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# RESEARCH TO DETERMINE THE LONG-TERM MECHANICAL PROPERTIES OF METALS SUBJECTED TO MECHANICAL STRESS AT ELEVATED TEMPERATURES AND NEUTRON IRRADIATION

(Annual Report 1965)

by



W. SIEGFRIED and S. ZIMERING
(Battele)

1966

EURATOM/US Agreement for Cooperation

EURAEC Report No. 1621 prepared by BMI
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## SUMMARY

Graphical methods have been devised for the three classes of mechanisms leading to long-time rupture : mechanism of deformation, growth of cracks, long-time changes in the material; these methods have been applied to the interpretation of time-to-rupture results and found to be also applicable in the case of neutron irradiated materials. Use of the exponential function of real order to improve the representation of the shape of time-to-rupture curves.

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# RESEARCH TO DETERMINE THE LONG-TERM MECHANICAL PROPERTIES OF METALS SUBJECTED TO MECHANICAL STRESS AT ELEVATED TEMPERATURES AND NEUTRON IRRADIATION

## 1. STATEMENT OF THE PROBLEM

To develop extrapolation methods for determining the influence of neutron irradiation on high temperature-creep properties of metals for the construction of nuclear reactors.

## 2. DESCRIPTION OF THE PRINCIPLE USED IN SOLVING THE PROBLEM

### 2.1. General considerations

The present problem stems from the field of the practical application of fundamental sciences to mechanical engineering. It differs, in its principle, from the problems of basic research. Fundamental

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sciences strive towards developing a logically uniform theory of phenomena. The result of an experiment is to promote general theory if possible. Thinking progresses in the atomic field, and attempts are made to attain first of all atomic knowledge of phenomena. For this purpose, one generally restrains oneself to pure metals and idealized conditions. This kind of consideration, however, lacks integral results which are needed by mechanical engineering and are regarded as trivial outcome of the general theory.

Quite opposite to this way of reasoning is the thinking of an engineer who has in mind practical applications. He is less interested in abstract thinking, in as much as he is concerned first of all with special cases. Thereby, observation is invariably made that fundamental sciences have not yet advanced as much towards solving practical problems by composition of elementary processes. As a rule, science is still quite incomplete. In this state of affairs, the engineer restricts himself to investigations that are purely phenomenological and answer only his special problems. This way of thinking is not satisfactory fundamentally, however, because numerous tests have to be carried out which, mainly as regards time-to-rupture under neutron irradiation, is no longer possible.

In the present state-of-the-art, the following procedure seems to improve the entire test technique. We try to obtain a synthesis of technological investigations and scientific theories. This implies a critical elaboration of results of scientific theories with the target of finding the possibility of simplifying working hypotheses where gaps still exist in scientific theory. On the basis of working hypotheses developed in this way, certain mathematical equations can be drawn up whose single parameters can be determined by means of technological tests. A more accurate analysis of simplifications made and of working hypotheses introduced makes it possible to draw certain conclusions about those physical processes which take place in practical creep tests.

We proceed in such a way that we develop our experimental method first by means of time-to-rupture tests without neutron irradiation. On the basis of the results obtained and the investigations already known concerning the influence of neutron irradiation on creep at high temperature, it is then possible to discuss the way in which extrapolation methods can be applied in the presence of neutron irradiation.

