Dynamic Strain Aging and Ductility Minima in Zirconium

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Tensile tests using coarse grained zirconium specimens were conducted at two strain rates, differing by 3 orders of magnitude, between 77° and 1032°K. At each strain rate, peaks were observed when the flow stress was plotted against the temperature. The temperature corresponding to a given peak was observed to rise with increasing strain rate. A pronounced minimum in the strain rate sensitivity of zirconium near 675°K can be explained in terms of the strain rate dependence of these peaks. At each strain rate, the zirconium tensile specimens also showed a minimum elongation at the hardening peak temperature. Since the reduction in area did not pass through a corresponding minimum, the elongation minima do not reflect a true ductility loss. What actually takes place is an increased tendency to neck at the hardening peak temperature. This tendency to promote a neck can be rationalized in terms of variations in the strain rate sensitivity caused by dynamic strain aging.

STRAIN aging is a well-known phenomenon that occurs in many materials, including mild steel. When a specimen is deformed beyond the yield point, unloaded, and then reloaded, a sharp yield point is not observed. However, if the specimen is aged sufficiently in the unloaded condition and tested again, the yield point returns. At those temperatures where it is normally studied, this phenomenon usually occurs over a finite period of time. However, strain aging can be explained in terms of dislocation pinning by diffusing impurity atoms[1] so that the kinetics of the process should depend largely on the diffusion rates of the impurity atoms. As a result strain aging phenomena may occur simultaneously with deformation if the temperature is suitably raised. When this happens, the metal is said to undergo dynamic strain aging. The dynamic aspects of strain aging that have been identified are a) the appearance of plateaus or peaks in flow stress-temperature diagrams, b) discontinuous yielding or the Portevin-LeChatelier phenomenon, c) abnormally low strain rate sensitivity, d) abnormal work hardening rates, and e) ductility minima.[2,3] Dynamic strain aging manifests itself, at least to some degree, in one or more of these aspects in most metallic materials.

The study of the variation of the compressive flow stress of columbium (niobium) with temperature by Wessel, France, and Begley[3] is worth noting. They observed that the flow stress, instead of decreasing steadily with a rise in temperature, showed peaks at intermediate temperatures. Two peaks were obtained at a strain rate of 10^{-4} sec^{-1}; one at about 230° and the other at 400°C. On increasing the strain rate by a factor of 10, the peaks moved to higher temperatures, namely, about 300° and 450°C, respectively. These peaks, which will be referred to as hardening peaks, are therefore strain rate dependent. Wessel, France, and Begley attributed the lower temperature peak to oxygen atoms in solution, and the higher temperature peak to nitrogen and/or carbon in solution.

Similar shifts in hardening peak temperatures with changes in strain rate have also been reported in mild steel by Manjoine[4] and Nadai and Manjoine,[5] and in Fe-N as well as Fe-Mn-N alloys by Baird and Jamieson.[6] In the hcp metal titanium, two hardening peaks have also been observed by Monteiro, Santhanam, and Reed-Hill.[7] They used specimens of 16 μ grain size which were tested between 77° and 1133°K at strain rates varying through 5 orders of magnitude.

The significance of these hardening peaks has been more or less ignored. However, at or around the temperature defining the center of these peaks, the other aspects of dynamic strain aging are usually most pronounced. Furthermore, as this paper will show, the abnormal strain rate sensitivity behavior, frequently observed in a dynamic strain aging material, can be related to the strain rate dependence of these peaks. It will also be shown that these peaks are closely associated with the apparent low ductility of some metals in the dynamic strain aging region. This paper specifically refers to data obtained using zirconium specimens, but the results may well have application to other metals.

