THE LOW-CYCLE FATIGUE AND CYCLIC BEHAVIOUR OF ZIRCONIUM

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ABSTRACT

Constant strain amplitude cyclic tests carried out on a commercial-purity zirconium in the annealed and in the prestrained (10% tensile) state indicate the occurrence of a cyclic softening stage similar to that previously observed on a commercial-purity titanium. Electron microscopy observations obtained on non-prestrained samples indicate that this stage is associated with the replacement of arrangements of long, screw dislocations initially produced with a dislocation microstructure more typical of fatigue and having lower long-range stress fields. For the samples prestrained in tension, this cyclic softening stage is observed on the σ_{min} - log N curves. The σ_{max} - log N curves in addition are influenced by cyclic softening associated with residual stress relaxation. At the lower strain amplitudes, the σ_{max} - log N curve initially displays cyclic hardening and the residual stress at the start of cycling cannot be taken as the difference between $|\sigma_{max}|$ and $|\sigma_{min}|$. After normalizing the results for the influence of the mean stress, annealed samples and samples chemically polished after prestraining obey the same fatigue life relationship.

KEYWORDS

Cyclic stress-strain behaviour, dislocation arrangements, long-range internal stresses, low-cycle fatigue, tensile prestraining, residual stresses, zirconium, titanium.

INTRODUCTION

The cyclic stress-strain behaviour of zirconium does not appear to have been previously reported. The monotonic deformation behaviour of zirconium is very similar to that of titanium, a material which, for certain compositions, shows an unusual cyclic softening behaviour (Dickson and co-workers, 1976, 1980; Munz, 1972, 1973; Stevenson and Breedis, 1975). The results of Lee (1972), moreover, suggest that recrystallized Zircaloy-2 shows similar softening. The present study was therefore carried out in order to compare the cyclic behaviour of a commercial-purity zirconium to that previously observed for commercial-purity titanium. A second objective of this study was to determine the influence of a tensile prestrain on the low-cycle fatigue behaviour of zirconium.

EXPERIMENTAL PROCEDURE

The material employed was a commercial-purity zirconium containing approximately 0.10 wt % of oxygen. Cylindrical specimens having threaded ends and a diameter of 6.4 mm over the reduced gauge length were employed. After mechanical and chemical polishing of the reduced sections, the samples were annealed for 1 hour at 750°C under vacuum in sealed Vycor tubes gettered with zirconium foil, followed by a new chemical polishing in a solution of 45% H_2O, 45% HNO_3 and 10% HF. The average grain size obtained was approximately 22 µm. Some of the samples were prestrained at room temperature to 10% elongation at a nominal strain rate of $3.3 \times 10^{-4}\ s^{-1}$.

The fatigue tests were carried out at room temperature using an Instron servo-hydraulic model 1331 testing system and Wood's metal grips. Fully reversed pull-push strain cycling, with a sinusoidal waveform and constant total strain amplitudes $\Delta\varepsilon/2$ of ± 0.15 to ± 1.0%, was employed. The product of frequency in Hertz and $\Delta\varepsilon/2$ in % was maintained constant at 0.35. Chosen hysteresis loops were recorded on a minicomputer and/or on an X-Y plotter. Fracture was taken as the start of the rapid semi-logarithmic decrease of maximum stress, $d\sigma_{max}$ / dlog N, associated with the presence of a macroscopic crack.

RESULTS AND DISCUSSION

CYCLIC RESPONSE - ANNEALED SAMPLES

Typical curves of both extreme stresses, σ_{max} and σ_{min}, versus log N are presented in Fig. 1 for different strain amplitudes. At $\Delta\varepsilon/2$ = ± 0.15%, the σ_{max} and σ_{min} curves are initially identical but then separate with a small compressive mean stress resulting. At $\Delta\varepsilon/2$ = ± 0.20 to 0.75% inclusive, the compressive mean stress is observed from the first cycle and varied little through most of the test. At $\Delta\varepsilon/2$ = ± 1.0%, the mean stress during the test is negligible. For $\Delta\varepsilon/2$ = ± 0.20% to 0.60%, similar initial cyclic softening followed by a cyclic hardening stage is observed for both extreme stress curves. For $\Delta\varepsilon/2$ = ± 0.15% the cyclic softening stage only occurs in the σ_{max} - log N curve, the σ_{min} curve actually shows cyclic hardening. At $\Delta\varepsilon/2$ = ± 1.0%, a few cycles of hardening are observed before the cyclic softening stage. Cyclic hardening following this cyclic softening stage is not observed prior to fracture for $\Delta\varepsilon/2$ = ± 0.75 and ± 1.0%.

