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The Effect of Texture and Strain Aging on Creep of Zircaloy-2

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ABSTRACT: Creep tests were carried out on specimens machined from heavily cold-worked Zircaloy-2 sheet material in both the rolling (longitudinal) and thickness (short-transverse) directions. The texture of the sheet was such that the majority of grains had basal-plane normals oriented 30 deg from the sheet-normal direction, and was independent of the metallurgical condition. The creep tests were carried out in the range 100 to 350 C. on both as-received (cold-worked) and annealed material.

The results varied with temperature and stress. At high stresses (all temperatures) such that rupture occurred in under 1000 h, the longitudinal specimens showed higher creep rates than the short-transverse specimens. However, at lower stresses the texture had very little effect on creep either in cold-worked material throughout the temperature range investigated, or in annealed materials between 250 and 310 C. In annealed materials tested at temperatures below 250 and above 310 C the longitudinal specimens had higher creep rates than the short-transverse ones.

The material exhibited strong dynamic strain aging which reached maximum intensity around 300 C. In some tests the creep came to a complete stop after about 1000 h. Activation energy for creep determinations showed little variations with texture, but a very marked peaking in the strain-aging temperature range.

Optical and electron microscope investigations of crept specimens revealed the formation of kink bands and a cell structure, especially in heavily deformed specimens, but little or no twinning.

KEY WORDS: texture, strain aging, creep, Zircaloy-2

Introduction

Zirconium alloys deform primarily by slip on prismatic planes, resulting in a variation of the tensile properties with texture [1-3].² It has been assumed in the past [2] that similar differences will exist during creep, that is, specimens

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² The italic numbers in brackets refer to the list of references appended to this paper.

oriented for prismatic slip will creep faster than those oriented for basal slip. Results with biaxially stressed pressure tubes [4] seemingly confirmed this assumption. On the other hand, uniaxial creep tests carried out with specimens machined from pressure tubes in the axial (longitudinal) and hoop (transverse) directions [5] and therefore with different preferred orientations showed little systematic variation in creep rate even though there were marked differences in their elongations at rupture. In order to clarify the situation, uniaxial creep tests were therefore carried out in the range 100 to 350 C on Zircaloy-2 specimens with widely different textures.

Experimental

The Zircaloy-2 specimens were obtained from thick sheets. Their fabrication details and chemical analyses are described in Table 1. The bulk of creep testing was carried out on batch B material. Round test specimens of 0.160-in. gage diameter and 1.0 in.-gage length were cut from the three principal directions of the sheet, Fig. 1.

The hardness and room temperature tensile properties of the longitudinal and short transverse specimens are given in Table 2. The average grain size of the annealed specimens was 20 microns.

The texture of the sheet was determined by X-ray diffraction using a Siemens texture diffractometer. Both batches had similar textures with the majority of grains having their C axis oriented 20 to 30 deg from the sheet normal direction towards the transverse direction, Fig. 2. The textures were the same in both the cold-worked and annealed condition.

The creep tests were carried out under argon atmosphere in constant-load creep machines manufactured by Instron Corp. A Chevenard creep machine was used to determine the activation energy for creep values by the Dorn temperature cycle method [6]; these tests were in air.

TABLE 1—*Materials classification and chemical analysis.*

Material	Fabrication Details	Constituent Analysis of Principal Alloy Elements, wt %				
		Sn	Fe	Cr	Ni	O
Zircaloy-2 Batch A . . .	Cold-rolled ~ 50% after annealing at 800 C	1.46	0.13	0.09	0.06	0.086
Zircaloy-2 Batch B . . .	Cold-rolled ~ 40% after annealing at 760 C	1.39	0.14	0.10	0.05	0.101

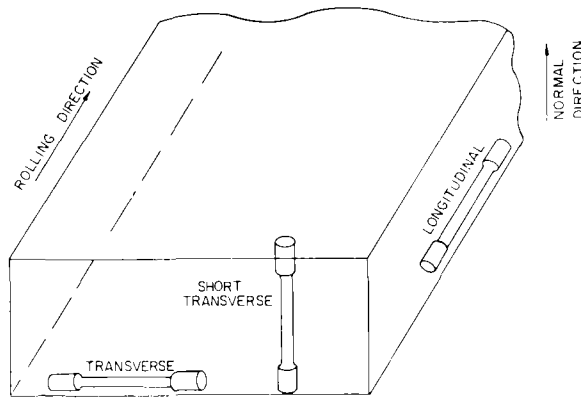


FIG. 1—Specimen orientation in the rolled sheet.