EXPERIMENTAL

Reactor grade zirconium bar stock was obtained from Reactive Metals, Inc. The average ingot composition of the metal is given in Table I. The bars were swaged to 75 pct reduction in area. Tensile specimens prepared from the swaged rods were recrystallized by annealing in a gettered vacuum for 1 hr at 580°C. They were then critically strained[8] and reannealed in vacuum for 200 hr at 570°C to produce a uniform 0.15 mm grain size. The $1\frac{1}{4}$ in. long by $\frac{1}{8}$ in. diam gage sections

Table I. Average Ingot Analysis of Reactor Grade Zirconium

Element	ppm	Element	ppm	Element	ppm
Al	28	Fe	260	O	1050
B	0.27	H	10	Pb	<20
C	149	Hf	84	Si	49
Ca	<20	Mg	<10	Sn	<20
Cd	<0.25	Mn	<20	Ti	<20
Co	<10	Mo	<20	U	<0 2
Cr	<20	N	36	V	<20
Cu	<20	Na	<10	W	<20
		Ni	<20		

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were chemically machined in a solution of 5 parts by volume H_2O, 4 parts HNO_3, 2 parts HCl, and 1 part HF. Since chemical machining tends to introduce surface hydrides, the gage sections were finished by electropolishing at -55°C in a bath of 59 parts by volume methanol, 35 parts n-butanol, and 6 parts $HClO_4$, using a zirconium cathode. The diameter was measured carefully at intervals along the gage length, using an optical comparator. The specimens were tested in an Instron machine at two different strain rates, 1.3×10^{-5} and 1.3×10^{-2} sec^{-1}, at temperatures from 77° to 1032°K. Pure dry argon atmosphere was employed in the higher temperature tests. In the lower temperature tests the specimens were immersed in suitable refrigerants.

RESULTS

a) Hardening Peaks

The flow stress-temperature curves, corresponding to zirconium specimens deformed to several strains at the higher strain rate, are plotted in Fig. 1. The hardening peaks, indicated by arrows, are not as pronounced as the similar peaks observed in 16 μ grain size titanium specimens. Their small size may be a result of the coarser grain size of the present zirconium specimens. The peaks are easier to see when the

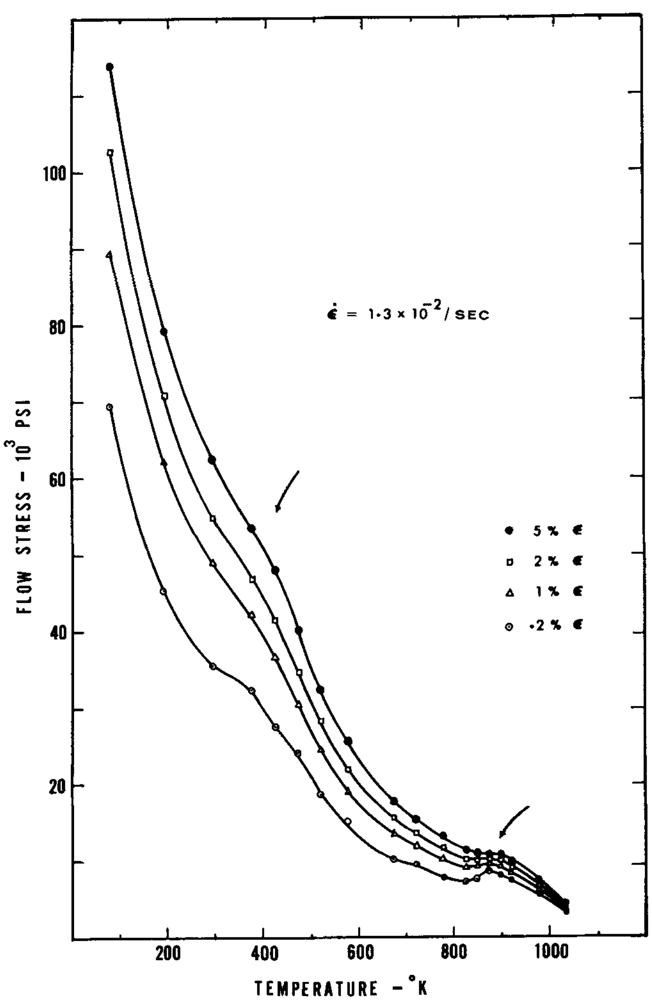

Fig. 1—Flow stress of zirconium for various strains vs temperature at a strain rate of 1.3×10^{-2} sec^{-1}.

