - \sigma^*)$ in the region where deformation processes represented by Equation (1) are not important, M is a constant and is found to equal the inverse of the slope at the translation path in this region, and M is anelastic modulus.

Equation (3) is assumed to represent dislocation glide controlled processes. The rate parameter $\dot{a}^*$ reflects the effects of lattice friction, impurity drag, etc., and can depend on temperature in a complex way. It is less dependent on temperature than the parameter $\dot{\epsilon}^*$.

In the region where Equation (3) is applicable, the measured $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curves are concave upward. At a given temperature, the curves for different hardness are related through a translation path with a slope $1/M$. At a given hardness, the position of the curves depends on the value of the parameter $\dot{a}^*$. As the applied stress approaches the value of σ^* , the plastic strain rate according to Equation (3) will approach zero.

In the intermediate temperature range (between the two regions described previously), deformation processes represented by both Equations (1) and (3) will be important such that

$$\sigma = \sigma^* \exp[-(\dot{\epsilon}^* / \dot{\epsilon})^\lambda] + M(\dot{\epsilon} / \dot{a}^*)^{1/M} \quad (4)$$

where the two terms on the righthand side correspond to σ_a and σ_f

respectively.

In this temperature range the apparent activation energy for plastic deformation will reflect the temperature dependence of both $\dot{\epsilon}^*$ and $\dot{a}^*$. Consequently the measured apparent activation energy usually increases with increasing temperature.

The scaling relations discussed previously will no longer be applicable in the transition region between those represented by Equation (1) and (3). The shape of the curves at different hardness can be described by Equation (4).

In the reported work the load relaxation data of type 316 stainless steel exhibited the entire range of behavior represented by the deformation model.¹¹ It should be noted that at elevated temperatures the effects of strain aging and thermally induced microstructural changes will cause some of the parameters in Equation (4) to change with time. Under these circumstances, the measured load relaxation data will no longer be represented by the deformation model.

3. EXPERIMENT

The test specimen is schematically shown in Figure 1. It was machined from Zircaloy-4 tubing with nominal dimensions 0.422" (1.07 cm) O.D. x 0.372" (0.95 cm) I.D. x 6" (15.24 cm) length. The tubing was produced from Wah Chang Heat Number 3880310 by cold pilgering in four passes after hot extrusion. Ingot chemistry is given in Table 1. Initial cleaning of the tubing was accomplished by degreasing in an ultrasonic acetone bath. The sections of tubing were subsequently scrubbed with Alconox laboratory detergent (both I.D. and O.D.), and ultrasonically rinsed in tap water, distilled water and ethanol, in that order. The as-pilgered tubing was then annealed for three hours at 700°C (973°K) to an average grain size of 8 μ m.

The principle and detailed experimental procedures of the load relaxation experiment are given in references 1 and 6. During the load relaxation experiment the specimen was first deformed to the desired plastic strain level. The crosshead was then fixed. The load relaxation resulted from converting the elastic strain of the test system to the plastic strain of the specimen. The rate of load relaxation is related to the plastic strain rate of the specimen through the machine constant of the test system. The load vs. time data yield $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curves at essentially constant hardness. The overall accuracy of the data was estimated to be 5%. For initial loading the crosshead speed was 0.02 in/min.

It should be noted that the load relaxation and tensile data obtained in this work were for uniaxial test conditions in the direction parallel to the tube axis only. Zircalloys are noted for their anisotropic properties, however, this variable was not investigated in the present work.

TABLE 1
INGOT ANALYSIS OF THE ZIRCALOY-4

Element	Composition in Percent		
	Top	Middle	Bottom
Sn	1.38	1.42	1.38
Fe	0.21	0.21	0.21
Cr	0.11	0.11	0.10

Element	Composition in ppm		
	Top	Middle	Bottom
Al	49	48	49
B	0.2	< 0.2	0.2
C	160	160	170
Cd	< 0.2	< 0.2	< 0.2
Cl	< 5	< 5	< 5
Co	< 10	< 10	< 10
Cu	20	20	18
H	< 25	< 25	< 25
Hf	47	50	61
Mg	< 10	< 10	< 10
Mn	< 25	< 25	< 25
Mo	< 25	< 25	< 25
Ni	< 35	< 35	< 35
Pb	< 50	< 50	< 50
Si	65	71	71
Ta	< 200	< 200	< 200
Ti	< 20	< 20	< 20
U	< 0.5	< 0.5	< 0.6
V	< 25	< 25	< 25
W	< 5	< 5	9
N	24	24	29
O	1000	1030	1180

4. RESULTS AND DISCUSSION

In this section tensile and load relaxation data as a function of temperature will be presented first with a brief discussion of the various deformation mechanisms involved. They will be followed by a detailed analysis of the load relaxation data at each temperature to support the mechanisms suggested. The discussion will also include the following phenomena: strain aging, strain rate sensitivity and grain boundary sliding.