Results

Batch A material was tested in all three principal directions but only at 300 C. The results are summarized in Table 3 with typical creep curves shown in Fig. 3.

Batch B material was tested only in two directions—longitudinal and short transverse in the temperature range 100 to 350 C. Tests with transverse specimens were omitted on the assumption that they would not add any significant new information, because the tension tests of Ells and Cheadle [3] which were carried out on specimens from the same sheet showed that the transverse specimens had yield strengths close to those of the longitudinal specimens. Table 4 summarizes all the creep results in the longitudinal direction and Table 5 does that for the short-transverse specimens.

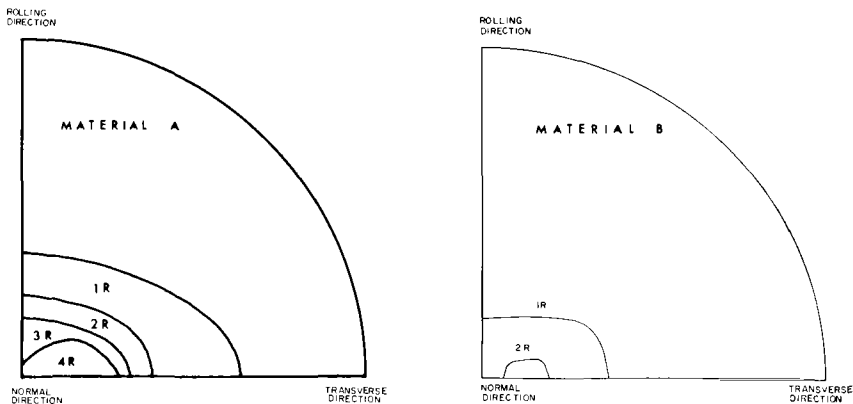


FIG. 2—Conventional (0002) pole figures.

TABLE 2—Room-temperature properties of Zircaloy-2 sheet materials (average of three specimens).

Condition	Orientation	Properties of Material A					Properties of Material B				
		Tensile					Tensile				
		Hardness* VHN	0.2% Y.S., kpsi	U.T.S., kpsi	Un.El., %	Hardness* VHN	0.2% Y.S., kpsi	U.T.S., kpsi	Un.El., %		
Annealed	Longitudinal	158	49.0	65.0	10.0	156	49.8	69.7	13.5		
	Short-transverse	192	73.0	76.0	8.0	193	69.7	75.4	8.5		
Cold-worked	Longitudinal	201	89.5	92.0	2.5	193	81.1	92.5	2.5		
	Short-transverse	236	97.0	100.0	2.0	226	85.3	96.7	1.5		

* Measured in the direction of tensile axis.

TABLE 3—Creep test results, material batch A.

Test No.	Specimen Orientation	Material Condition	Test Temperature, deg C	Stress, kpsi	Loading Strain, %	Duration (h)	Total Strain, %	Minimum Creep Rate (h ⁻¹)
T310.....	Longitudinal	Annealed	300	30	13.4	0.3 ^a	41.0	6×10^{-1}
T280.....	Longitudinal	Cold-worked	300	30	0.46	2 600	0.70	2.0×10^{-7}
T277.....	Longitudinal	Cold-worked	300	50	2.36	0.1 ^a	13.3	...
T311.....	Transverse	Annealed	300	30	4.6	0.2 ^a	32.0	1.4
T279.....	Transverse	Cold-worked	300	30	0.41	2 600	0.62	1.7×10^{-7}
T278.....	Transverse	Cold-worked	300	50	failed on loading		10.1	...
T309.....	Short-transverse	Annealed	300	30	0.36	188	0.81	1.2×10^{-5}
T281.....	Short-transverse	Cold-worked	300	30	0.45	2 600	0.76	2.1×10^{-7}
T276.....	Short-transverse	Cold-worked	300	50	0.76	2 800	2.38	1.7×10^{-6}

^a Specimen ruptured.