## 2. 2. Evaluation of the results of scientific theories

This evaluation showed that formation of rupture in long-time tests at high temperature is caused by three classes of mechanism, viz :

a) Mechanisms of deformation.
Various models for the movement of dislocations during the creep process fall among these mechanisms. They are characterized by the fact that, in the case where they exist singly, formation of rupture would take place by a constriction with 100 % local elongation of rupture. Furthermore, it can be assumed that the energy of activation of these mechanisms is equal,in first approximation to the energy of activation for the self-diffusion.

b) Growth of fissures.
These mechanisms are characterized by the fact, in cases where they alone are effective, that they lead to a rupture without plastic deformation and elongation of rupture. Until now solid-state physics indicated only one mechanism for long-time rupture tests, namely that given by Cottrell, in which the formation of fissures takes place by stress-directed diffusion of atoms. This mechanism shows an energy of activation which again can be compared to that for the self- diffusion.

c) Long time transformations of material.
Most of the materials technically applied are in a metastable state which changes to the thermodynamically stable state with long periods of time and elevated temperature. First of all, precipitations with overageing and furthermore tempering operations are eligible for creep tests. These mechanisms are characterized by the fact that they develop very slowly over long periods and show an energy of activation which differs essentially from that for the self-diffusion.

On the basis of the rheological and thermodynamical differences of the various classes of mechanism mentioned, it is now possible to separate these by evaluating results of time-to-rupture tests at different temperatures.

## 2. 3. Evaluation of practical time-to-rupture tests

Practical time-to-rupture tests have been carried out, namely in abundance on steel, with the attempt to obtain bases of construction for the building of steam and gas turbines. Duration of tests was frequently extended to more than 10 years. For the evaluation, the procedure was that stress in dependence of time-to-rupture was represented in a double logarithmic diagram and test values practically determined were combined by arbitrary series of curves. The shape of the curve obtained by such a procedure is determined by the number of test values and by the personal concepts of the evaluating scientist. In this state of affairs, practice is in a position to give only a relatively broad scatter band (factor 100 for time to rupture ). The determination of such a scatter band needs an extremely large number of tests and makes it imperative to decrease the admissible stresses for important elements of construction in which premature rupture would lead to considerable damage. Very expensive materials are partially employed in nuclear power plants. On the other hand, because of the parasitic absorption of neutrons, one strives at keeping the weight of the material very low; furthermore, time-to-rupture tests at high temperatures under neutron irradiation are so expensive that only a small number of values can be determined. The determination of the scatter band cannot therefore be carried out reliably a priori.

To improve on this state of affairs, a new mathematical function was used which determines the shape of time-to-rupture curves by means of a few parameters more accurately than all functions known so far. This is the exponential function of real order which has been studied in the course of the past few years. By means of this function the problem of scattering and the evaluation of practical time-to-rupture tests can be put on a new basis.

### 2.4. Combination of the kinds of investigation mentioned above

On the basis of the elaboration of working hypotheses concerning the developing creep processes which agree with the results of theoretical investigations on the creep process, and moreover of the improved mathematical determination of phenomenological test results, it is possible to throw a bridge between theory and practical application in this field. This synthesis between physical investigations and technologically determined mechanical properties results in a whole series of important consequences :

a) One is in a position to explain physically, to a certain degree, those phenomena which are technologically observed. Mainly, the influence of heat and ageing treatments on the shape of the time-to-rupture curve can be estimated to some extent.

b) It is possible to refine essentially the existing temperature-extrapolation procedures and also to predetermine the fields of embrittlement by tests of shorter duration and at elevated temperature.

c) It is to be expected that in a given material it can be determined, by means of tests of relatively short duration, whether the ultimate stress values after long times are located at the upper or lower border of the scatter band.

d) Development of new heat-resistant alloys and determination of optimum heat treatment can be carried out with an importantly smaller number of tests than was the case until now.

The synthesis indicated here, i. e. of the results of solid-state physics and practical technological investigations, will be fully successful when the physical meaning of the parameters of the exponential function of real order can be given which are applied for the mathematical determination of the technological properties.

