

THERMOSTIMULATED CREEP AND STRESS-DEPENDENT CHANGE BETWEEN ATOMIC MECHANISMS(*)

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It is the purpose of this paper to give an improved methodical proposal for creep experiments and an evaluation of creep and activation parameters. The interpretation of the physical processes involved and their participating atomic or more complex lattice defects are discussed.

1. PHYSICAL BACKGROUND—CREEP AND LIFETIME

Sometimes we are interested in pure creep behaviour, but obviously there are some distinct relations between creep and further mechanical properties, too. Macroscopic fracture is always connected with microplasticity, in various materials with different attributions with respect to localization, retardation in time and amount. Fracture is a multi-stage process; the separate stages can involve generation, motion interaction or annihilation of some kind of atomic defects like vacancies, interstitials, solution of impurities, but also multiplication of dislocations, nucleation of pores, microcracks, new phase boundaries and so on.

In a sense, all such stages are for themselves thermally-activated processes and this means that they proceed in time, and even fracture. Therefore, such experimentally observable values like fracture, yield stress, strengthening factors and others, should be dependent not only on temperature and stress, but on the stress-temperature-time-history. Here it seems that some of the material parameters used in solid mechanics, mainly as constants, do not reflect this situation quite well. But it is necessary to take this into account when designing construction elements to be used in a wide temperature range, under variable temperature influences and for dynamical and impact loading; it is also necessary in material technology, mechanical and heat treatments, in failure control etc.

The stress- and temperature dependence of the resulting macroscopic deformation velocity (here only the stationary or secondary creep should be considered) are governed in two different regions by the following phenomenological constitutive laws:

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$$(1.1) \quad \dot{\epsilon}(T, \sigma) = \begin{cases} \frac{AGb}{kT} D_0 e^{-Q/kT} \left(\frac{d}{b}\right)^m \left(\frac{\sigma}{G}\right)^n & \text{for small } \sigma, \\ & \text{but of interest for high } T; \end{cases}$$

$$(1.2) \quad \dot{\epsilon}_0 e^{-\frac{U_0 - \gamma\sigma}{kT}} \quad \begin{cases} \text{for high } \sigma, \\ \text{of interest for low and medium } T. \end{cases}$$

The above laws are not deduced from the original physical principles. Here m, n — dimensionless constants; $G(t)$ — shear modulus, d — grain size, b — Burgers vector, Q — activation energy of self-diffusion, U_0 — activation energy (physical interpretation under dispute), γ — activation volume;

It can be noted that in a few cases the same laws have been observed to govern the mobility of single dislocation lines. Further, one should still take into account the fact that in a large number of experiments on very different classes of materials the life-time τ of samples under stress and isothermal condition was found to obey a law [1] which is just the counterpart to Eq (1.2).

$$(1.3) \quad \tau(T, \sigma) = \tau_0 e^{\frac{U_0 - \gamma\sigma}{kT}}$$

It was found that independently of temperature, stress and material, the value of $\tau_0 \approx 10^{-12} \dots 10^{-14}$ sec was equivalent to the period of the Debye frequency (this is of course an oversimplification permitted for our purpose); for more details see the original literature ([2, 3 and 4]).

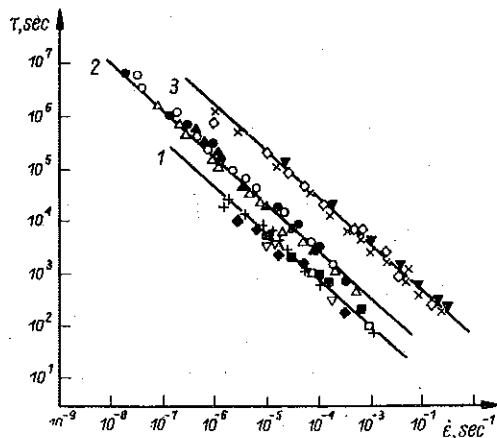


FIG. 1. Correlation between life-time $\tau(T, \sigma)$ and stationary creep-rate $\dot{\epsilon}(T, \sigma)$ for copper (1), high-temperature zircon-alloy (2) with 0.1% Fe+1.1% Cr, and plexiglass (3); each point in this diagram is constructed from two absolutely independent measurements for τ and $\dot{\epsilon}$; points belonging to the same temperature have been obtained for different applied stresses;

temperatures: for copper: ● 673K, +—723K, ◆—773K,
 ▽ 823K, ■—903K, □—973K;
 for Zr-alloy: ○ 693K, ●—723K, △—753K;
 for plexiglass: × 293K, ◇—308K, ▼—333K;

(Fig. 1 is taken from [6], original publications for copper: P. FELTHAM, J. O. MEAKIN Acta met 7, 614 (1968); for Zr-alloy: K. MILIČKA, F. DOBEŠ, Kovove materialy 1976; for plexiglass: M. И. Бессонов, Е. В. Кувшинский, ФТТ 3, 950 (1961).