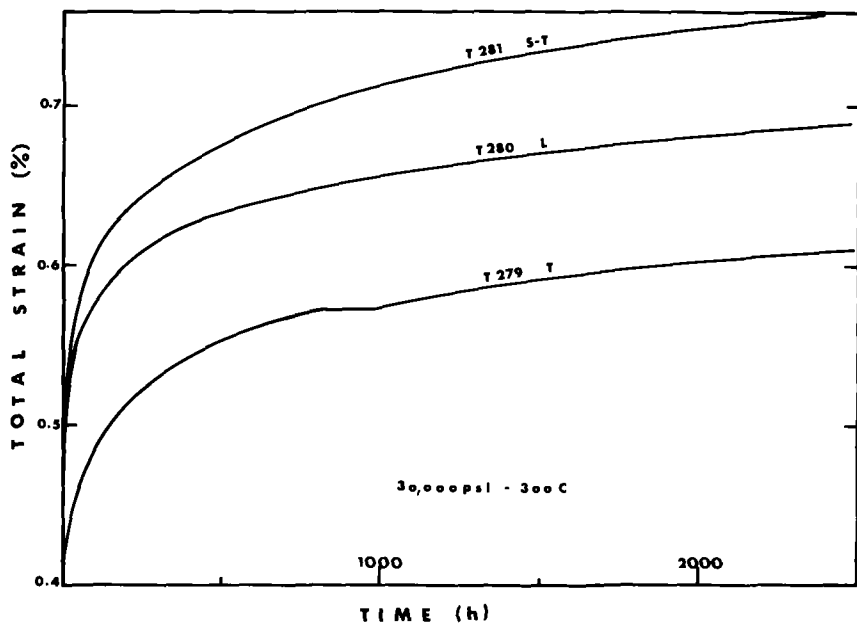


FIG. 3—Creep of cold-worked Zircaloy-2, Material A, at 30,000 psi and 300 C. L = longitudinal, T = transverse, S-T = short-transverse direction.

When comparing the results from these two tables one has to take into account the decreasing creep rate with time. Only a few tests (those that ruptured within 2 h) exhibited all three stages of creep. The remainder followed the relation

$$\epsilon = A t^m + c,$$

where ϵ = creep strain, t = test time, A , m , and c = constants, for test times greater than about 10 h.

The values for m , taken from $\log \epsilon / \log t$ plots (Fig. 4), ranged from nearly zero at around 300 C to 1/3 at 100 C. In some cases m was difficult to determine because the creep rate decreased very rapidly below the limit of resolution of the strain read-out equipment ($\sim 5 \times 10^{-8} \text{ h}^{-1}$). The comparison between the longitudinal and short-transverse specimens is therefore best made after identical test times, as shown in Table 6.

At 300 C and stresses below 50,000 psi, all cold-worked short-transverse specimens exhibited larger primary strains than the longitudinal ones. This trend appears to be reversed at both higher and lower test temperatures (Fig. 4).

Optical and electron microscope investigations of crept specimens revealed the formation of kink bands, Fig. 5, and a cell structure, Fig. 6, especially in

heavily strained specimens. However, even after very prolonged testing at 300 to 350 C at slow rates of straining, the recovery which the cell structure is taken to represent covers only a small fraction of the specimen. Only a few twins were observed in longitudinal creep specimens after 20 to 30 percent deformation. The number of twins present increased with the stress and decrease in test temperature in short-transverse specimens which had more than 0.5 percent strain during the first hour of testing. It was concluded that most of the twinning occurred during or shortly after loading, since specimens with low loading strains (<0.5 percent) but with extensive (10 percent) deformation in creep had only few twins, Fig. 7.

Discussion of Results

In the range 100 to 350 C the texture had little effect on creep at stresses that resulted in rates of straining below approximately $1 \times 10^{-4} \text{h}^{-1}$. The large differences, which one might have expected between longitudinal and short-transverse specimens on the basis of the tensile results, are noticeable only in short-term stress-rupture tests in which the longitudinal specimens exhibited the expected higher creep strains and rates. Under creep conditions (corresponding to creep rates of $\sim 10^{-5} \text{h}^{-1}$ or less) differences were found only below