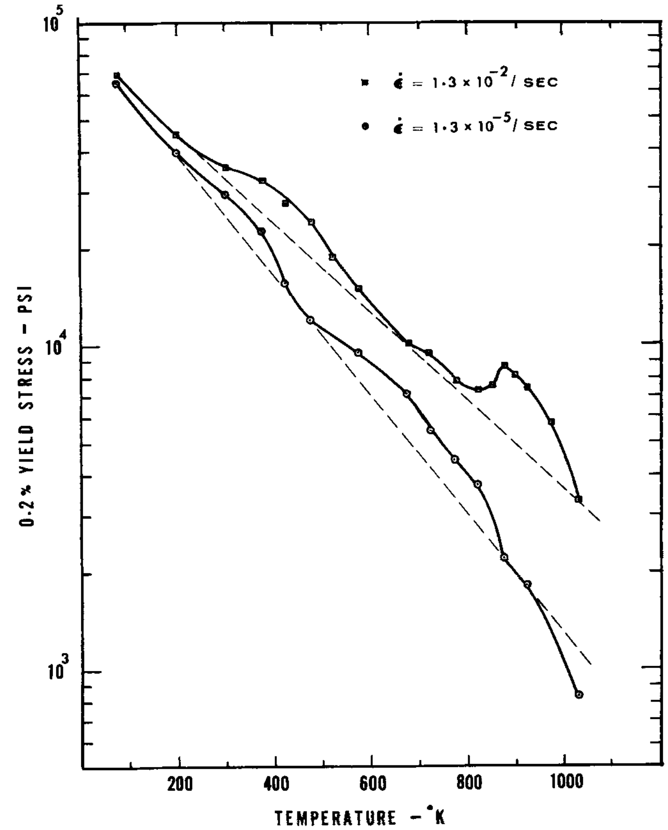

Fig. 2—Logarithm of the 0.2 pct yield stress of zirconium as a function of the temperature.

flow stress data are plotted on semilogarithmic coordinates. This is done in Fig. 2 for 0.2 pct strain. Similar curves have been obtained for the flow stress at higher strains. In general, the logarithm of the flow stress of zirconium and titanium tends to decrease linearly with increasing temperature.[9-11] It has also been observed empirically that positive deviations from these lines occur in the same temperature ranges where those phenomena normally associated with dynamic strain aging are observed.

At the higher strain rate, as may be seen in Fig. 2, the hardening peaks lie at 875° and 425°K, while at the lower strain rate they occur at 675° and about 300°K, respectively. Note that the higher temperature peak is more pronounced than the lower temperature one.

Next consider the high temperature hardening peak. A 3 orders of magnitude strain rate change shifts this peak by about 200 deg. A further increase in strain rate should move this peak to even higher temperatures. In this regard, it is interesting that Simcoe and Thomas[12] obtained a hardening peak in zirconium at 1073°K, using a strain rate equivalent to that employed in hot rolling. This was about $2\frac{1}{2}$ orders of magnitude higher than the faster strain rate of the present work. The location of this peak is thus in reasonable agreement with the present results.

b) Elongation Minima

Fig. 3 shows the elongation of the zirconium specimens as a function of temperature. Note that at each strain rate the elongation tends to be a minimum at the temperature of the hardening peak. This effect, which is most easily seen at the higher temperature hardening peak, suggests a close association between dynamic strain aging and the elongation minima.