The second fact established was that in a wide range of applied stresses and temperatures, the deformability of a given material having a definite microstructure was always exhausted at a very specific value of deformation $\Delta\epsilon^*$ [1, 5 and 6]. This agrees well with the combination of Eqs. (1.2) and (1.3):

$$(1.4) \quad \Delta\epsilon^* = \dot{\epsilon}\tau = \text{const.}$$

Remember that $\dot{\epsilon}$ and τ are measured in entirely independent experiments (Fig. 1).

2. WHY BETTER QUANTITATIVE CREEP DATA?

For the verification of Eq. (1.3) a large number of samples has to be stressed, each up to its failure; this means that each sample supplies only one discrete pair of observations $\tau(T_i, \sigma_j)$. In this kind of experiments the mean statistical scatter for a set of samples is large due to remaining micronecks in the surface after preparation and for similar reasons. For a precise evaluation of the parameters, the results should be taken into account both from short time (down to $10^1 \dots 10^{-2}$ sec) and up to long time (up to $10^5 \dots 10^7$ sec) duration, and these should be managed correctly in experiments.

For the verification of the creep rate law (Eqs. (1.1) or (1.2)) usually at relatively low creep rates ($10^{-1} \dots 10^{-4} \text{ sec}^{-1}$) the creep rates are not measured continuously, but are determined from the finite elongation after certain chosen time intervals. This means that some actual fluctuations and retardations have been excluded and lost.

Therefore, it seems to be more natural to use directly the elongation $\Delta\epsilon(T, \sigma)$ which is recorded in the experiment continuously and explicitly.

DORN [2] proposed to perform more than one creep measurement with only one sample, but always after a sudden change of the temperature of the stressed specimen; so it is possible to get some pairs of observations $\dot{\epsilon}(T_i; \sigma = \text{const})$ with identical sample structure, dimensions and so on, and from such pairs he could derive the activation energy value for the creep of that sample.

In practice it is possible to get only a few pairs within one sample; the temperature jump cannot be arbitrarily small, the new temperature should uniformly be approached and the stationarity of creep must be proved.

Later on it will be shown that systematic errors may occur when more than one single thermoactivated process occurs and only very few experimental values $\dot{\epsilon}(T_i, \sigma)$ are available for the evaluation of activation parameters.

3. HOW DOES CREEP DEFORMATION PROCEED WITH INCREASING TEMPERATURE?

A consequent extension of Dorn's idea leads to steadily changing temperature. It was shown to increase with a constant heating rate $q = dT/dt$ [7] in many other physical thermo-stimulated effects, like in thermoluminescence, as a process in the electronic sublattice, and thermo-ionic current, as a kinetic process in the atomic sublattice. Let us investigate how the deformation proceeds with this prescribed temperature-time history.

In a common form instead of Eqs. (1.1) or (1.2) and for a constant stress $\sigma_c = \text{const}$ the derivative with respect to temperature $T(t)$ is

$$(3.1) \quad \frac{d\varepsilon}{dT} = \frac{B(\sigma_c, T)}{q} e^{-U(\sigma_c)/kT}$$

$$\text{with } U(\sigma_c) = \begin{cases} Q & \text{or} \\ U_0 - \gamma\sigma_c & \end{cases} \quad B(\sigma; T) \text{ from Eqs. (1.1) or (1.2).}$$

Then the increase of elongation with temperature (or time), starting the heating from a certain low temperature T_0 (in the limit $T_0 \rightarrow 0$), is

$$(3.2) \quad \Delta\varepsilon(T; \sigma_c) \equiv \varepsilon(T, \sigma_c) - \varepsilon(T_0) = \frac{1}{q} \int_{T_0}^T B(\sigma_c; T') e^{-U(\sigma_c)/kT'} dT'.$$

In comparison to the exponential term in the integral $B(\sigma_c; T')$ is a very slowly varying function of temperature; for instance, for most materials (and without phase changes) we obtain $\frac{1}{G} \frac{dG'}{dT} = 0,5 \cdot 10^{-3}/K$. This means that even above temperature of 200 degrees the shear modulus changes only by 10%.