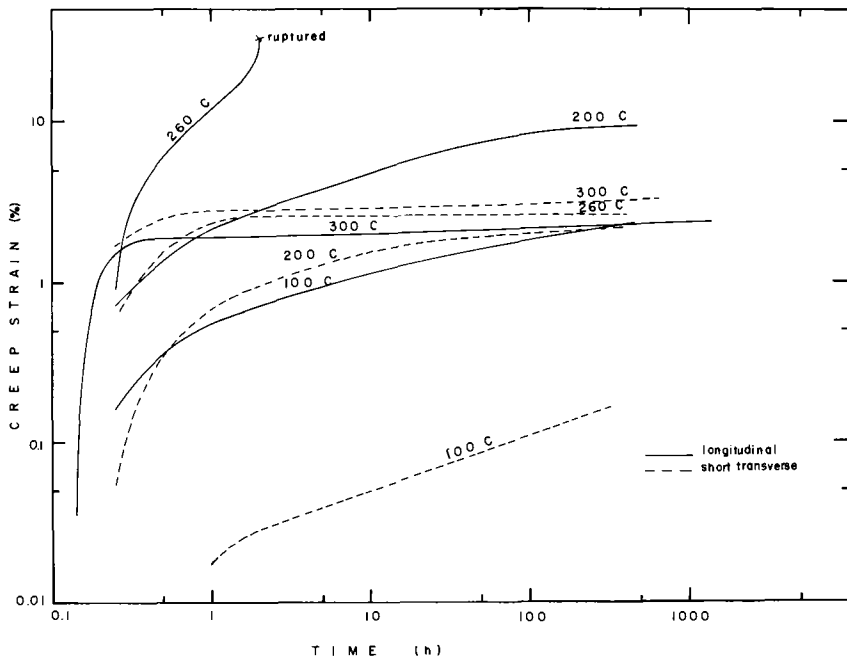


FIG. 4—Effect of temperature on creep of annealed Zircaloy-2, Material B, at 30,000 psi.

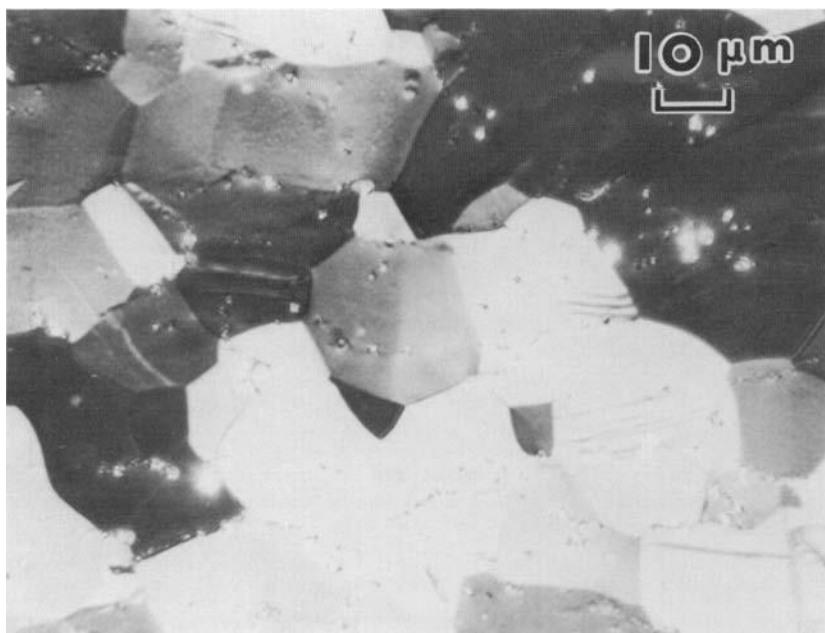


FIG. 5—Optical micrograph of longitudinal creep specimen after 19 percent deformation at 300 C.

about 250 or above 310 C. Between 200 and 310 C the material exhibited strong dynamic strain aging, resulting in a large drop in creep rate (Fig. 8). This effect overshadowed any differences in creep behavior that might have been due to texture.

The strain aging during creep of Zircaloy-2, which has been noted by other workers [5, 7] is very similar to that reported in titanium [8] and can be related to the strain-aging effects observed in short-time tension tests [5, 9, 10] at elevated temperatures. The enhanced creep resistance at 300 C can, furthermore, be directly related to the decrease in ductility [5] which in the short-time tension tests is observed at about 400 C.

The difference in the temperature at which maximum strain-aging effects occur is due to the difference between the strain rates in the two types of test. Decrease in strain rate causes the temperature of maximum strain aging to decrease and its effectiveness to increase [11, 12]. The same argument can be used to explain the difference in the onset temperature of the strain-aging region between longitudinal and short transverse creep specimens, shown in Fig. 8. The data for this figure were obtained from activation-energy tests, during which the temperature was raised in steps of 10 to 20 C every 1 to 2 h. The values for the creep rate shown are those measured near the end of each