In order to check whether the elongation minima are associated with brittle creep-rupture,[13] the specimens were sectioned and examined metallographically. In the specimens deformed at temperatures near the upper hardening peak, some porosity was found. However, similar porosity was also observed, although to a lesser degree, in specimens deformed at other temperatures well above and below the temperature of the hardening peaks. It was therefore concluded that the tensile ductility minima were not due to intergranular fracture of the type discussed by Rhines and Wray.[13]

c) Reduction in Area

In order to check whether the ductility is really a minimum in the dynamic strain aging region and whether the porosity is associated with brittle fracture, the percentage reduction in area of the specimens was measured at all temperatures. The results are shown in Fig. 3 for both strain rates. It is clear that the reduction in area increases continuously with temperature at both strain rates and there is no minimum in the reduction in area corresponding to the elongation minimum. Furthermore, in the upper dynamic strain aging region, the reduction in area is very large, about 90 pct, indicating a basically ductile fracture. Similar features are implied in the published results on columbium (niobium).[3] Thus it is seen that dynamic strain aging conditions reduce the elongation of the metal but not its reduction in area. In other words, in these metals dynamic strain aging is apparently not associated with brittle fracture.

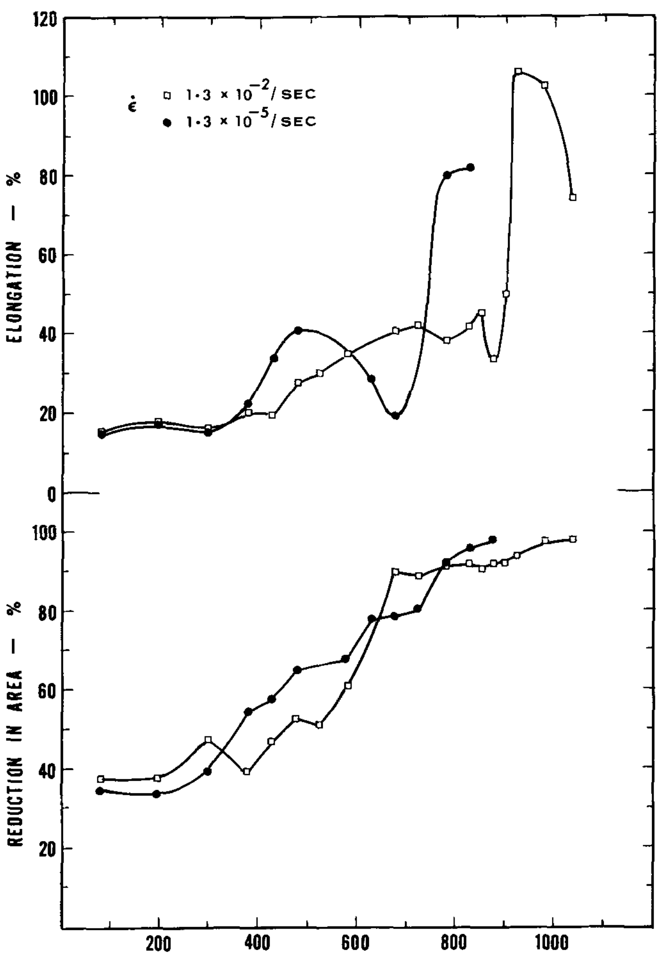

Fig. 3—The percentage elongation and percentage reduction in area of zirconium vs the temperature for the two strain rates.

DISCUSSION

a) Dynamic Strengthening

Dynamic strain aging in zirconium is characterized by the appearance of two peaks in the flow stress-temperature plot. It is believed that they are the result of the interaction of impurity atoms with moving dislocations. Since the hardening peaks are temperature and strain rate dependent, coupling between moving dislocations and diffusing impurity atoms, when the velocities of the dislocations and impurity atoms are roughly comparable, is implied. In other words, at a given strain rate there apparently exists an optimum temperature for interaction between impurity atoms and dislocations.