Also the direct reciprocal temperature influence $B \sim \frac{1}{T}$ for processes like Eq. (1.1) can be accounted (see Appendix 1); for the qualitative purpose we may take as a constant the mean value $B(\sigma_c, T') \cong B(\sigma_c)$ and for the case of Eq. (1.2) this is fully correct. Now one can take into account the fact that physical processes occur always at temperatures at which $U/kT \gg 10$. So one gets

$$(3.3) \quad \Delta\varepsilon(T; \sigma_c) = \frac{B(\sigma_c)}{q} \int_0^{T_0} e^{-U/kT} dT' = \frac{B(\sigma_c) kT^2}{qu(\sigma_c)} e^{-U(\sigma_c)/kT} \eta \left(\frac{kT}{U} \right).$$

The auxiliary function $\eta \left(\frac{kT}{U} \right)$ varies very slowly between 0.95 and 0.85 for values of $\frac{kT}{U} = 0.01 \dots 0.05$ [8]. This again for our purpose allows us to neglect the η — dependence in further considerations and to take approximately $\eta \cong 1$. Representation of the transformed observations drawn in the coordinates $\ln(\Delta\varepsilon) = f(1/T)$ is a practically straight line [9].

$$(3.4) \quad \ln \Delta\varepsilon = -\frac{U(\sigma_c)}{kT} + 2 \ln T + \ln \left(\frac{Bk\eta}{qU} \right)$$

with the slope expressed in terms of the activation energy

$$(3.5) \quad \frac{d \ln \Delta\varepsilon}{d(1/kT)} = -U(\sigma_c) - 2kT.$$

Still more unique is a corrected ordinate $\ln\left(\frac{\Delta\varepsilon}{T^2}\right)$ which tends to a completely unique slope of the elongation curve:

$$(3.6) \quad \frac{d \ln(\Delta\varepsilon/T^2)}{d(1/kT)} = -U(\sigma_e).$$

For convenience these curves $\ln(\Delta\varepsilon)$ or $\ln(\Delta\varepsilon/T^2)$ as functions of $\frac{1}{T}$, $\frac{1}{kT}$ or $\frac{T_m}{T}$ shall be named thermostimulated creep curves (T_m melting temperature).

4. MANY ACTING PROCESSES

A single thermoactivated process starts at any low temperature as was mentioned above and proceeds in the proposed coordinates along a straight line according to the schematic Fig. 2 starting at the right lower side.

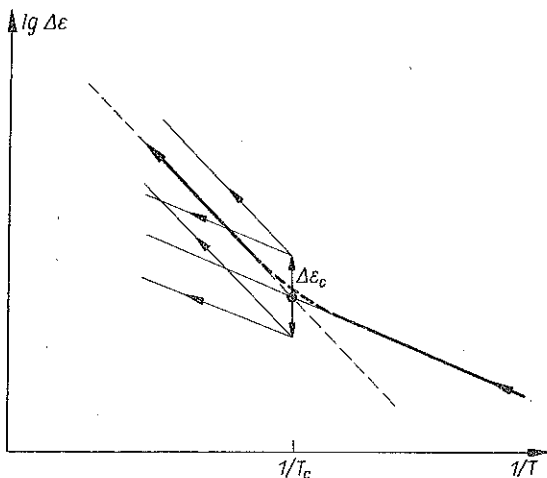


FIG. 2. Straight thermostimulated creep curve and various possibilities of additional instabilities in the moment the temperature T_c is reached (see text!).

Let us assume now that at any higher temperature T_c the matrix undergoes a phase change. Then this can be connected with a specific additional elongation $\Delta\varepsilon_c$, which can be positive or negative (Fig. 2). This change can be instantaneous or with a small delay in time. When the matrix is not strongly disturbed, a similar moving defect can continue its action; this means with nearly the same activation energy (and nearly the same slope of the curve). Such relatively unimportant changes can, for instance, be a magnetic phase transition or some changes of the dislocation network. More important changes like transitions in the crystallographic lattice type will, even for the similar moving atomic defect, result in another slope. The analogous slope change appears if another kind of defects replaces the first

kind. A special case can be the limited coexistence of two acting defect types, then the slope will show a continuous transition.

