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Rheological analysis of stress change experiments during high temperature creep

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The main results of stress drop experiments during high temperature creep (i.e. the occurrence of zero and sometimes negative creep rates, the existence of two parts with different slopes in the curves of the incubation time Δt_r versus the stress decrement $\Delta\sigma$, the zero or positive values of the stress variations in stress relaxation tests) are analysed from a rheological point of view. The basis of the proposed interpretation is the existence of a creep criterion, represented by a limit surface in the stress space, which explains through the usual plastic flow rules the occurrence of zero or negative creep rates. The work hardening splits into a translation (kinematic hardening) and a deformation of this limit surface. The recovery of kinematic hardening is slower than the recovery of deformation. On this basis, the concept of internal stress used either in the activated glide of dislocations or in the Bailey–Orowan relationship and the nature of negative creep rates are discussed.

1. Introduction

High temperature creep of crystalline solids is often analysed in terms of internal stress. This stress, built up by the long-range actions of the defects contained in the material, has too large a wavelength to be overcome by thermal activation at the strain-rates of the test [1, 2].

Starting from the idea that the recovery of the internal stress σ_i must be small and slow (by definition), Alquist and Nix [3] have proposed determining the average value of σ_i by the so-called "strain transient dip test". For this, one measures the instantaneous strain-rate $\dot{e}_t$, following a rapid drop, $\Delta\sigma$, of the applied stress, σ, produced during stationary creep ($\dot{e}_s$). In many cases it is observed that $\dot{e}_t$ changes from positive values for small $\Delta\sigma$ to negative ones when $\Delta\sigma$ increases. It is then assumed that the value $\Delta\sigma_d$ of $\Delta\sigma$ for which $\dot{e}_t = 0$, is equal to the effective stress σ^* while σ_i is given by

$$\sigma_i = \sigma - \sigma^* \qquad (1)$$

A similar method has been worked out for the relaxation test.

The main argument which favours this method comes from the fact that, in many a case where the apparent activation enthalpy $H = [\partial \ln(\dot{e}_s)/\partial(1/kT)]$ and the coefficient of the power law $m = [\partial \ln(\dot{e}_s)/\partial \ln \sigma]_T$ are functions of the temperature, T, and the applied stress, σ, the corresponding effective values $H^* = -[\partial \ln(\dot{e}_s)/\partial(1/kT)\Delta\sigma_d$ and $m^* = [\partial \ln(\dot{e}_s)/\partial\, \mathrm{kb}\, \Delta\sigma_d]_T$ are constants and H^* can be compared to the activation energies of some diffusion coefficients. This technique has been widely used and some examples can be found in [4–9].

However, some criticisms have been made of this method. Mainly, since the experiments of Mitra and McLean on aluminium and nickel [10], it is known that $\dot{e}_t$ can be zero for a large range of $\Delta\sigma$ and not only for one single value. Mitra and McLean propose then to take the rate of recovery introduced in the Bailey–Orowan equation as

$$r = \lim_{\Delta\sigma \to 0} \frac{\Delta\sigma}{\Delta t_r} \qquad (2)$$

where Δt_r is the incubation time needed by the sample to return to a steady state flow after the stress reduction $\Delta\sigma$ has been made. It has also been said that this incubation time always exists at low values of $\Delta\sigma$ but that it is not always observed because Δt_r may be too small [11]. The fact that Δt_r seems to be zero only for $\Delta\sigma = 0$ has been interpreted by Wilshire *et al.* as a proof that $\sigma^* = 0$ during steady state creep [11].

However, the occurrence of negative creep rates when $\Delta\sigma$ increases is still to be explained. In the critical examinations of the "strain transient dip test" it is commonly proposed that these negative creep rates have to be interpreted as an anelastic contraction following the stress reduction [11]. The origin of the corresponding relaxation could be the bowing of dislocation segments in a viscous way or the sliding of grain boundaries. Moreover, the idea of Mitra and McLean that the incubation period Δt_r represents the time required by the structure to recover to the new steady state value has been criticized by Hart [12] who contends that the zero flow rate is caused by negative anelastic contributions to the strain competing with the positive creep rate.