While it is tempting to ascribe the difference between the σ_{max} and σ_{min} levels to the occurrence of twinning, such a suggestion can be shown to be invalid since a similar mean stress also develops in samples cycled at $\Delta\varepsilon/2$ = ± 0.15% and the number of twins observed after cycling to failure at this low amplitude is very small (less than 0.1% of the volume is twinned) and there are no indications of cyclic twinning-untwinning. A significant difference between σ_{max} and σ_{min}, moreover, was not observed in titanium (Dickson and co-workers, 1976, 1980). A comparison of the monotonic stress-strain curves of zirconium, however, indicated that for strains approximately between 0.23 and 0.72%, the flow stress in compression was initially slightly higher than in tension, and that this difference in flow stress (≤ 24 MPa) was similar to the difference (Fig. 2) between $|\sigma_{max}|$ and $|\sigma_{min}|$ observed in the cyclic tests.

The results of Lee (1972) obtained at constant plastic strain amplitudes on recrystallized Zircaloy-2 show very similar trends although cyclic hardening is not observed following the softening stage. The results on titanium obtained by Dickson and co-workers (1976, 1980) and Stevenson and Breedis (1975), except at the lowest strain amplitudes, show significant cyclic hardening prior to this softening stage, although those obtained by Munz (1972, 1973), who, however, did

not employ high amplitudes, do not. Our electron microscopy observations obtained on zirconium and titanium will be published in detail elsewhere. The observations of Stevenson and Breedis (1975) on titanium have previously been published. These observations show that the initial cyclic hardening observed in annealed titanium is associated with a build-up of long screw dislocations (Fig. 3a). In zirconium, the presence of mainly screw dislocations are also initially observed (Fig. 3b), but their density does not build up significantly. Williams, Sommer and Tung (1972) have shown that, depending on deformation temperature and oxygen concentration, somewhat similar planar arrangements of dislocations can be produced during the monotonic deformation of titanium and that a particularly high long-range stress is associated with these arrangements. The cyclic softening observed is associated with the progressive destruction of this microstructure and its replacement by microstructures having much lower long-range stresses and more typical of fatigue at lower plastic strain amplitudes (Kuhlmann-Wilsdorf and Laird, 1977), such as patches of edge dislocation dipoles (Fig. 3c) or polygonization dipolar walls at higher $\Delta\varepsilon/2$. The occurrence of the cyclic softening stage in annealed commercial-purity zirconium and titanium thus appears to occur primarily as a result of the particularly high long-range or internal stress associated with the dislocation microstructure initially produced. The cyclic softening stage is not observed in Zircaloy-2 at 350°C (Lee and Hill, 1976) or in high-purity titanium (Dickson, Owens and Plumtree, 1973), since, in both cases, the planar arrangements of long, screw dislocations are not expected to form. The results of Munz (1973) indicate that the number of cycles to the start of the cyclic softening stage is influenced by the exact material composition. The subsequent cyclic hardening stage observed at lower amplitudes in the present study is associated with an increase in the density of patches of dipoles and a decrease in the clear regions between patches (Fig. 3d).

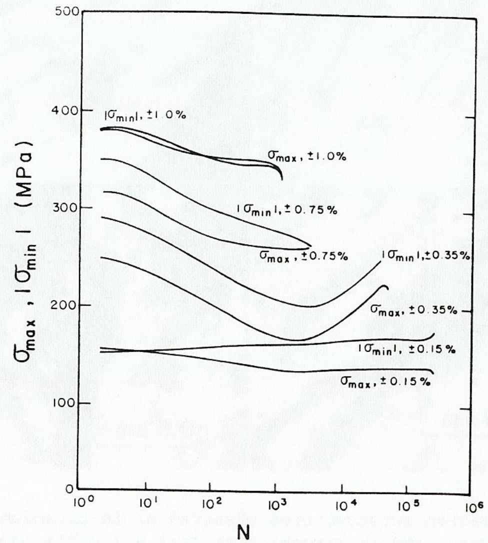

Fig. 1. Cyclic response curves for annealed samples.

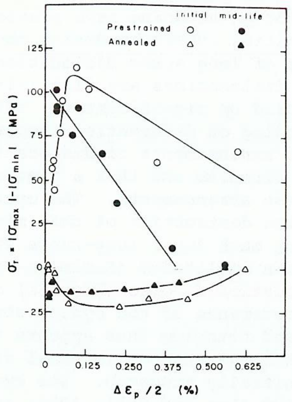

Fig. 2. Curves of σ_r as a function of $\Delta\varepsilon_p/2$ at the start of cycling and at mid-life.