Our special interest now are processes described by the laws (1.1) and (1.2). For a given σ_c let us seek for such temperature $T_{21}(\sigma_c)$, (here $i=1$ and 2 corresponds to processes described by Eqs. (1.1) or (1.2)), at which the momentum contribution of the second process becomes greater than that of the first. This will be

$$(4.1) \quad T_{21}(\sigma_c) = \frac{U_2(\sigma_c) - U_1}{k} / \ln \left(\frac{B_2 U_1}{B_1(\sigma_c; n, d) U_2(\sigma_c)} \right)$$

and (see Appendix 2)

$$(4.2) \quad \frac{dT_{21}(\sigma_c)}{d\sigma} = \frac{kT_{21}^2}{\sigma} \cdot \frac{n - \frac{\gamma\sigma}{kT} \left(1 + \frac{kT}{U_0 - \gamma\sigma} \right)}{U_0 - Q + kT - \gamma\sigma}$$

The last equations mean that T_{21} decreases when the stress $\sigma > nkT/\gamma$ is further increased; also the transitions occur earlier in fine rather than coarse-grained materials. Schematically this is illustrated in Fig. 3a. Before the transition points the

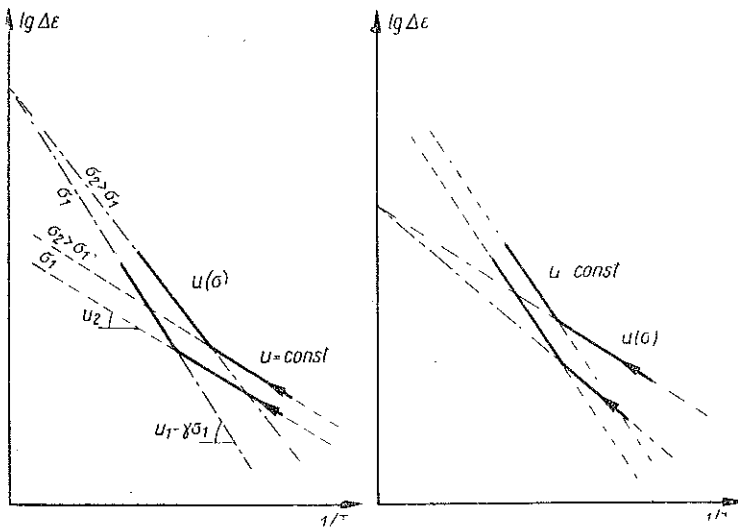


FIG. 3. Shift of thermostimulated creep curves and typical transition points with different applied stresses $\sigma_2 > \sigma_1$; a) the process with stress-dependent activation energy $u(\sigma)$ is realized above T ; it starts earlier and is more important the higher the stress is; case usually expected for metals, b) inverse succession of processes with stress-dependent and stress-independent activation energies

same slope is always observed, whereas afterwards the slopes of curves for different stresses are different.

It should be mentioned that the transition temperature T_{21} is not affected by the heating rate q , as an experimental condition, although the creep curve, Eqs. (3.3) or (3.4), can slightly be shifted if one chooses another q .

It will be shown later that the processes (1.1) and (1.2) in many metals can be due to distinct atomic processes, especially to vacancy and to interstitial atomic

mass transfer. Therefore, in some materials it may be possible that the atomic mechanisms transform into one another in a reverse sense; then a situation, as indicated in Fig. 3b, will occur.

For instance, an analogous reverse effect occurred with diffusion. In all known cases diffusion in metals at high temperatures is due to vacancy migration, except for silicon in the case of which many arguments show that interstitials are the predominant migrating particles. As a matter, silicon is a material of great interest due to its importance in semiconductor devices and in microelectronic technologies at high temperatures. Here, certain internal stresses can appear due to thermal and concentration gradients, phase boundaries and misfit situations. However, there are as yet no available rigorous creep data for silicon at medium and high temperatures. Neither have creep curves like those shown in Fig. 3b been found for other materials.