The aim of this paper is to propose a rheological analysis of these stress reduction experiments. Anelasticity and plasticity can be distinguished through their flow rules, and before any microscopic interpretation of the results, it is necessary to analyse a test, which is more complex than the mere monotonic creep experiment, from a phenomenological point of view.

2. Experimental results

We shall present the main general experimental results to be explained before starting the theoretical analysis. Since many of them can be found in the literature we shall only summarize original experiments that we have carried out on ordered β brasses. The tests were performed at temperatures between 320 and 460° C and with applied stresses ranging from 0.4×10^7 to $2 \times 10^7\,\mathrm{N\,m^{-2}}$. The complete creep properties of these alloys and their dependence on structure and order will be presented elsewhere.

2.1. Curves $\Delta t_r = f(\Delta\sigma)$

In Fig. 1 the incubation time Δt_r is plotted versus $\Delta\sigma$ for different values of the applied stress. The curves show two stages, i.e. a value $\Delta\sigma_t$ exists such that for $\Delta\sigma > \Delta\sigma_t$, Δt_r increases rapidly with $\Delta\sigma$ whereas Δt_r remains small for $\Delta\sigma < \Delta\sigma_t$. This phenomenon is not proper to β brasses. It has been observed, for instance, by Bergman in a 20 Cr/35 Ni austenitic stainless steel and in a γ' hardened nickel-base alloy [13] and by Wilshire *et al.* in Cu, Fe and Zn [11]. But it must be noticed that for

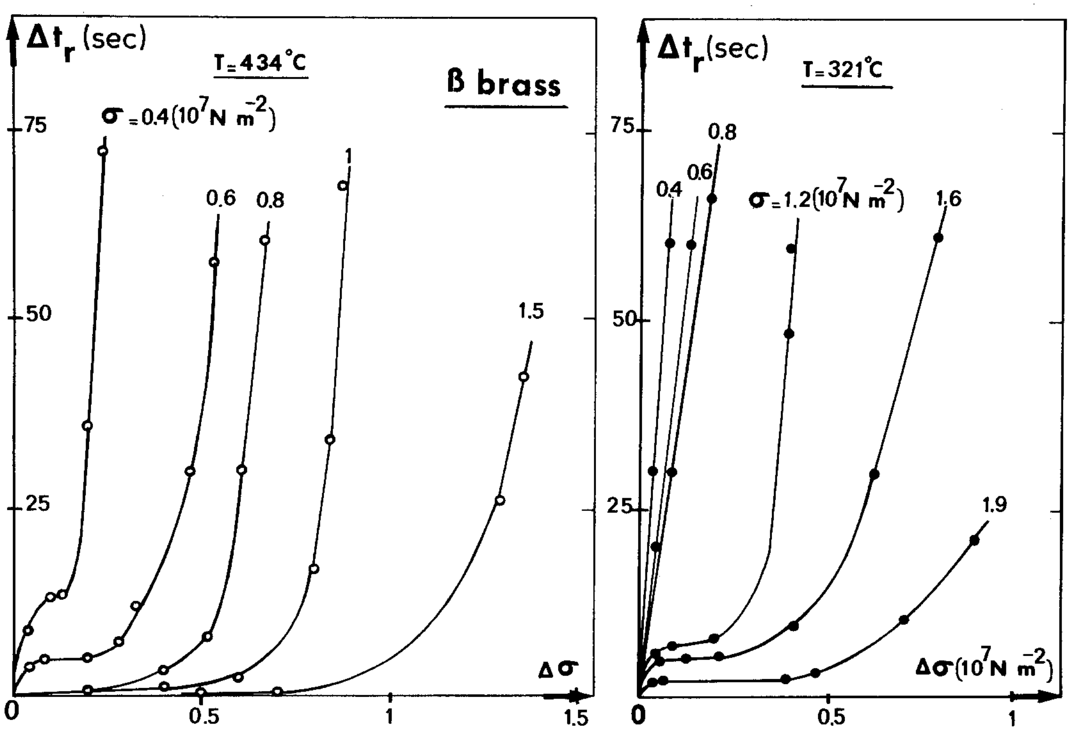

Figure 1 Incubation time versus stress decrement in β' brass.

small $\Delta\sigma$ a positive instantaneous creep rate has often been reported [5, 8].