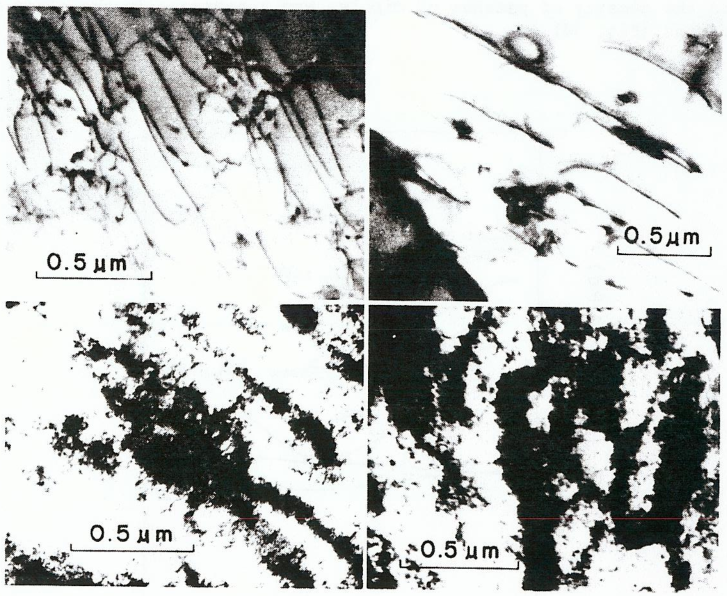

Fig. 3. Dislocation substructures observed a) in titanium at $\Delta\varepsilon/2 = \pm$ 0.25%, 300 cycles and in zirconium at $\Delta\varepsilon/2 = \pm$ 0.35% and b) 10 cycles, c) 1400 cycles and d) 45,000 cycles. Making the same assumptions as Stevenson and Breedis (1976), the long parallel dislocations in a) and b) are screw dislocations. In c) and d), patches of edge dislocation dipoles are observed.

CYCLIC RESPONSE - PRESTRAINED SAMPLES

For samples prestrained 10% in tension and cycled at $\Delta\varepsilon/2 \geq \pm$ 0.50%, relaxation of the residual stress left by the tensile prestrain occurs from the start of the test and influences the σ_{max} - log N curve (Fig. 4). The σ_{min} - log N curves, however, appear to only show the type of cyclic softening observed for the annealed samples, although especially for $\Delta\varepsilon/2$ between $\pm$ 0.45% and $\pm$ 0.75%, the amount of cyclic softening observed on the σ_{min} curves for the prestrained samples is less than that observed for the annealed samples . At $\Delta\varepsilon/2$ of $\pm$ 0.25% and $\pm$ 0.35%, the amount of cyclic softening observed on the σ_{min} curves for the prestrained samples is only slightly less than that observed for the annealed samples. At $\Delta\varepsilon/2 = \pm$0.35%, the σ_{max} curves show some initial cyclic hardening, which causes an increase in $\sigma_r = |\sigma_{max}| - |\sigma_{min}|$, the parameter employed as the measurement of the residual stress. Later in the test, however, some relaxation of residual stress occurs. At $\Delta\varepsilon/2 = \pm$ 0.25% the σ_{max} curve shows some cyclic hardening for approximately the first 1000 cycles, and no significant relaxation of residual stress subsequently occurs. The results obtained at $\Delta\varepsilon/2 = \pm$ 0.35% also clearly indicate that the cyclic softening stage that influences the σ_{min} - log N curve also similarly influences the σ_{max} - log N curve.

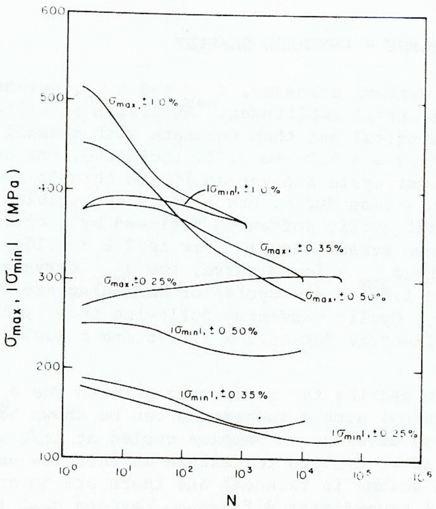

Fig. 4. Cyclic response curves for prestrained samples.

In Fig. 2, the initial residual stress measured and that at mid-life are plotted versus the respective values of plastic strain amplitude, $\Delta\varepsilon_p/2$. The initial value passes through a pronounced maximum with strain amplitude, while the mid-life value shows the more expected behaviour of a continuous decrease with increasing amplitude. This decrease appears to be approximately linear with $\Delta\varepsilon_p/2$. These results therefore suggest that for the lower amplitudes, the value of $\sigma_r^p = |\sigma_{max}| - |\sigma_{min}|$ measured at the start of cycling underestimates the residual stress because of the small value of $\Delta\varepsilon_p$. As cycling proceeds, σ_r increases towards the true residual stress.