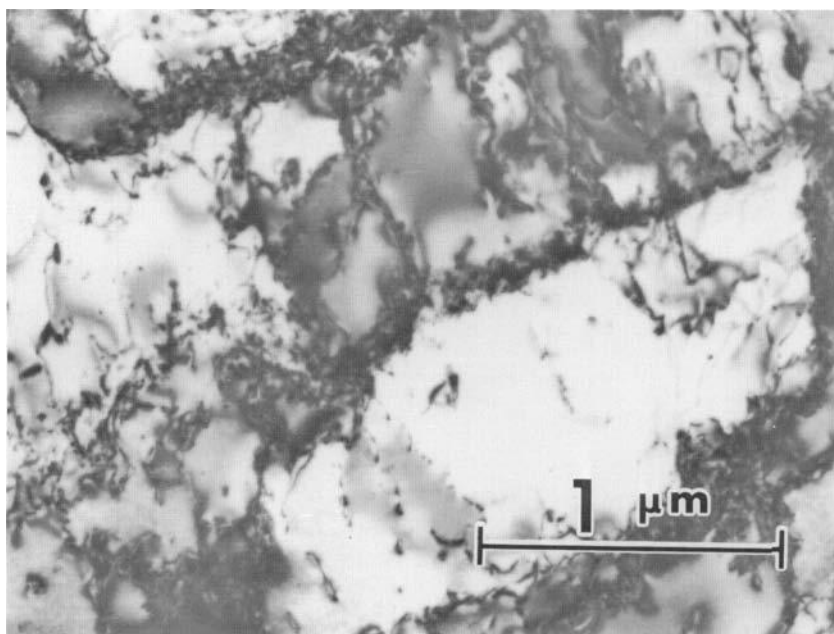


FIG. 6—*Electron transmission micrograph of longitudinal creep specimen after 19 percent deformation at 300 C.*

step. Figure 8 should not be used to derive absolute creep rates at any given temperature, since it does not take into account the variation of creep rate with time. But as a measure of relative behavior, it is quite accurate in that it can be considered to have been determined at approximately constant structure. The whole temperature range was covered in less than 24 h and the total change in strain amounted to less than 0.5 percent.

Comparison of Fig. 8 with the results in Tables 4 and 5 shows a close agreement in the pattern of behavior between the two sets of results. In the range 200 to 350 C, both the longitudinal and short-transverse specimens exhibit a pronounced drop in creep rate, which in some cases (test T 397) comes to a complete stop after 800 h. Corresponding to this region of lower creep rates is a peak in the apparent activation energy for creep *versus* temperature plot, Fig. 9. Assuming a single rate-controlling process, the values for the activation energy are given by

$$Q = \frac{R\Delta T \ln (\dot{\epsilon}_2/\dot{\epsilon}_1)}{T_1 \times T_2},$$

where Q = activation energy for creep, R = gas constant, T_1, T_2 = temperature in deg K, $\dot{\epsilon}_1, \dot{\epsilon}_2$ = creep rates immediately before and after a small change in temperature, ΔT .

At temperatures below about half the melting point (below 650 C for dilute zirconium alloys [13]) one would normally expect the value of the activation energy for creep to be below that for self-diffusion [14], that is, below 46,000 cal/mole [15]. The anomalously high values that were obtained with Zircaloy-2 between 250 and 400 C during this investigation (Fig. 9) as well as by Holmes [16] are attributed to a solute-dislocation interaction. Similar peaking of activation energy for creep has been observed in nickel [17] and aluminum-magnesium alloys [18], and is ascribed to Cottrell locking of dislocations, the activation energy in this range being the sum of that for cross slip and athermal unlocking of dislocations [18]. In explaining their results,

TABLE 4—Creep test results, longitudinal direction, material batch B.

Test No.	Test Temp., deg C	Stress, kpsi	Loading Strain, %	Duration (h)	Total Strain, %	Minimum Creep Rate (h ⁻¹)
COLD-WORKED						
T458a . . .	100	10	0.037	646	0.057	not measurable
T449a . . .	100	20	0.408	360	0.485	1.5×10^{-7}
T433 . . .	100	30	0.566	671	0.667	1.5×10^{-7}
T460a . . .	100	40	0.451	526	0.490	not measurable
T441a . . .	200	20	0.281	358	0.352	3.2×10^{-7}
T432 . . .	200	30	0.333	726	0.448	8×10^{-8}
T443a . . .	300	10	0.165	348	0.218	2.2×10^{-7}
T398 . . .	300	20	0.198	2 660	0.292	8×10^{-8}
T419 . . .	300	30	0.268	503	0.455	9.0×10^{-7}
T430 . . .	300	40	0.561	377	0.898	3.2×10^{-6}
T423 . . .	300	50	1.67	0.33 ^a	11.4	6.7×10^{-2}
T445a . . .	350	20	0.253	356	0.429	1.7×10^{-6}
T439 . . .	350	30	0.577	335	1.179	6.0×10^{-6}
ANNEALED						
T449b . . .	100	20	0.188	359	0.632	3.7×10^{-6}
T425 . . .	100	30	0.651	391	2.930	1.1×10^{-5}
T460b . . .	100	40	3.45	169	39.6 ^b	1.5×10^{-3}
T441b . . .	200	20	0.625	350	1.738	3.0×10^{-6}
T421 . . .	200	30	3.64	463	13.02	1.3×10^{-5}
T447 . . .	260	30	7.99	2.0 ^a	42.6	1.1×10^{-1}
T397 . . .	300	20	1.545	2 688	1.841	not measurable
T405 . . .	300	30	6.65	1 420	9.08	1.4×10^{-6}
T445b . . .	350	20	2.20	300	2.56	4×10^{-6}
T426 . . .	350	30	failed on loading		44	...