2.2. Negative transient creep

A critical value $\Delta\sigma_c$ of $\Delta\sigma$ exists such that if $\Delta\sigma > \Delta\sigma_c$ the creep rate $\dot{e}_t$ is negative; this is the phenomenon which has been explained by anelasticity. It has been observed in many alloys. In our case $\Delta\sigma_c$ and $\Delta\sigma_t$ are of the same order, $\sigma - \Delta\sigma_c$ is a decreasing function of the temperature and varies only a little in relation to the stress. This is not a general result and Wilshire *et al.* for instance did not find values of $\Delta\sigma$ giving rise to $\dot{e}_t < 0$ even for $\Delta\sigma > \sigma$ in single crystals [11]. This led them to ascribe the assumed anelasticity (responsible for the negative creep rate) to grain boundary relaxation. Moreover the onset of $\dot{e}_t < 0$ could not be observed even at small stresses in an eutectoid Cu–Al [14].

2.3. Stress transient dip test

The experimental process is the following: on a sample which is straining in the steady state creep, a strain drop is realized followed by a relaxation test at a constant strain equal to the creep deformation minus the strain drop. In β brasses the instantaneous stress can be a decreasing, constant or increasing function of time according to the creep stress and to the strain drop amplitude. Fig. 2 shows an example of a stress transient dip test carried out at large creep stresses on an eutectoid Cu–Al showing a positive instantaneous stress rate (inverse relaxation). It is obvious that a direct correlation exists between $\dot{e}_t < 0$ in creep tests and $d\sigma/dt > 0$ in relaxation tests.

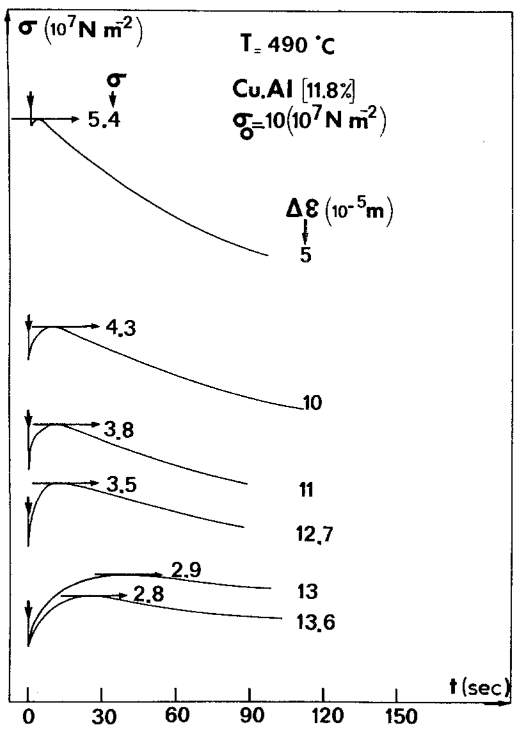

Figure 2 Strain transient dip test for $(\alpha + \gamma_2)$ solution in Cu–Al (11.8 wt %).

2.4. Steady creep rates

In the case where the power law

$$\dot{e}_s = A\sigma^n \tag{3}$$

is to be used, two methods may be applied to measure n: either by testing one sample at each applied stress or by decreasing stress on the same sample and then measuring the steady creep rate. These two methods give different results [10] and in β brass one finds that the stationary creep rate $\dot{e}(-)$ at an applied stress $\sigma_1 = \sigma - \Delta\sigma$, obtained after some steady creep at σ and the drop $\Delta\sigma$, is smaller than $\dot{e}(+)$ obtained when the sample is directly loaded with σ_1. This result is qualitatively similar to those obtained by Bergman [13] and by Mitra and McLean [10] on their own materials.

3. Interpretation of results

3.1. Basis of the analysis

The interpretation must consider the four preceding experimental points and the fact that either the negative creep rates or the increase of stress in the stress transient dip test are not observed in all materials.