Tensile data at different temperatures, in a plot of log stress vs. non-elastic strain, are shown in Figure 2. The non-elastic strain was measured by using the method given in reference 8. These data were generated in the load relaxation experiment during loading prior to the load relaxation run. At each temperature several load relaxation runs were made. The specimen was reloaded to consecutively higher strains (equivalently higher hardnesses) as shown in Figure 2.

The effects of strain aging as indicated by the yield drop in the tensile data, are more pronounced at 300°C (Fig. 2). The concept of strain aging adopted in the present work is that of long range diffusion causing a dragging force due to the formation of an atmosphere of solute atoms behind moving dislocations.¹³ The yield drop was less pronounced at high strains. This result can be rationalized from the increase in the flow stress due to work hardening such that the yield drop will decrease when the flow stress approaches the level of the drag force mentioned above. This decrease in the yield drop at higher strains will influence the load relaxation data and will be discussed in a latter part of the paper.

Typical load relaxation data at various temperatures are shown in Figure 3. Before discussing the data at each temperature in

detail we shall first discuss qualitatively the various mechanisms involved as a background for the data analysis.

It is suggested that 200°C, 300°C, 350°C and 385°C data in Figure 3 exhibit the deformation behavior represented by the phenomenological model described previously. Qualitatively, the 200°C data reflect the behavior at low homologous temperatures that the constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curve is concave upward. As the temperature increases, the branch which predominates at high homologous temperatures, becomes important. The 350°C and 385°C data show, therefore, an inflection point and become concave downward at low strain rates. The same type of behavior was observed in type 316 stainless steel.¹¹ The solid line in Figure 3 were calculated based on the phenomenological model (see Appendix) and will be discussed further. At 385°C the data in Figure 3 deviate at low strain rates from the solid line which represents deformation controlled by grain matrix processes. This deviation suggests the introduction of the contribution of grain boundary sliding. The 450°C and 500°C data in Figure 3 are also expected to contain the contribution of grain boundary sliding and cannot be represented by the deformation model.

The room temperature data do not belong to the family of curves representable by the phenomenological model either. If it were in the same family, based on the model and the results on type 316 stainless steel, it would be a curve which is concave upward and with a slope higher than that of the 200°C curve.

In the following sections we shall discuss the data at each temperature separately to support the suggestions made above. We shall start with the data in the temperature range where the phen-

phenomenological model for deformation is applicable.

4.1.1. 200°C Load Relaxation Data

As pointed out previously the constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data at 200°C exhibit the typical behavior expected at low homologous temperatures. The data shown in Figure 4a and 4b can be represented essentially by Equation (3). The solid curves in the figure were calculated based on Equation (4) and the procedure for the numerical analysis is given in the Appendix. The applicability of Hart's plastic equation of state can be demonstrated also by the scaling behavior in that the curves of different hardness can be translated along the straight line shown in Figure 4a to overlap each other. The composite curve shown in Figure 4b is the result of the translation. The vertical scale of Figure 4b is expanded to show more clearly the scaling behavior. The slope of the translation path is found to be the inverse of the value of the exponent M in Equation (4) which is obtained in the numerical analysis to have a value of 8. This agreement can be used to support the phenomenological model used.

4.1.2. 300°C Load Relaxation Data

The 300°C constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data at high hardnesses (strains) can also be represented by the phenomenological model. The solid curves in Figure 5a are calculated based on Equation (4) (see Appendix). At this temperature, the high temperature branch (Equation 1) begins to contribute. The top two curves (high hardnesses) show good agreement between the calculated curve and the measured data. These curves can be translated along the translation path with a slope of $1/8$ to overlap each other. The composite curve (the lowest curve) in Figure 5b is the combination of the top two curves in Figure 5a by scaling.