## 2. 5. Time-to-rupture tests under neutron irradiation

Once we succeeded in separating the creep process and the long-time rupture process during creep in three classes of mechanism, and in determining the parameters for these processes by evaluation of isothermal time-to-rupture tests, it became possible to verify, on the basis of available investigations of the influence of neutron irradiation on creep processes, up to what extent neutron irradiation affects the different classes of mechanism. It has been shown that an extension of methods of determining the various classes of mechanism without neutron irradiation on creep tests under neutron irradiation is readily possible. We are therefore in a position to determine in a simple way, by evaluating time-to-rupture tests wiht and without neutron irradiation, up to what extent neutron irradiation has a direct effect on the following classes of mechanism :

a) Mechanisms of deformation (mechanisms of dislocation).

b) Extension of fissures (mean number of fissure germs).

c) Structural changes.

Obviously, determination of the influence of neutron irradiation on the three classes of mechanism mentioned above considerably reduces the number of investigations necessary for the determination of the technological properties of metals under neutron irradiation. It has also been shown that a temperature extrapolation of long-time ultimat stress values under neutron irradiation is possible in principle.

# 3. DETAILED DESCRIPTION OF WORK CARRIED OUT IN 1965

## 3. 1. Development of methods for the separation of the mechanisms of deformation and rupture

### 3. 1. 1. Specification of working hypotheses

The following working hypotheses have been elaborated on the basis of an evaluation of results of solid-state physics concerning the creep process at high temperature.

Rupture process after long times is based on three classes of mechanism, viz :

a) Mechanisms of deformation.
These mechanisms show the following rheological and thermodynamic properties : Formation of rupture results from the formation of a constriction, and rupture is a pure rupture of deformation. The energy of activation for these processes can be regarded, in first approximation, as being equal to that for the self-diffusion.

b) Extension of fissures.
In case rupture takes place according to this mechanism, we have a deformationless rupture without elongation. The process of extension of a fissure shows an energy of activation which is equal to that for the self-diffusion.

c) Influence of time.
Usually, a change of the test material with time in time-to-rupture tests of long duration is observed. Since, however, this process takes place very slowly, we can determine this influence by understanding the parameters of our mathematical equations as mean values that are dependent only on time to rupture.

### 3.1.2. Mathematical definition of working hypotheses and combination of both processes

a) Processes of deformation.

To determine the processes of deformation, the following equation was used

$$v = v_o(\sigma) \sinh \left\{ \frac{\sigma}{\sigma_1 (\sigma)} \right\} \qquad (1)$$

$v$ = Creep rate

$v_o(\sigma)$ = Function of stress or time to rupture

$\sigma_1(\sigma)$ = Function of stress or time to rupture.

By means of this equation we can practically determine the rheological behaviour of any theoretical model concerning mechanisms of dislocation during the creep process. Two special cases are of particular importance :

(i) $v_o(\sigma) = \text{const} = v_o$

and

$\sigma_1(\sigma) = \text{const} = \sigma_1$

In this case a creep law according to a hyperbolic sine is obtained.

$$v = v_o \sinh \left( \frac{\sigma}{\sigma_1} \right) \qquad (2)$$

Most of the models concerning climbing of jogs in screw dislocations lead to such a creep law.

(ii)

$$v_o(\sigma) = A\ \sigma^n$$

$$\sigma_1(\sigma) = \text{const}\ .\ \sigma\ .$$

$$v = \text{const}\ .\ \sigma^n \qquad (3)$$

As a rule, this creep law is observed at low stresses and corresponds for instance to the climbing of jogs in edge dislocations.

Mechanisms of the extension of fissures.

For this case, we restrict ourselves to the model given by Cottrell, which leads to the following mathematical formula :

$$\frac{dr}{dt} = \frac{3\,\Omega\,\sigma\,D\delta}{a\,r\,k\,T} \qquad (4)$$

r = radius of a circular fissure

$\sigma$ = stress

a = mean distance between two fissures

$\Omega$ = volume of an atom

D = diffusion coefficient

$\delta$ = thickness of grain boundary.

This model is valid for the formation of fissures for time-to-rupture tests of a certain duration. For very short time to rupture (fractions of hours), other models of formation of rupture must still be considered.

Combination of formulas for deformation and extension of fissures.