One must first point out that the interpretation by anelasticity of negative creep rates after a stress drop [11–13, 15] is not very consistent with the existence of a domain $\Delta\sigma < \Delta\sigma_c$ where $\dot{e}_t$ is zero over periods which can be important. Hart's assumption, which explains zero creep rates as the balance of positive strain flow by a negative anelastic strain rate, is difficult to admit: in the case of β brasses, after stress reductions, the creep rate is definitely zero even when the strain measurements are much more sensitive after the stress drop than before (a magnification of 10^2 to 5×10^2 was used). Such a precision in the equilibrium of two different mechanisms at several temperatures and applied stresses can hardly be accepted. The anelasticity can modify some results but it is not the main phenomenon [16].

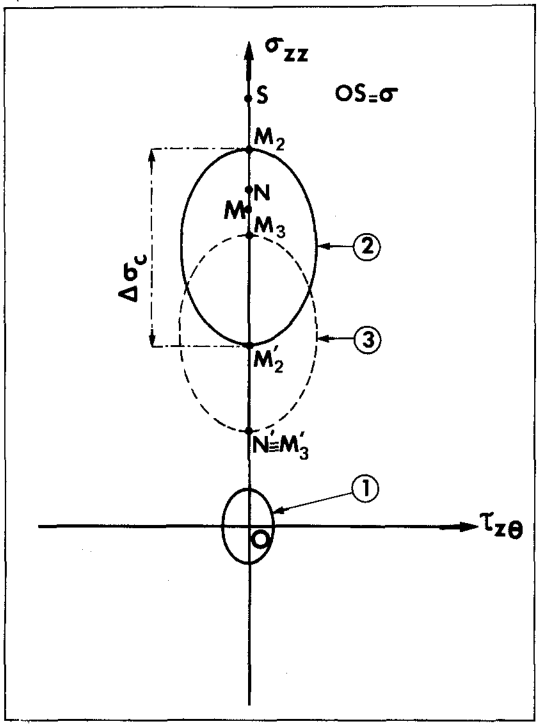

Figure 3 Evolution of the yield locus during creep tests.

A scheme of the rheological analysis that we propose is seen in Fig. 3 where a stress rate in a traction–torsion test can be represented in σ (tension) $\tau_{z\theta}$ (shear stress) axes. The experiments being uniaxial ones, the interest of the $\tau_{z\theta}$ axis is only to make Fig. 3 easier to use. In this reference the closed curve 1 represents a viscous flow criterion, which means that a state of stress, the representative point of which is inside curve 1, cannot give rise to recovery creep (but to instantaneous plastic and/or elastic strain only). In the following, such a curve will be termed a limit curve. On the analogy of the mathematical theory of plasticity, if a Von Mises type criterion is used, then the equation of curve 1 will be:

$$\sigma_{zz}^2 + 3\tau_{z\theta}^2 = Y^2 \tag{4}$$

where Y is the lowest stress value for high temperature creep to occur in a pure tension test [17].

In our case, for the aim of the present paper, determination of the exact equation of curve 1 is useless. We only know that the initial dimensions of curve 1 are small because it is very difficult to detect a threshold stress on a virgin sample.

Curve 2 in Fig. 3 represents the same flow criterion after the stress σ has been applied and the steady state creep reached. The transition from curve 1 to curve 2 corresponds to the primary creep during which the equilibrium between recovery and strain-hardening is not yet established [18]. In Fig. 3, it is seen that the resulting hardening can be split into two parts: (1) isotropic hardening (the limit curve 1 simply expands to dimensions which are fixed by the applied stress) [19]; (2) kinematic hardening (translation of curve 1).

In the studies of plasticity the representative point of the applied stress must be on the limit curve because an increment of the plastic strain demands a stress increment (stability). We shall first consider this case. But during creep this stability does not exist so that it is possible to have the representative point of the applied stress outside the limit curve.