The data of lower hardnesses in Figure 5a cannot be represented by the calculated solid curves nor do they exhibit the scaling behavior. Figure 5b shows that only the portion of the data at high strain rates which is represented in the calculated curves can be translated to overlap each other. Some remarks can be made as to the deviation of the experimental data for the calculated curves in Figure 5a: 1) the deviation is in the direction which produces lower strain rates as compared to strain rates calculated by use of the phenomenological model; and 2) the deviation decreases and eventually disappears at higher hardnesses. This latter result is consistent with the tensile data that at higher hardnesses the yield drop decreases due to the increase in flow stress.

If we assume that the phenomenological model can be used to represent the behavior of Zircalloys in the absence of strain aging, the observation made in the previous paragraph suggests that the deviation from the calculated curves shown in Figure 5a, reflects the effects of strain aging. This possibility should be explored in future research as it may provide a quantitative measure of the effects of strain aging in addition to that obtained based on tensile and creep data.

4.1.3. 350°C Load Relaxation Data

The 350°C constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data in Figure 6 shows an inflection point. According to the phenomenological model they exhibit the behavior of the intermediate homologous temperature range where both branches of the model are important. In this temperature range the scaling behavior, as pointed out previously, will no longer exist.

In the data analysis we assume that the higher two curves in

Figure 6 (higher hardnesses) do not contain either the effects of strain aging or those of grain boundary sliding. Thus, based on these data and Equation (4) we can determine the required parameters, especially those characterizing the branch which predominates at higher homologous temperatures (Equations 1 and 2). The lowest solid curve in Figure 6 is calculated by using the parameters determined above and a hardness value which fits the higher strain rate portion of the measured data. It is seen that the measured data deviate slightly from the calculated curve in the direction showing the effects of strain aging.

4.1.4. 385°C Load Relaxation Data

At this temperature the tensile data suggest that the effects of strain aging are not important. The 385°C data in Figure 7 is analyzed with the values of the parameter $\dot{\epsilon}^*$ calculated by use of Equation (2) and the values of $\dot{\epsilon}^*$ determined from the 350°C data. In this calculation we have used an activation energy of 58.5 kcal/mole.² The parameter $\dot{\epsilon}^*$ determines the position of the constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curve at high homologous temperatures (Equation 1) along the strain rate axis at a given hardness. The phenomenological model^{10,11} requires that at a particular hardness the values of $\dot{\epsilon}^*$ of two different temperatures be related through Equation (2) with the value of Q being that for self diffusion. This requirement allows us to detect the contribution of grain boundary sliding because it will predetermine the position of the calculated curve representing grain matrix deformation in Figure 7.

The contribution of grain boundary sliding is usually in the direction that increases the strain rate from that resulting from grain matrix deformation alone.^{14,15} The solid curves in Figure 7

are calculated by use of Equation (4) with the parameter $\dot{\epsilon}^*$ determined as described above and the value of hardness σ^* based on the high strain rate portion of the measured data. It is seen that at lower strain rates the measure data deviate from the calculated curves. We suggest that the observed deviation indicates the contribution of grain boundary sliding based upon the following two results: 1) the observed deviation is in the direction which increases the strain rate at a given stress; and 2) the observed deviation increases with the increasing hardness of the specimen. Both results are consistent with existing concepts of grain boundary sliding^{14,15} and will be discussed further.

4.1.5. 450°C and 500°C Load Relaxation Data

At these temperatures based on the tensile data the effects of strain aging are absent. However, the effects of grain boundary sliding are expected to continue to be significant.^{14,15} At these temperatures there is a finite probability that the microstructure of the specimen may change with time during the load relaxation run and, therefore, influence the shape of the observed $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curves.¹¹ The effects of microstructural change can be determined by a reloading experiment¹ which was not performed in the present work. With the uncertainty on the effects of microstructural instability and the lack of an adequate grain boundary sliding model^{1,15} we are not in the position to analyze the data in Figures 8 and 9 quantitatively. It should be noted that the 500°C data shows a higher slope as compared to that of the 450°C data. This relative change in the slope of the $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data is consistent with the current concept of grain boundary sliding and with the phenomenological model.^{10,14,15}

4.1.6. Room Temperature Load Relaxation Data

The room temperature $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data are shown in Figure 10. As discussed previously these data do not belong to the family of curves which can be represented by the phenomenological model.

The mechanism of deformation twinning is known to be operative in Zircalloys at room temperature.⁵ We suspect that the data in Figure 10 reflects the microstructure produced by deformation twinning during preloading before the load relaxation run and/or that deformation twinning is also involved in the deformation processes during the load relaxation run. Further work is required to support this suggestion.