Comparing the present results with those obtained on the titanium employed by Dickson and co-workers (1976, 1980), the cyclic softening stage for the annealed samples and on the σ_{min} - log N curves for the prestrained samples occurs more easily in the present material. Since at least in the annealed samples, this cyclic softening appears associated with annihilation of screw dislocations of opposite signs, it should be caused by cross-slip over short distances (Mughrabi, Ackermann and Herz, 1979). Other mechanisms contribute to the relaxation of residual stress, which in contrast occurred more completely in the titanium previously employed than in the present zirconium.

FATIGUE LIFES

Fig. 5 represents Manson-Coffin type plots for the annealed and for the prestrained samples, including for two samples not chemically polished after prestraining. The average plastic strain amplitude for each test, calculated from the recorded hysteresis loops, was employed, although this value of $\Delta\epsilon_p/2$ was only slightly different from that measured at mid-life. The results are not described well by the usual single straight line and, therefore, two straight lines have been employed for the two more complete sets of results. On this basis, except at the highest amplitude ($\Delta\epsilon/2 = \pm 1.0\%$), prestraining reduces the fatigue lifes, especially for samples not chemically polished after the prestrain.

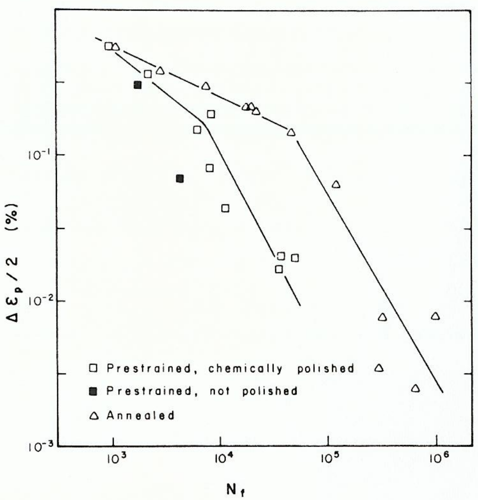

Fig. 5. Manson-Coffin plots for annealed and prestrained samples.

Since except at $\Delta\epsilon/2 = \pm 1.0\%$, the prestrained samples are cycled throughout the test with a significant mean tensile stress and the annealed samples with a smaller mean compressive stress, the parameter suggested by Smith, Watson and Topper (1970),

$$\Delta\sigma'/2 = \{ (\sigma_o + \Delta\sigma/2)\ (\Delta\epsilon/2)\ E\}^{0.5} \quad , \qquad (1)$$

where σ_o is the mean stress and E is Young's modulus, was employed to test the importance of the influence of the mean stress on the reduction of fatigue life

caused by the prestrain. After normalization of the results for this influence (Fig. 6), all results, except for those obtained on samples not chemically polished after prestraining, agree with a single straight line relationship. As expected, there is greater scatter at the lower strain amplitudes. The decrease in fatigue lifes caused by the prestraining therefore can be accounted for by a combination of surface or near-surface defects or stress concentrations produced by the prestrain and of the tensile mean stress that results from this prestrain. The influence of the surface of near-surface defects can be eliminated by chemically polishing after prestraining. After such chemical polishing, twins produced during the prestrain, therefore, are shown to have no influence on fatigue life. A number of previous studies (for example, Golland and Beevers, 1971; Warren and Beevers, 1970) have shown that twinning can have an important influence on fatigue crack initiation and propagation. The reason for this apparent discrepancy is that the twins produced during a prestrain are at the strain amplitudes employed in this study, much less susceptible (Dickson and co-workers, 1973, 1976) to cyclic twinning-untwinning than twins produced during cycling and it is twins that undergo cyclic twinning-untwinning that most strongly influence fatigue cracking.

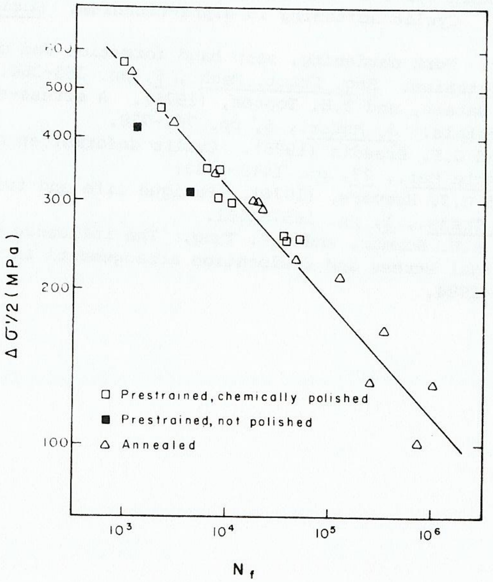

Fig. 6. Fatigue life relationship based on $\Delta\sigma'/2$ parameter.

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