According to Povolo and Bisogni,[14] there are three internal friction peaks in zirconium due to hydrogen at 144°, 230°, and 292°K. These fall within the observed low temperature dynamic strain aging region. Internal friction peaks due to oxygen, nitrogen, and carbon in zirconium have not been reported. However, in titanium, internal friction peaks due to these elements have been reported in the high temperature dynamic strain aging region at 700°, 788°, and 673°K respectively.[15,16] It is quite probable that similar peaks may also exist in zirconium in the upper dynamic strain aging temperature range. These internal friction results are consistent with an assumption that the low temperature peak in zirconium may be due to dissolved hydrogen and the high temperature peak due to oxygen, nitrogen, and/or carbon. The difference between the two regions is probably only one of degree.

b) Strain Rate Sensitivity

The strain rate sensitivity of zirconium, calculated from yield stress data at the two strain rates, is plotted as a function of temperature in Fig. 4. The general shape of the curve is quite similar to that obtained by

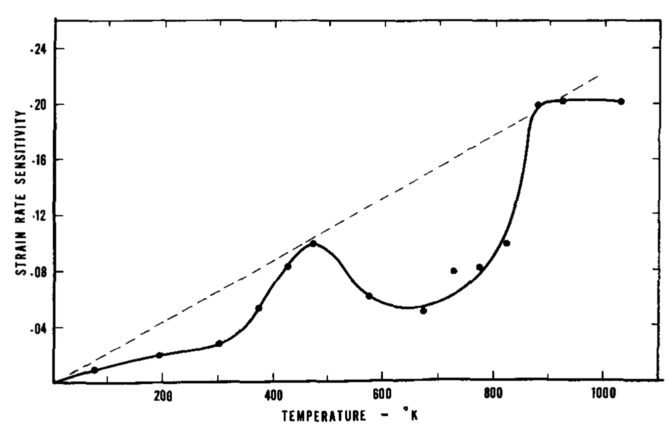

Fig. 4—The effective strain rate sensitivity of zirconium as a function of the temperature.

Ramaswami and Craig[17] for zirconium, and Monteiro *et al.*[11] and Orava *et al.*[18] for titanium. In general, the strain rate sensitivity tends to increase more or less linearly with temperature in a number of metals. When it falls below this straight line dynamic strain aging phenomena are also observed. Note the tendency for the strain rate sensitivity to be low in both the dynamic strain aging region near 300°K as well as that centered about 675°K. The regions of abnormally low strain rate sensitivity in Fig. 4 can be explained in terms of the tendency for the temperature corresponding to the hardening peaks to shift with a change in strain rate. This can be understood from Fig. 2. Let us, for example, consider the higher temperature dynamic strain aging region. At about 675°K the two curves are close to each other, implying a very small effective strain rate sensitivity. Above and below this temperature the curves diverge, suggesting an increase in strain rate sensitivity. Thus one is able to rationalize the strain rate sensitivity minimum at 675° K, shown in Fig. 4. The other minimum at 300°K can also be explained in the same manner. It should be pointed out that the exact temperature of the minimum strain rate sensitivity depends on the choice of strain rates. With a different choice of strain rates, a different temperature should be obtained for the minimum strain rate sensitivity. This is because the peak falls at a different temperature at each strain rate.

c) Elongation Minima

In the dynamic strain aging region, elongation minima are observed which are not accompanied by corresponding minima in the reduction in area. This implies that the elongation minima are associated with an increased tendency towards neck formation. When necking begins it is normally assumed that the increase in stress due to the reduction in specimen cross section area balances the corresponding increase in strength of the material due to strain hardening. However, in addition to these factors, when a neck forms the effective length of the gage section is also markedly decreased. This means that the strain rate in the necked region has to increase. If the strain rate sensitivity is high then the increase in strain rate may increase the resistance of the metal to flow in the neck sufficiently so that deformation tends to occur elsewhere. On the other hand, this does not occur if the strain rate sensitivity is very low or zero, and the concentration of the deformation in the necked region continues, with the result that a sharp neck occurs.