^a Specimen ruptured.

^b Reached strain limit of the machine.

TABLE 5—*Creep-test results, short-transverse direction, material batch B.*

Test No.	Test Temp., deg C	Stress, kpsi	Loading Strain, %	Duration (h)	Total Strain, %	Minimum Creep Rate (h ⁻¹)
COLD-WORKED						
T459a...	100	10	0.169	356	0.197	not measurable
T455a...	100	20	0.262	359	0.354	not measurable
T436....	100	30	0.273	647	0.351	5×10^{-8}
T461a...	100	40	0.694	450	0.812	3.0×10^{-7}
T442a...	200	20	0.165	357	0.223	not measurable
T431....	200	30	0.382	670	0.509	8×10^{-8}
T453a...	300	10	0.199	357	0.276	1.5×10^{-7}
T391....	300	20	0.331	1 404	0.565	6.0×10^{-7}
T406a...	300	30	0.396	630	0.671	1.0×10^{-6}
T437....	300	40	0.826	407	1.222	1.5×10^{-6}
T427....	300	50	1.545	13.3 ^a	8.9	8.0×10^{-4}
T457a...	350	20	0.283	359	0.541	2.4×10^{-6}
T440....	350	30	0.506	359	0.868	4.0×10^{-7}
ANNEALED						
T455b...	100	20	0.322	266	0.399	4.2×10^{-7}
T429....	100	30	0.233	311	0.387	1.8×10^{-6}
T461b...	100	40	0.816	350	4.002	2.8×10^{-5}
T442b...	200	20	0.331	300	0.436	8×10^{-7}
T424....	200	30	0.282	382	2.475	6.1×10^{-6}
T448....	260	30	2.575	406	5.233	8.0×10^{-7}
T393....	300	20	0.413	2 149	0.626	5.3×10^{-7}
T406b...	300	30	5.101	629	8.398	3.8×10^{-6}
T457b...	350	20	0.312	320	0.428	1.5×10^{-6}
T428....	350	30	failed on loading		29.5	...

^a Specimen ruptured.

Borch et al [18] dismiss the Cottrell and Jaswon [19] concept of long-range diffusion causing a dragging force due to the formation of an atmosphere of solute atoms behind the moving dislocations on the basis that in this model, the creep rate should be directly proportional to the diffusivity, the activation energy for creep being that for diffusion of the solute atoms. They consider the dislocations in the temperature range of activation-energy peak to be fully locked by solute atoms and can only be moved athermally at a high stress causing an entire avalanche of dislocations. This "breakaway" model is also used to explain both the serrated yielding during tension tests and delayed yielding (incubation creep) in iron-carbon alloys. Arsenault and Weertman [20] in their work on incubation creep of Fe-C alloys have discounted the "breakaway" theory, because it could not account for the stress independence

TABLE 6—Temperature dependence of Zircaloy-2 creep rate at 30,000-psi stress.

Time, h	Annealed				Cold-worked			
	100 C	200 C	300 C	350 C	300 C	350 C	300 C	350 C
	ϵ	ϵ	ϵ	ϵ	ϵ	ϵ	ϵ	ϵ
On Loading...	0.65	3.64	—	6.65	—	0.27	—	—
1.....	1.18	5.72	1.1×10^{-2}	8.4	5.8×10^{-5}	0.28	1.4×10^{-4}	—
10.....	1.73	8.2	1.0×10^{-3}	8.63	3.0×10^{-5}	0.33	2.0×10^{-5}	—
100.....	2.48	12.0	1.2×10^{-4}	8.77	7.5×10^{-6}	0.39	3.4×10^{-6}	—
300.....	2.82	12.9	1.6×10^{-5}	8.86	2.6×10^{-6}	0.43	1.4×10^{-6}	—
LONGITUDINAL SPECIMENS								
On Loading...	0.23	0.28	—	5.10	—	0.39	—	—
1.....	0.25	0.95	2.7×10^{-3}	7.90	1.8×10^{-3}	0.45	2.3×10^{-4}	—
10.....	0.28	1.74	3.2×10^{-4}	7.98	2.9×10^{-5}	0.50	2.3×10^{-5}	—
100.....	0.34	2.26	1.1×10^{-5}	8.13	1.0×10^{-5}	0.58	4.4×10^{-6}	—
300.....	0.39	2.41	7.0×10^{-6}	8.28	6.0×10^{-6}	0.63	1.6×10^{-6}	—
SHORT-TRANSVERSE SPECIMENS								
On Loading...	0.23	0.28	—	5.10	—	0.39	—	—
1.....	0.25	0.95	2.7×10^{-3}	7.90	1.8×10^{-3}	0.45	2.3×10^{-4}	—
10.....	0.28	1.74	3.2×10^{-4}	7.98	2.9×10^{-5}	0.50	2.3×10^{-5}	—
100.....	0.34	2.26	1.1×10^{-5}	8.13	1.0×10^{-5}	0.58	4.4×10^{-6}	—
300.....	0.39	2.41	7.0×10^{-6}	8.28	6.0×10^{-6}	0.63	1.6×10^{-6}	—