Thermal-mechanical histories at higher temperatures were found not to influence the shape of $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ data in Figure 10. Between the data represented by the two upper hardnesses and those of the three lower hardnesses several load relaxation runs were made at 200°C (200°C data not shown). Other than the difference in the hardness level, these two sets of data show essentially the same qualitative behavior and thus demonstrate the absence of the effect of exposure at higher temperatures.

With the results of the data analysis as a background we shall discuss further the following three topics: 1) Strain-aging; 2) strain rate sensitivity; and 3) grain boundary sliding.

4.2 Strain Aging and Dislocation Mobility

The effects of strain aging can influence strongly the creep properties of a material. For example Fidleris et al reported a creep rate minimum at 300°C for Zircaloy-2.³ They associated the observed minimum with the effects of strain aging.

We have pointed out that the observed deviation from the de-

formation behavior represented by the phenomenological model (Figures 5a and 6) in the load relaxation data is consistent with the tensile data such that the effects of strain aging decreases as the hardness of the specimen increases. We shall show that this observed deviation is consistent also with the results of creep tests.

In Figure 11 the solid curves are calculated curves by using the parameters in Table 2 and Equation (4). These curves are of the same hardness at different temperatures. According to these curves the strain rate at a given applied stress increases with increasing temperature, as is expected in the absence of the effects of strain aging. The measured data in Figure 11 are the same as those of the lowest hardnesses in Figure 5a and show the deviation from the calculated curve for 300°C. If one extrapolates the 300°C data by following their trend, it is reasonable to expect that the extrapolated data will cross the calculated curve for 200°C. In the region after crossing, the strain rate at 200°C will be higher than that at 300°C for the same stress as observed in creep tests by Fidleris et al.³

The effects of strain aging of interest to the present work is believed to be associated with the drag force produced by the solute atmosphere around a moving dislocation.¹³ At the present the theory for these effects is not fully developed.¹³ The data such as those shown in Figures 5a and 6 should aid in the development of the required theory.

In the absence of the effects of strain aging the magnitude of dislocation mobility is measured by the parameter $\dot{\epsilon}^*$ in Equation (3). According to the phenomenological model the temperature dependence

Table 2

The Values of the Parameters in Equation (4)
Based on the Data in Figures 4a, 5a, 6 and 7

Temp. (°C)	Strain (%)	$\sigma^* \left(\frac{MN}{m^2} \right)^+$	$\dot{\epsilon}^* (\text{sec}^{-1})$	$M \dot{\sigma}^{*-1/M} \left(\frac{MN}{m^2} \right)$	λ	M
200	3.79	174.92	3.250×10^{-28}	274.70	0.15	8
200	5.63	201.11	6.529×10^{-28}	274.70	0.15	8
200	7.46	220.20	1.028×10^{-27}	274.70	0.15	8
300	6.32	218.64	2.157×10^{-18}	116.40	0.15	8
300	7.96	235.08	3.099×10^{-18}	116.40	0.15	8
300	11.53	257.27	4.864×10^{-18}	116.40	0.15	8
350	6.83	236.99	4.545×10^{-15}	64.25	0.15	8
350	8.66	254.76	2.277×10^{-14}	64.25	0.15	8
385	5.10	244.46	2.206×10^{-12}	16.11	0.15	8
385	6.98	264.92	4.108×10^{-12}	16.11	0.15	8

+ The values of σ^* at temperatures higher than 200°C are given in the stress scale of 200°C. To obtain the value in the stress scale of the temperature of interest, multiply the value given by the ratio of the shear modulus at the temperature of interest to that at 200°C.

of this parameter will not be exponential with an activation energy corresponding to that for self-diffusion. The calculated values of $M\dot{A}^*-1/M$ are plotted as a function of temperature in Figure 12. (The values plotted are those obtained in the numerical analysis, Table 2). The temperature dependence shown is not exponential thereby supporting the deformation model. A similar temperature dependence was observed for the parameter $\dot{A}^*$ for Type 316 stainless steel.¹¹

4.3 Strain Rate Sensitivity

The strain rate sensitivity of the flow stress $(\partial \ln \sigma / \partial \ln \dot{\epsilon})_{\sigma^*}$ of the Zircalloys has been reported in the literature by various investigators.⁴ It has been suggested that a reduced strain rate sensitivity is associated with the strain aging behavior. The possibility is consistent with the results of the present work in that the deviation from the calculated curves due to strain aging shown in Figures 5a and 6 is in the direction of reducing the strain rate sensitivity.