To unite the two processes for the formation of rupture mentioned above, the differential equation for the formation of a constriction was drawn up by means of equation (1) and was then combined with equation (4) by considering in equation (4) the growth of stress resulting from the formation of a constriction. Thus, the growth of stress as a result of the extension of fissures was considered in the differential equation for the formation of a constriction. In this way, two simultaneous differential equations are obtained which, however, can be integrated with certain simplifications. In proceeding in this way it is assumed that the process of deformation influences the number of developing fissure germs.

### 3. 1. 3. Determination of the parameters for the different classes of mechanism by analyses of isothermal time-to-rupture tests

A graphic method was developed which permits simple determination of the following values :

a) $v_o$ in dependence of time to rupture or stress
b) $\sigma_1$ in dependence of time to rupture or stress
c) Mean number of fissure germs in dependence of time to rupture or stress.

Furthermore, graphic representations have been given which allow conclusions to be drawn on the creep law resulting from the shape of these values in dependence of stress or time to rupture.

### 3. 1. 4. Verification of theoretical investigations by means of results of long time-to-rupture tests

Numerous isothermal time-to-rupture tests, extended up to about 100 000 hours, have been investigated. It became obvious that essentially there are only two creep laws available, namely, at high stresses, that according to a hyperbolic sine and at low stresses an exponential law. This verification agrees with the research of solid-state physics, and we can therefore regard this result as a confirmation of the practicability of our working hypotheses.

3.1.5. Application to time-to-rupture tests with notched specimens

In a time-to-rupture test with a notched specimen, the mechanical behaviour originates in a very small area in the ground of the notch. It has not yet been possible to obtain more accurate information about the interaction of different factors which take place rheologically in the notched specimen, for a precision-elongation-measurement at high temperature in the ground of the notch is impossible. Since, for our analysis, we need only the shape of the time-to-rupture curve, our considerations are also applicable to the investigation of time-to-rupture tests on notched specimens, in case the behaviour of the metal in the ground of the notch is determining solely for the formation of rupture. We have made this evaluation on a large number of time-to-rupture tests with noteched specimens, and we have been able to draw some interesting conclusions about the embrittlement.

3.1.6. Fundamental possibility of partial replacement of time-to-rupture tests by determination of hot hardness

The results of our investigations have put us in a position to determine up to what extent time-to-rupture tests can be replaced partially by determination of hot hardnes s. Bases for this possibility have been roughly investigated.

## 3. 2. Separation of mechanisms of deformation and formation of fissures from those of long-time structural changes

### 3. 2. 1. Development of working hypotheses

As already mentioned, structural changes are ditinguished from mechanisms of deformation and formation of fissures by the fact that the former show an energy of activation that in the first approximation can be compared to the energy of activation for the self-diffusion, whereas the latter show an energy of activation that is given first of all by the chemical potential of structural change. This chemical potential, however, is given by the equilibrium lines in the phase diagram and by the amount of undercooling. If ageing and heat-treatments are modified, this part in the energy of activation will be changed above all. We can therefore draw conclusions on the structural changes from the dependence on temperature of values $v_o$, $\sigma_1$ and a determined in section 3. 1. 3. This is only possible, however, if the general structure of the equations is known by means of which the influence of structural changes on the mechanisms of deformation is determined. To determine this structure, we proceed as follows:

It was possible to calculate these ratios for an important special case, namely for precipitation and growth of a secondary phase from solid solution in combination with climbing of screw dislocations across these precipitations. The following equations have been obtained for the values $v_o$ and $\sigma_1$ :

$$\log v_o + \frac{\Delta H_1}{2,3\ RT} = \varphi_2 \left\{ \frac{\Delta H_3}{2,3\ RT} - \log t_B \right\} \qquad (5)$$

$$\log \sigma_1 - \frac{\Delta H_4}{2,3\ RT} = \varphi_4 \left\{ \frac{\Delta H_5}{2,3\ RT} - \log t_B \right\} \qquad (6)$$

$\Delta H_1$, $\Delta H_3$, $\Delta H_4$, $\Delta H_5$ = heat of activation

$\Delta H_1$ and $\Delta H_4$ strongly dependent on the chemical potential

$\Delta H_3$ and $\Delta H_5$ not very different from heat of activation for self-diffusion.