5. TRANSITION TEMPERATURE TRACE AND THE VACANCY-INTERSTITIAL HYPOTHESIS

A further consequence of Eq. (4.1) is the strong dependency of T_c on the exponent n for which typical constant values are between $n=1$ and $n=5$ for many metals. In Fig. 4 a sketch is drawn, which shows the action of the two creep me-

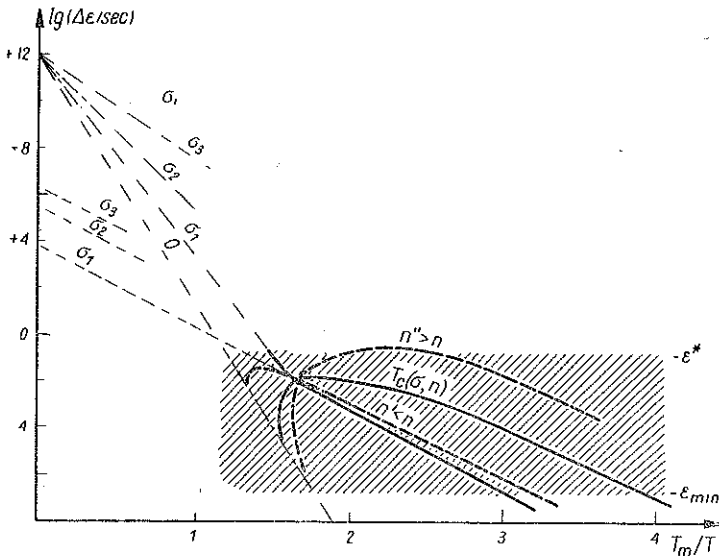


FIG. 4. Trace-line for the dependency of the transition-temperature on stress and on the exponent n above the trace-line creep-law (1.2) is acting and the activation-energy is stress-dependent.

chanisms (1.1) and (1.2) for some stresses σ_i and their combined transition effect in T_c ; as a function of stress this yields a trace line $T_c(\sigma; n)$. Schematically it is shown that the region where the first creep law is predominant (that means below this trace line) becomes more important for higher n and vice versa.

It is also indicated (the shaded area) that creep and creep-rates can practically be observed only in some limited region, in fact, of the lowest measurable elongation $\Delta\epsilon_{\min}$ to the upper fracture limit $\Delta\epsilon^*$, and correspondingly from a certain experimentally lowest temperature to the highest temperature well below the melting point.

Perhaps a short time deviation may rarely be observed in the immediate vicinity of the starting point (finite temperature sudden loading) due to infinity of $\lg(\Delta\epsilon)$ for $\Delta\epsilon \rightarrow 0$ and for primary creep, too.

But of direct interest, in spite of physical interpretation, is the curvature in the neighbourhood of any transition $T_c(\sigma)$; it seems that only by means of a continuously measured and mathematically transformed thermostimulated creep curve can details of the transitions be evaluated. Let us keep in mind that for isothermal creep elongation in fixed time intervals or for isothermal creep rate data only few observable points of the representation $\lg(\Delta\epsilon)$ or $\lg(\dot{\epsilon}) \sim f(1/T)$ are obtained and that they usually contain some data scattering. In the case of, for instance, four points as indicated in Fig. 5 and even if there is no individual scatter, authors usually expect

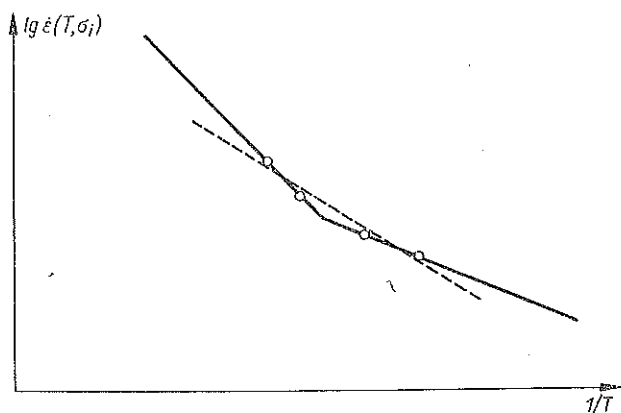


FIG. 5. Sketch illustrating the approximation of few experimental creep-rate points (●) and artificial error in the estimation of an effective activation energy.