Now let us apply these considerations to the case of zirconium in the upper dynamic strain aging interval. Consider a specimen being deformed at the slower strain rate at a temperature where the effective strain rate sensitivity is a minimum. Therefore, when necking starts, even though there is an increase in strain rate in the necked region, the flow stress should not increase appreciably. Hence, necking should continue in the same region and failure without appreciable elongation should occur.

Now consider a slightly higher temperature where the effective strain rate sensitivity is higher. Here strain rate hardening should take place, tending to force deformation to occur elsewhere in the specimen. The result should be a reduction in the sharpness of the neck. These considerations are in good agreement with the zirconium stress-strain curves, shown in Fig. 5, corresponding to the two temperatures 873° and 923°K and a strain rate 1.3×10^{-2} sec^{-1}. At this strain rate, 873°K corresponds to a minimum effective strain rate sensitivity, while 923°K corresponds to a strain rate sensitivity that is considerably larger. Note that at 873°K, the maximum stress is reached after 17.4 pct strain, but the specimen fails with a total strain of only 34 pct, whereas at 923°K, even though the maximum stress is reached earlier at 11.8 pct strain, the specimen continues to elongate to about 108 pct before failure. Thus the effective strain rate sensitivity is a factor controlling the elongation of the metal. In this connection it is interesting to note a plot from Woodford's summary relating the strain rate sensitivity to the percentage elongation for a variety of materials under different testing conditions.[19] The increase in elongation with increase in strain rate sensitivity over a very wide range is significant. In the present work on zirconium, the temperature dependence of the elongation with a minimum in the dynamic strain aging region is in agreement with this point of view. The present results are also in excellent agreement with the interpretation of the role of strain rate sensitivity in superplasticity by Backofen, Turner, and Avery.[20]

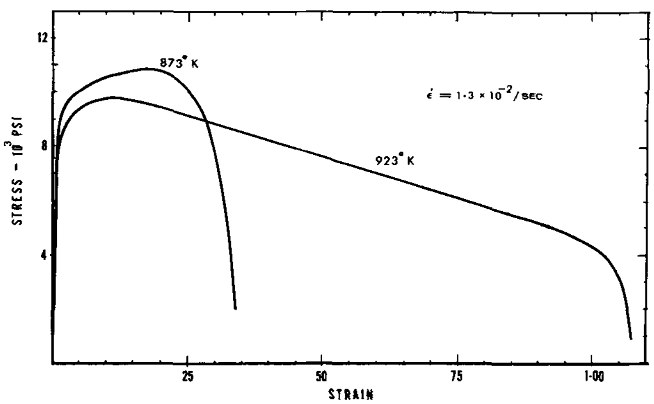

Fig. 5—Stress-strain curves of zirconium at 873° and 923°K. Strain rate 1.3×10^{-2} sec^{-1}.

CONCLUSIONS

1) Two hardening peaks have been observed in the flow stress-temperature curves of coarse grained zirconium. These peaks are characteristic of dynamic strain aging. The low temperature peak is probably due to interactions between dislocations and hydrogen atoms, and the high temperature peak due to oxygen, nitrogen, and/or carbon.

2) The hardening peaks are strain rate dependent, shifting to higher temperatures with increase in strain rate.

3) At the temperature of the center of the peak, the tensile elongation of zirconium tends to be a minimum at each strain rate.

4) The reduction in area, however, is independent of dynamic strain aging. In fact, it is quite large in the upper dynamic strain aging region, indicating ductile fracture.

5) The observed variation of the effective strain rate sensitivity with temperature, with a pronounced

minimum at 675°K, is directly associated with the shift in the hardening peak temperature with changes in strain rate.

6) The prevailing strain rate sensitivity which is a minimum under dynamic strain aging conditions leads to minimum elongation in the dynamic strain aging region.

7) Beyond the dynamic strain aging region, the effective strain rate sensitivity can be large and can lead to large elongations.

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