3.2. Explanation of the experimental results

3.2.1. Transient creep rates

During steady state creep the limit curve has come into curve 2 and the representative point of the applied stress σ is in M_2 (Fig. 3). This will correspond to the situation where if a stress reduction $\Delta\sigma < \Delta\sigma_c$ is made, as small as $\Delta\sigma$, $\dot{e}_t$ is zero. This seems to happen in β brass and in other materials [10, 11, 13]. According to the value of $\Delta\sigma$, two cases can be anticipated:

(i) $\Delta\sigma < M_2M_2'$ the representative point of stress after the stress reduction is in N and $\dot{e}_t = 0$;

(ii) $\Delta\sigma > M_2M_2'$ the new representative point is N'. It is clearly established, when a yield criterion exists for plastic flow, that the strain-rate must be normal to the yield locus 2 [19]. Consequently, if N' is in M_2' ($\Delta\sigma = M_2M_2'$), then $\dot{e}_t < 0$. Of course, the same holds true when $\Delta\sigma > M_2M_2'$ (N' being then between 0 and M_2'). Until now we have only considered the case where $\dot{e}_t \leqslant 0$. But as we have said, unlike in low temperature plasticity, we have no stable state, i.e. $\dot{e} \neq 0$, for a constant stress, so that it is also possible to have the representative point in S outside the limit curve 2 and, therefore, to get $\dot{e}_t > 0$ for $\Delta\sigma < M_2S$. This possibility of having $\sigma > 0M_2$ has been discussed by Phillips [20]. In any case, if $\Delta\sigma > \Delta\sigma_c$, with $\sigma - \Delta\sigma_c = OM_2'$, then $\dot{e}_t < 0$. The instantaneous plastic strain produced by $\Delta\sigma$ can either give rise to a cold work which transforms curve 2 in curve 3, M_2M_2' going to M_3M_3' with M_3' on N' (as it would happen in low temperature plasticity) or leave N' outside the locus until the steady creep occurs.

This result implies that the negative strain-rate which can be observed after a stress reduction, $\Delta\sigma$, is not due to anelasticity, but is of the same type as the positive one observed before the stress drop.

When $\dot{e}_t < 0$ is not observed, even on complete unloading of the specimen, one can expect from the above interpretation that M_2' corresponds to a negative stress. There are two possible reasons for this:

(i) the influence of anisotropy which has a great influence on the yield loci [19]. This explains the results obtained on single crystals [11];

(ii) the weakness of kinematic hardening. For instance if work-hardening is but a pure dilation of the limit curve, a stress reduction $\Delta\sigma = 2\sigma$ is needed to obtain a negative value for $\dot{e}_t$. Such a reduced value of the kinematic component of hardening can be the origin of Cu–Al eutectoid behaviour at low stresses. At higher stresses, for this alloy it is possible to obtain $\dot{e}_t < 0$ for $\sigma - \Delta\sigma > 0$ [14].

3.2.2. Effects of $\Delta\sigma$ on Δt_r

The recovery which occurs during either creep or incubation periods, Δt_r, must correspond to strain-hardening and is made up of two components: (1) an isotropic recovery (reduction of curve 2 dimensions); (2) a kinematic recovery (backward translation of 2).

If we admit that steady creep is reached when the strain-hardening is exactly balanced by recovery, then curve 2 is stable. When $\Delta\sigma$ is done, for a new steady state to appear, the limit curve must transform to a new shape and a new position with M_2M_2' going to M_4M_4'. The fact that the curves $\Delta t_r = f(\Delta\sigma)$ distinctly show two parts with different slopes indicates that the two kinds of hardening recover with different kinetics. Δt_r is small when $\Delta\sigma < \Delta\sigma_t$ (with $\Delta\sigma_t$ of the same order as $\Delta\sigma_c$ for β brass). This is the case when it is possible to achieve the complete transition of the uppermost point of the limit curve from M_2 to N by isotropic recovery alone. When $\Delta\sigma > \Delta\sigma_t$ some kinematic recovery is necessary (this is especially evident if $\Delta\sigma > \Delta\sigma_c$). From these observations it may be concluded that kinematic recovery is much slower than isotropic recovery.

Moreover, the initial dimensions of curve 1 are small; if we assume that in some cases the isotropic recovery tends to bring back the dimensions of the yield curve 2 to their initial values (i.e. those of curve 1), we may conclude that $\Delta\sigma_t \simeq \Delta\sigma_c$.