———— possible creep-rate dependence, similar to fig. 2 and 3; - - - - - averaged linear approximation.

some scatter and the best they can do is to calculate an averaged linear approximation. This is incorrect and the obtained "effective" activation energy is physically meaningless. Rarely has the jump in the values of the activation energy been as seriously investigated as this was done by MONTEMAYOR et al. [10] for KBr below and above 487°C or by ROHDE and PRIT [11] for nickel above and below 130 K.

Many of the published creep activation energy data should be re-analysed for their experimental conditions, especially when these values are compared with activation energy for diffusion or with heat of sublimation and when their relatively small differences are used as an evidence for any mechanism model. It should be noticed that for high stresses the numerical value of $U_0 - \gamma\sigma$ can reach the same

order as Q ; i. e. nearly the same activation energy, but two qualitatively different processes! This also demands a high performance of the experimental conditions and the measurement records.

It now seems that more precise thermostimulated creep curves would be helpful in the interpretation of the reliability of the transition between the two laws (1.1) and (1.2) and the changes in the underlying physical processes. INDENBOM [12] suggested that in a sample under stress the chemical potential is changed in such a way that sinks of vacancies become sources of interstitials, so that by both processes atoms are transferred in the same direction. A further consequence will be that plastic deformation at medium temperatures caused by high stress is due to generation and migration of interstitials. It has been supposed for a long time that interstitials are annealed at much lower temperatures; but even if their concentration and generation is lower than that of vacancies, their high mobility yields a strong plastic contribution.

CONCLUSION

The method proposed for investigating the creep behaviour of materials is characterized by thermostimulation with a constant heating rate and a fixed stress in the sample. It is convenient to transform the recorded experimental observations to the following variables:

$$\lg \Delta \varepsilon \left(\text{or } \lg \frac{\Delta \varepsilon}{T} \text{ or } \lg \frac{\Delta \varepsilon}{T^2} \right) \quad \text{and} \quad \frac{1}{T} \left(\text{or } \frac{1}{kT} \text{ or } \frac{Tm}{T} \right).$$

From these thermostimulated creep curves the appearance of single thermoactivated processes, of their superposition and of other transitions can be observed; and from the slope of these creep curves the activation parameters for the atomic plasticity processes can be evaluated. Concluding, some advantages regarding discrete isothermal creep data should be mentioned:

It is possible to follow the creep behaviour continuously, with only one specimen and for one applied stress, over the whole temperature range (up to fracture).

This method is efficient from the point of view of the investigation time and the number of necessary specimens.

It allows to avoid much of the scatter in the experimentally determined isothermal values $\dot{\varepsilon}(T_i; \sigma)$ or $\tau(T_i, \sigma)$ due to structural and dimensional differences between different samples.

The qualitative results of structural changes or instabilities with instantaneous or transient effects on the elongation or deformation rate can easily be seen directly from the curves.

In every region in which only a single physical process is acting or dominating, the characteristic activation energy can be determined quantitatively.

A transition in the mass transporting process will give an instantaneous or a delayed change of the slope of the thermostimulated creep curves,

A systematic comparison of the slope and the activation energy values for experiments under different stresses makes it possible to determine the stress dependence of $U(\sigma) = U_0 - \gamma\sigma$ and also of the other activation parameters $B(\sigma; T)$, γ , n .

It should be possible and useful to investigate the influence of various material states, after initial deformation, ageing and annealing, by alloying and doping, after and during irradiation, reactions with gases and moisture at the surface and in the bulk.

Of special interest can be a comparison of a series of specimens well distinguished only in grain size to evaluate the parameter m in the law (1.1) or other characteristic length in the microstructure, for instance cell size or, in polymers, regions of partial ordering.

It will also be of interest to vary the type of loads, and apply also combined torsion, shear, compression and cyclic loading, and to look for their influence on the expired mechanisms and their activation parameters.

Finally, the principle of this method is applicable to every kind of plastic materials, not only for pure metals, but also for polymers [13], superplastic alloys, salts and soils.

New accurately determined activation parameters will facilitate the interpretation of the physical processes, the moving defects, the barriers, sinks and springs. They are more objective than some of the material constants and internal variables used in solid mechanics, used there actually because of the lack of better knowledge.

It will be possible to draw for a given material of any definite structure something like an improved deformation mechanism map (see Fig. 4) as compared to the maps proposed by ASHBY [14]: mechanism-stress-temperature, or by MOHAMED and LANGDON [15]: mechanism-grain size-stress at a fixed temperature.