A simple reflection shows in case other structural changes and creep mechanisms do exist that in principle equations with the same structure are obtained.

Graphic methods have then been developed which permit the calculation of values $\Delta H_1$, $\Delta H_3$, $\Delta H_4$, and $\Delta H_5$ by means of the dependence on time of values $v_o$ and $\sigma_1$.

### 3. 2. 2. Verification of results

Results have been checked on time-to-rupture tests of four steels, viz. : a CrMoV-steel, the alloy S 816, the alloy S 590, and an austenitic CrNi-steel, type 304. This verification also proved the correctness of equations (5) and (6). The energy of activation for the precipitation, calculated from the values for the various heats of activation, shows plausible values. Moreover, the calculated influence of heat treatment on structural changes corresponds exactly to the result that was to be expected on the basis of practical experience.

### 3. 3. Description of the rate process by an exponential function of real order

During the reporting year, numerous time-to-rupture tests have been analyzed by means of the exponential function of real order and the variation of parameters has been determined as a function of temperature and stress.

Moreover, the theoretical bases for an objective determination of scattering of time-to-rupture tests have been created and explained in greater detail in different examples. Finally, it might soon be possible for us to determine the relationship between the parameters of the exponential function of real order and those of the mechanisms of deformation and structural changes.

## 4. POSITION WITH RESPECT TO NEUTRON IRRADIATION

A former investigation, using test results of creep tests under the influence of neutron irradiation, showed the result, presupposing stationary conditions, that at least the decomposition described above into the different classes of mechanism is still valid in principle. Equations are somewhat modified by determining the mechanisms of structural changes, for we have longer pure rate-processes, and a linear term must still be added to these equations. However, it is possible in principle to indicate explicitly the mathematical equation for these ratios.

On the basis of these results, the following conclusions can be drawn :

By comparison of time-to-rupture tests with and without neutron irradiation at different temperatures, the effect of neutron irradiation on the following classes of mechanism can be determined separately :

a) Mechanisms of deformation (kind of process of dislocation)
b) Formation of fissure germs
c) Effect on structural changes.

## PUBLICATIONS DURING 1965

1) W. SIEGFRIED : On the theory of notch-brittleness in steels occuring during time-to-rupture tests. EURAEC Report No. 1350.

2) W. SIEGFRIED : Determination of the elementary mechanisms of the rupture process at creep by evaluation of time-to-rupture tests. "Symposium sur le comportement à chaud des alliages métalliques", Torino, 29th September, 1965.

3) W. SIEGFRIED : Nouvelle approche pour l'étude de phénomènes de fragilisation au cours des essais de fluage. "Journées d'Automne de la Société Française de Métallurgie", Paris, 11th - 15th October, 1965.

4) S. ZIMERING : Une nouvelle méthode d'extrapolation du temps de rupture d'un alliage industriel. Les Mémoires Scientifiques de la Revue de Métallurgie, LXII, No 12, 1965, pp. 939-950.

5) W. SIEGFRIED : Untersuchungen über den Zusammenhang zwischen der Form der Zeitbruchlinie warmfester Stähle und den verschiedenen Mechanismen des Kriechvorganges. Archiv für das Eisenhüttenwesen, Jg. 37, H. 1, 1966, pp. 65-78.

6) S. ZIMERING : La fonction exponentielle d'ordre réel et son application pour la description du procédé de fluage. Will be published in : Zeitschrift für Angewandte Mathematik und Physik, Basel, 1966.

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Alfred Nobel

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