3.3.3. Nature of negative creep

Before any stress reduction, recovery tends to balance the strain-hardening and then to reduce the dimensions of curve 2 and to take it toward lower stresses. When $\Delta\sigma > \Delta\sigma_c$, the recovery has still the same action on the limit curve, but as $\dot{e}_t < 0$, the kinematic hardening will tend to move the limit curve downwards and the isotropic hardening to reduce its dimensions because, for the new stable state of the limit curve to be reached, the plastic strain energy must be positive, i.e. for the positive applied stress $\sigma - \Delta\sigma$, creep rate must be positive. This implies that the uppermost point of the limit curve is N ($ON = \sigma - \Delta\sigma$) or under N.

This means that when $\Delta\sigma > \Delta\sigma_c$, during the transient negative creep, hardening and recovery act in the same direction and can never balance each other. $|\dot{e}_t|$ slows down only because the rates of hardening and recovery slow down too and because the stable state to be reached is with a positive creep rate. It is only when $\Delta\sigma > \sigma$ that a negative steady state creep may be expected.

This shows that negative creep rates are not due to anelasticity. Poirier [21] has given the opposite conclusion but this was reached by assuming that every recoverable strain is anelastic. Two objections can be made to this: it is not a complete definition of anelasticity from the point of view of rheology as far as many different flow rules are included in it [24] and, besides, it dodges the actual questi which is to know whether negative creep rates come from the reversal by internal stresses of the creep mechanism, or if they are due to another recoverable anelastic mechanism (grain-boundary relaxation or bowing of dislocation segments) superposed upon creep and which can be separated from the main creep strain after a stress drop. It must be pointed out that in Poirier's models, negative creep rates are finally attributed to the backward motion of the dislocations which produces the creep strain before the stress drop.

3.3.4. Measurements of n

The fact that n can depend on the experimental technique suggests that the creep rate, $\dot{e}_s$, actually depends on $\sigma - \sigma_i$, where σ_i is a variable, the recovery of which is slow and/or small. Then, it may be compared to kinematic hardening. It is likely that this kinematic hardening is a decreasing function of σ, so that, if we call $\sigma_i(+)$ the internal stress obtained on a sample crept at σ and on which a stress reduction $\Delta\sigma$ has been made, and $\sigma_i(-)$ the internal stress existing on a sample directly loaded at $\sigma - \Delta\sigma$, then we can write $\sigma_i(+) > \sigma_i(-)$ which gives $\dot{e}_s(+) \leqslant \dot{e}_s(-)$ where $\dot{e}_s(+)$ and $\dot{e}_s(-)$ are the two steady state creep rates obtained at $\sigma - \Delta\sigma$ with the two different ways of loading. This result is checked by experiment [11, 13].

4. Possibilities of comparison with creep models

The problem to be considered now is the possibility of comparing the different features of this rheological analysis with the microscopic ones. Of course it is not possible to make a direct comparison with the many possible particular mechanisms which have been already proposed, but we shall rather refer to such an analysis as proposed by Poirier [21, 22] who divides the high temperature creep models into two classes; recovery controlled creep (long-range internal stresses reduced to the level of the applied stress by diffusion-controlled recovery of the substructure and for which thermal activation cannot participate directly in the overcoming of the obstacles), and thermally activated creep, where localized short range obstacles can be overcome by thermal activation (cross-slip, unpinning of attractive junctions or jog drag, ...). Both types of obstacles can be present at the same time.

Fig. 4 shows a situation considered by Poirier where in a force–distance curve the two types of obstacles are present. In this figure, σ_a represents the applied stress before the stress decrement has been realized. The short-range obstacles can be jumped over through thermal activation so that for a stress drop $\Delta\sigma < \sigma_a - (\sigma_i)_m$ the instantaneous creep rate will be positive. On the other hand, the long-range forces represented by the curve of Fig. 4 are actually repulsive forces tending to move the dislocation backwards: they are back stresses and are considered so in the work of Poirier [21] as well as in the strain transient dip test experiments [6]. Therefore, if $\Delta\sigma$ is such that $(\sigma_i)_m < \sigma - \Delta\sigma < (\sigma_i)_M$ we shall first have a little negative motion of the dislocation (instantaneous, if no short-range obstacles exist, or appearing as creep if some viscous drag controls it). But as this