Lee has reported the measurement of the strain rate sensitivity of Zircaloy-2 as determined by the differential strain rate test.⁴ He reported his data as a function of strain rate and also as a function of temperature. The same type of information can be obtained from the load relaxation data. The slope of the constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curves gives the strain rate sensitivity. Figure 13 shows the calculated strain rate sensitivity at various temperatures and as a function of strain rate. The values shown are calculated from the data presented in Figures 4a, 5a, 6, 7, 8, and 9 with strains close to 5%. Figure 14 shows calculated strain rate sensitivity based on data in the same figures as a function of temperature for two different strain rates. The data of the present work

are not taken exactly at a strain of 3% and of 7% as indicated in Figure 14. The calculations for Figure 11 are based on estimated curves obtained by scaling for the strains of interest.

The results in Figures 13 and 14 show the same qualitative behavior as those reported by Lee.⁴ For example at 350°C both investigations show that strain rate sensitivity is high at high strain rates, decreases to a minimum with decreasing strain rate and increases again at low strain rates (Fig. 13). At a strain rate of 10^{-5} sec^{-1} both investigations also show a minimum in the strain rate sensitivity in the temperature range of 300°C to 400°C (Fig. 14). Based on the results of the present data analysis, the observed minimums in strain rate sensitivity simply reflect the deformation behavior represented by the phenomenological model, even though these minimums occur in the region (temperature, stress and strain rate range) where the effects of strain aging are important. The minimum in strain rate sensitivity corresponds to the inflection point observed in the load relaxation data (Fig. 6,7). According to the deformation model, an inflection point will exist in a constant hardness $\log_{10} \sigma - \log_{10} \dot{\epsilon}$ curve in the intermediate homologous temperature range and in the strain rate range of the present work where both branches of the model are important.

The effects of strain aging on the strain rate sensitivity can be seen however from the data in Figure 14. In this figure both high strain and low strain data are included. It has been shown previously that at low strains the effects of strain aging are more pronounced. The difference in the strain rate sensitivity indicated by the two sets of data in Figure 14 can be taken therefore as to reflect the effects of strain aging on the strain rate sensitivity.

4.4 Grain Boundary Sliding

Grain boundary sliding usually begins to become important in a f.c.c. metal near one half of its absolute melting temperature. The temperature range of 350°C - 385°C is significantly lower than one half of the absolute melting temperature of the Zircalloys which is of h.c.p. structure. One would be less inclined to suggest the occurrence of grain boundary sliding during deformation in this temperature range and in the strain rate range of the present work. Recently Sabol and McDonald have observed intergranular fracture at 350°C in Zircaloy-4 tubing at low strain rates.⁷ Intergranular fracture has been related to grain boundary sliding in Type 316 stainless steel tubing.¹⁶ The observation of Sabol and McDonald together with the results of the present work shown in Figure 7 suggest the possibility of grain boundary sliding in Zircalloys at these rather low temperatures (350°C - 385°C). The existence of grain boundary sliding at this temperature range is probably related to the temperature of $\alpha \rightarrow \beta$ transformation which occurs at 862°C. This possibility could have significant technological interest because the intergranular fracture found in the Zircaloy cladding of light water reactors has been usually associated with stress corrosion cracking.⁷ The discussion given above suggests an additional cause which is completely mechanical.

SUMMARY

The results of the present work have shown 1) the load relaxation data of Zircaloy-4 in the temperature range of 200°C - 385°C can be represented by the phenomenological model based on Hart's plastic equation of state; 2) the room temperature load relaxation data reflect the effects of deformation twinning; 3) at higher temperatures the load relaxation data suggest the contribution of grain boundary sliding; and 4) the effects of strain aging can be identified based on the load relaxation data.

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APPENDIX

In the following paragraphs the procedures for the numerical calculation for the solid curves in Figures 4a, 5a, 6, and 7 will be described. The task here is to determine the required parameters in Equations (2) and (4). There are in total six parameters involved σ^* , $\dot{\epsilon}^*$, λ , $M\dot{\sigma}^*^{-1/M}$, M , and m . Since at a given temperature it is possible for only one of two branches of the phenomenological model to predominate, we are not determining all of the six parameters at one time by data fitting.