For technical purposes the behaviour in long time exploitation and at various temperatures can be extrapolated in a more reliable way.

APPENDIX I

The integral

$$(A1) \quad \int_0^T \frac{e^{-U/kT'}}{T'} dT' = \frac{kT}{U} e^{-U/kT} (1 + y\eta)$$

can be evaluated by integration by parts

$$(A2) \quad \int_0^T e^{-U/kT'} dT' = \frac{kT^2}{U} e^{-U/kT} \eta.$$

Here the following notations have been used [7, 8]:

$$y \equiv T/u \ll 1,$$

$$\eta(y) \equiv 1 - R(y),$$

$$R(y) \equiv \sum_{n=1}^{1/y} (-1)^{n+1} (n+1)! y^n - \text{a semi-convergent series.}$$

For experimentally used conditions $y < 0.1$ and $R(y) < 0.15$; then $\frac{d\eta}{dT} = -\frac{dR}{dT}$ is negligibly small.

APPENDIX 2

If the creep contributions of the two different laws (1.1) and (1.2) are denoted by $\Delta\epsilon_n$ and $\Delta\epsilon_\gamma$, then at $T_{21} [\sigma_c]$ the ratio of these two contributions $V \equiv \Delta\epsilon_n / \Delta\epsilon_\gamma$ equals unity:

$$(A3) \quad V(T_{21} [\sigma_c]) = 1.$$

The shift of the transition temperature in dependence of the applied stress can be found from

$$(A4) \quad \frac{\partial V}{\partial \sigma} \Big|_{T_{21}} d\sigma + \frac{\partial V}{\partial T} \Big|_{T_{21}} dT = 0.$$

This gives

$$(A5) \quad \frac{dT_{21}}{d\sigma} = - \frac{(\partial V / \partial \sigma) / T_{21}}{(\partial V / \partial T) / T_{21}} = \frac{kT_{21}^2}{\sigma} \frac{n - \frac{\gamma\sigma}{kT_{21}} \left(1 + \frac{kT_{21}}{U_0 - \gamma\sigma} \right)}{U_0 - Q + kT_{21} - \gamma\sigma}$$

or

$$(A6) \quad \sigma \frac{d(1/kT_{21})}{d\sigma} = - \frac{n - \frac{\gamma\sigma}{kT_{21}} \left(1 + \frac{kT_{21}}{U_0 - \gamma\sigma} \right)}{U_0 - Q + kT_{21} - \gamma\sigma}.$$

As a rough estimate, in many materials it will be

$$u_0 - Q \approx 1eV, \quad kT_{21} = \text{several } 10^{-2} eV;$$

There is a limiting situation $\sigma \rightarrow 0$, but this perhaps could not have been experimentally observed:

$$(A7) \quad \lim_{\sigma \rightarrow 0} \sigma \frac{d(1/kT_{21})}{d\sigma} = - \frac{n}{u_0 - Q + kT} < 0.$$

As far as a finite stress $\sigma > \frac{nkT}{\gamma}$ is concerned, the transition point changes with increasing stress to lower temperatures $T_{21}(\sigma)$ since then

$$(A8) \quad \sigma \frac{d(1/kT_{21})}{d\sigma} > 0.$$

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STRESZCZENIE

O ПЛЫНИЕЦУ СТЫМУЛОВАНЫМ ТЕРМИЧНИЕ I МЕХАНИЗМАХ АТОМОВЫХ
ЗАЛЕЖНЫХ ОД НАПРЯЖЕНИЯ

Celem pracy jest sformułowanie propozycji dotyczących badań doświadczalnych efektów płynięcia i pełzania określenie parametrów aktywacji. Rozważa się interpretację fizyczną wspomnianych procesów i towarzyszący im wzrost defektów sieci krystalicznej.

Резюме

О ТЕЧЕНИИ СТИМУЛИРОВАННОМ ТЕРМИЧЕСКИ И АТОМНЫХ МЕХАНИЗМАХ
ЗАВИСИЯЩИХ ОТ НАПРЯЖЕНИЯ

Целью работы является формулировка предположений, касающихся экспериментальных исследований эффектов течения и ползучести, определение параметров активации. Рассматривается физическая интерпретация упомянутых процессов и сопутствующий им рост дефектов кристаллической решетки.

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