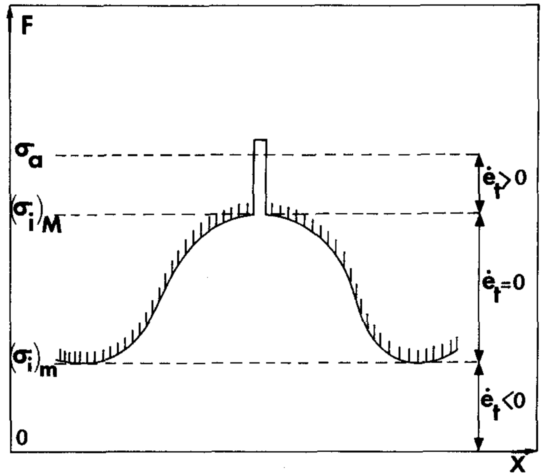

Figure 4 Force–distance curve with a non-uniform internal stress.

motion cannot exceed one half of the average wavelength of the internal stress, the corresponding strain is small. This strain is then followed by a zero strain-rate until recovery has led to a new value of $(\sigma_i)_M$: $(\sigma_i)_{M3} = \sigma - \Delta\sigma$ where again positive creep rate is observed. Finally, if $\Delta\sigma > \sigma - (\sigma_i)_m$, then the back stress is always greater than the new applied stress $\sigma - \Delta\sigma$, therefore, when the dislocation has reached a point where $\sigma_i = (\sigma_i)_m$ the backwards motion goes on (the back stress increasing again until a new σ_i maximum is reached). The negative creep rate will finish when recovery has given a new $(\sigma_i)_m = \sigma - \Delta\sigma$. This last situation has not been considered by Poirier so that the occurrence of negative creep needs, according to his explanations, a friction force due to some viscous drag. Actually, the occurrence of negative creep rates when $\sigma - \Delta\sigma > 0$ only demands that $(\sigma_i)_m > 0$.

As far as our rheological interpretation is concerned one can identify during steady creep, OM_2 and OM_2' with $(\sigma_i)_M$ and $(\sigma_i)_m$, while the distance which may exist between the applied stress and M_2 comes from the existence of short-range obstacles. Therefore, kinematic hardening would come from the occurrence of an internal stress while the isotropic hardening would be due to its non-uniformity. (A perfect isotropic hardening is obtained when $(\sigma_i)_m = -(\sigma_i)_M$, when the long-range energy barriers are perfectly symmetrical). The cases where the "strain transient dip tests" will be working well are those in which $M_2M_2' \neq 0$ and then those where a uniform internal stress exists (purely kinematic hardening).

Another point to be noticed is the strong analogy existing between the Bauschinger effect in low temperature plasticity, and the negative creep observed during stress drop tests at high temperatures and which can be pointed out from the rheological point of view as well as from its interpretation through the existence of internal stresses. It has been shown in Bauschinger effect studies that back stresses are released when they produce a backwards motion of the dislocations and therefore that there is a strong reverse kinematic hardening [23]. This corresponds to a kinematic hardening moving the limit curve downwards in the same sense as recovery, as has been explained in Section 3.3.3.

5. Conclusions

The theory which has been proposed completely explains the principal experimental observations: the existence of a zero transient creep rate for $\Delta\sigma < \Delta\sigma_c$, the existence of a negative strain-rate when $\Delta\sigma_c$ exists and for $\Delta\sigma > \Delta\sigma_c$, the two slopes of the curve $\Delta t_r = f(\Delta\sigma)$, the possible differences between the isotropic samples and the single crystals as far as $\dot{e}_t$ is concerned.

This theory is based on the idea that, from a rheological point of view, the high temperature creep strain is an irreversible permanent strain, and is submitted to the rules of plasticity as far as a yield criterion and the normality law are concerned.

The main differences come from the possibility of the applied stress being outside the limit curve and from the hardening laws. From this analysis several conclusions can be derived:

(1) the hardening can be separated into a kinematical component and an isotropic one;

(2) the kinematic hardening corresponds to a slower recovery than in isotropic hardening;

(3) negative creep rates are not due to anelasticity which may exist, but are in addition to unrecoverable creep;

(4) at the present state of analysis, kinematic hardening can be related to the creation of an internal stress, the inhomogeneity of which gives rise to isotropic hardening. The short-range obstacles, which can be overcome by thermal activation, allow the representative point of the applied stress to be outside the limit curve.

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