The physical significance of the required parameters provides considerable insight into their determination and will therefore be described. The parameter σ^* determines the position of both the curves represented by Equations (1) and (3) along the stress axis. The parameters $\dot{\epsilon}^*$ and $\dot{\sigma}^*$ determine the position of the same two curves respectively along the strain rate axis. The parameter λ determines the shape of the curve of Equation (1). The slope of the translation path is given by $1/m$ and $1/M$ for the curves of Equations (1) and (3) respectively. The exponent M determines also the shape of the curve of Equation (3). Based on the experience of our past work^{1,10} reasonable estimates of the value of λ and m were taken to be 0.15 and 0.2 respectively. A good estimate of the value of M is 8 which is the inverse of the slope of the translation path in Figure 4a. With these estimates, the number of unknowns is reduced essentially to three or less which can be found by a nonlinear least square fit to the measured data at each hardness.

It should be noted that the estimated values of λ and M are close to those of the same parameters used in analyzing the load relaxation data of Type 316 stainless steel.

Before performing the numerical calculations the stress values of the data for 300°, 350°, and 385°C are first modulus corrected to that for 200°C^{1,10,17} such that the value of σ/G for these temperatures are the same as that for 200°C.

For the 200°C data we need to determine essentially only the value σ^* and $M\dot{a}^{*-1/M}$ because the high homologous temperature branch (Equation 1 or the first term of the righthand of Equation 4) is not important at this temperature.

The 350°C data at the two higher hardnesses will be analyzed first because they contain a significant contribution of the high homologous temperature branch and are assumed not to be influenced by the effects of strain aging and grain boundary sliding. These data are used to determine the values of σ^* , $\dot{\epsilon}^*$ and $M\dot{a}^{*-1/M}$. The curve for the lowest hardness at 350°C is calculated by using the same value of $M\dot{a}^{*-1/M}$, a value of σ^* that will fit as much of the measured data as possible at high strain rates, and a value of $\dot{\epsilon}^*$ given by the value of σ^* used and the translation path (Equation 2).

The 300°C curves are calculated by using the value of $\dot{\epsilon}^*$ which is related to the $\dot{\epsilon}^*$ determined from the 350°C data through Equation (2) and the value of σ^* and $M\dot{a}^{*-1/M}$ which will allow the calculated curves to overlap the measured data at high strain rates as much as possible. The 385°C curves are calculated by using the same procedure.

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FIGURE CAPTIONS

- Figure 1: Schematic Drawing of Tensile Specimen.
- Figure 2: Stress-strain curves for Zircaloy-4 at various temperatures.
- Figure 3: Constant hardness $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at several temperatures.
- Figure 4: a) $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy at 200°C and several plastic strain levels.
b) Master constant hardness curve for Zircaloy-4 from three 200°C curves.
- Figure 5: a) $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 300°C and several plastic strain levels. Stress values are modulus corrected to those at 200°C.
b) $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 300°C. All data are translated along the solid line. Stress values are modulus corrected to those at 200°C.
- Figure 6: $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 350°C and several plastic strain levels. Stress values are modulus corrected to those at 200°C.
- Figure 7: $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 385°C and several plastic strain levels. Stress values are modulus corrected to those at 200°C.
- Figure 8: $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 450°C and several plastic strain levels.
- Figure 9: $\log_{10}\sigma$ vs. $\log_{10}\dot{\epsilon}$ data for Zircaloy-4 at 500°C and several plastic strain levels.

Figure 10: $\log_{10} \sigma$ vs. $\log_{10} \dot{\epsilon}$ data for Zircaloy-4 at room temperature and several strain levels. Several load relaxation tests were made at 200°C between the tests of two upper hardnesses and those of three lower hardnesses.

Figure 11: Constant hardness $\log_{10} \sigma$ vs. $\log_{10} \dot{\epsilon}$ curves at various temperatures. They are calculated by using the parameters in Table 2 and Equation (4). The measured data deviate from the calculated curve showing the effect of strain aging. Stress values are modulus corrected to those at 200°C.

Figure 12: The value of $M(\dot{\alpha}^*)^{-1/M}$ in Equation (4) vs. temperature, showing non-exponential temperature dependence.

Figure 13: The strain rate dependence of the strain rate sensitivity of flow stress, $\partial \ln \sigma / \partial \ln \dot{\epsilon}$ calculated from load relaxation data, for Zircaloy-4 at various temperature.

Figure 14: The temperature dependence of the strain rate sensitivity of flow stress for Zircaloy-4 at selected strain rates and strain levels.

Figure 1

Figure 2

Figure 3

Figure 4a

Figure 4b

Figure 5a

Figure 5b

FIGURE 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13

Figure 14