NOTE: ϵ = total strain, %; $\dot{\epsilon}$ = creep rate (h^{-1}).

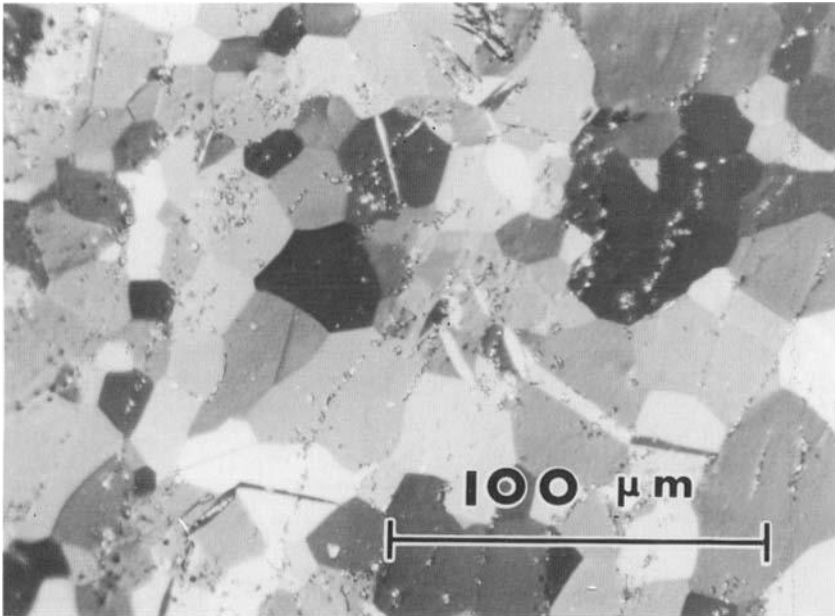


FIG. 7—Optical micrograph of short-transverse creep specimen after 10 percent deformation at 350 C.

of the activation energy of deformation during the incubation period that precedes rapid deformation. Their experimental evidence supports the Cottrell and Jaswon idea of dragging of an interstitial impurity atmosphere by a dislocation. In addition to the long-range diffusion interaction, Arsenault [21] has shown stress-induced ordering in iron-carbon alloys caused by moving dislocations at higher rates of strain than those prevailing during incubation creep experiments which does not require long-range diffusion. Stress-induced ordering was first proposed by Schoeck [22]; it is induced by the stress field of the dislocations and also gives rise to the Snoek peak [23] in internal friction measurements.

The nature of the interaction in Zircaloy-2 which causes the strain-aging behavior during creep around 300 C is not yet known. It has been assumed [24] to be due to interstitial impurities such as oxygen and nitrogen, but the relatively small tetragonal strain fields due to these interstitials result in binding energies of around 0.2 eV, compared to 0.75 eV for carbon in iron. It is interesting to note that Piercy [25] using 0.2 eV for the binding energy calculates a creep rate for atmosphere drag of $6 \times 10^{-6} \text{ h}^{-1}$ at 300 C and 20,000 psi in a Zr-1 percent Sn alloy which is in good agreement with the values obtained during activation energy measurements (Fig. 8), but not with the data

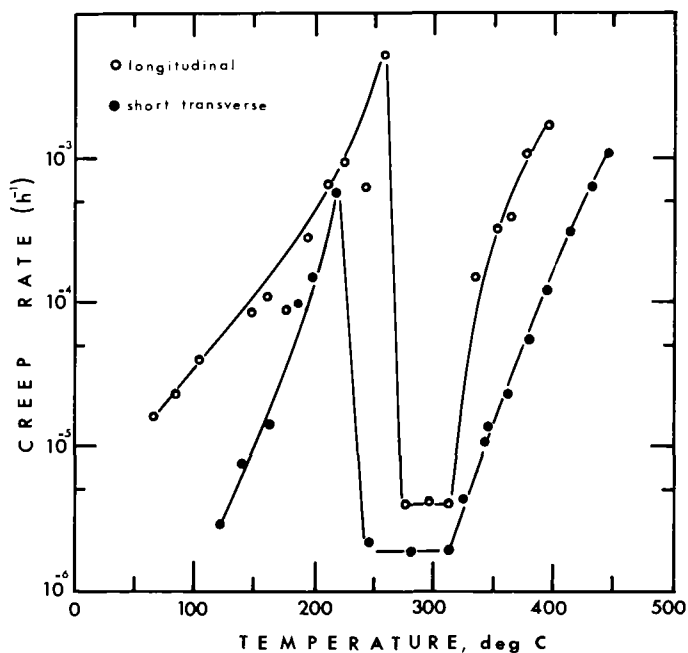


FIG. 8—Variation of creep rate of annealed Zircaloy-2, Material B, with temperature at 20,000 psi (results obtained during activation energy for creep determinations).

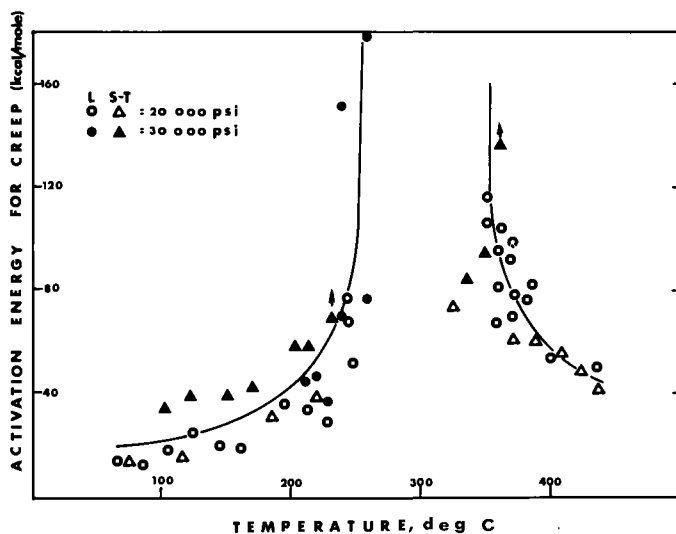


FIG. 9—Temperature dependence of the activation energy for creep of annealed Zircaloy-2, Material B. L = longitudinal, S-T = short-transverse direction.

of Tables 4 and 5 (that is, after prolonged time). It is unlikely that tin has a large effect on the strain-aging interaction in zirconium alloys, since the same athermal creep and peaking of the activation energy values as well as incubation creep was obtained in crystal bar zirconium [26] in which the main impurities were oxygen (200 ppm) and iron (170 ppm). Furthermore, a still higher purity crystal bar zirconium specimen exhibited two activation energy peaks [26], one around 200 C and the other around 450 C. The latter was at considerably higher strain rates and probably can be associated with the stress-induced ordering observed by Arsenault [21] in Fe-C alloys. Recent internal friction measurements in zirconium [27] appear to confirm this point of view, in that an internal friction peak has been found at around 460 C. A similar peak was obtained in titanium-oxygen alloys [28] around 420 C with the peak height a function of the square of the concentration, which was interpreted as an oxygen atom pair orientation process. In ternary titanium-oxygen alloys the characteristics of the internal friction peaks were very different, suggesting a dependence on oxygen-substitutional impurity atom pairs.

The strain-aging effect observed around 300 C (Fig. 8) is therefore most likely to be due to Cottrell locking of dislocations by impurity atmospheres, the creep process then becoming diffusion-controlled.

Conclusions

Texture does not appear to have much of an effect on the strain aging. At strain rates approaching those of tension tests, dislocation pinning by stress-induced ordering becomes effective and probably controls the deformation process at around 450 C, as reported by Veevers and Rotsey [10].

Creep tests that were carried out with specimens oriented for prismatic or basal slip showed that in the temperature range 100 to 350 C texture had little effect at stresses that resulted in rates of straining below approximately $1 \times 10^{-4} \text{ h}^{-1}$. At higher stresses the longitudinal specimens exhibited the expected higher creep rates. Between 250 and 310 C the material exhibited strong dynamic strain aging, which appeared to be independent of texture. The strain-aging interaction is interpreted as Cottrell locking of dislocations by impurity atmospheres.

Acknowledgments

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