JOURNAL OF MATERIALS SCIENCE 9 (1974) 1045–1049

The effect of the crystal structure on the creep of cobalt at moderate temperatures

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The creep of polycrystalline cobalt has been studied at temperatures in two ranges: 20 to 300° C for α-cobalt and 500 to 575° C for β-cobalt. Analysis of the temperature- and stress-dependence of the steady state creep rate gave different values for the activation energy and activation volume in the two temperature ranges.

1. Introduction

According to the investigation by Zhurkov and co-workers [1], the dependences of the rate of creep $\dot{\epsilon}$ and the life time τ on stress σ and temperature T for a great number of materials at temperatures less than 0.5 T_m (T_m is the absolute melting temperature) are described by the relations

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp\,[-\,(v_0 - \gamma\sigma)/kT] \tag{1}$$

$$\tau = \tau_0 \exp\,[(v_0 - \gamma\sigma)/kT] \tag{2}$$

where $\dot{\epsilon}_0$, τ_0, v_0, and γ are constants of the material. It is found that at moderate temperatures the activation energy, v_0, approximately coincides with the sublimation energy, and τ_0 coincides with the period of vibrations of atoms in a crystal lattice. The activation volume γ is the only parameter in Equations 1 and 2 reflecting the effect of structural variables on the creep of materials.

The question concerning the nature of the activation v_0 is not yet settled. The works by Dorn and the co-workers [2] are widely known and according to these the activation energy of creep is close to the activation energy of self diffusion of vacancies. In [3] it has been shown that the parameter v_0 agrees with the self diffusion energy through the interstitials. Recently several models have been proffered wherein creep is associated with one or other rearrangement of the dislocation structure: for instance, with jog migration on screw dislocations [4], with dislocation freeing from the subgrain boundaries [5] etc. Here, the parameter v_0 will define the height of the energy barrier determining the rate of the said dislocation process.

From this viewpoint it seemed expedient to study the effect of the polymorphic transformation on the activation energy of creep of cobalt whose two modifications are rather similar in their crystallochemical properties. In cobalt a transformation occurs at a moderate temperature (about 450°C) without changing the co-ordination number and scarcely changing the interatomic spacing. Hence, the binding forces between the atoms in the different modifications and the corresponding sublimation energies are expected to be similar. Then, in accordance with [1] one should suppose that the parameter v_0 will not change at the $\alpha \leftrightarrow \beta$ transition. Verification of this supposition is the main goal of the present work.

Creep in cobalt has been studied in earlier works [6, 7]. However, Feltham and Myers, and Kamel and Halim [6] carried out the investigation at temperatures above the transformation temperature and their results do not even qualitatively agree with ours, and with the results of common knowledge for such metals. Therefore, it was not possible to employ the results obtained in [6] for discussing the question of interest. In [7] the investigation was carried out in the temperature range of 300 to 500°C. Below it will be seen that in this temperature range Equations 1 and 2 are not fulfilled; therefore, the results obtained in [7] cannot be used for the discussion.

2. Experimental methods

K-1A polycrystalline cobalt of 99.31% purity was used. Creep test specimens prepared from forged rods, were vacuum annealed at 850°C for 1 h in order to relieve the internal stresses and to stabilize the structural state. This annealing defined the grain size in the specimens which did not change upon subsequent annealings (α-Co) and tests (α-Co and β-Co). The grain size was about 40 μm. Several specimens intended for low temperature tests in the region of α-Co (HCP) were then subjected to two-stage annealing: at 300°C for 50 h and at 380°C for 100 h. Such a thermal treatment was needed for fuller $\beta \rightarrow \alpha$ transformation and for obtaining of the single/phase state [8]. An X-ray study using the technique described in [9] showed that after the said two-stage treatment the quantity of the high temperature phase did not exceed 5%.

Creep tests were carried out at constant stress. The gauge length of the specimens was 20 mm and the diameter 2.5 mm. The tests were carried out at temperatures of 20, 150, 200 and 300°C for a α-cobalt and at temperatures of 500, 550 and 575°C for β-cobalt. Using electrical resistivity and dilatometer techniques it was established that for the cobalt used in this work, the phase transformation occurred within the temperature range 360 to 480°C. The temperature range of 300 to 500°C was, therefore, excluded from the creep tests. Tests were not carried out above 575°C since above this temperature oxidation occurs, and also it has been shown that Equations 1 and 2 are only valid at $T < 0.5\, T_m < 610°\text{C}$.

In the low temperature tests, as a rule, the specimens were not brought up to fracture and, therefore, only the rate of their creep was determined. For the β-cobalt specimens the creep curve was recorded until fracture ensued.

The dislocation structure of the specimens was investigated on a JEM-150 electron microscope. The foils for transmission were prepared by the technique described in [10].

3. Experimental results

The tests showed that the creep curves of α- and β-cobalt have, as do the majority of pure metals, the unsteady, steady-state and accelerated stages. Fig. 1 represents the results of the investigation of creep in semi-logarithmic co-ordinates. For both modifications of cobalt, the dependences ($\log \dot{\epsilon}$) versus σ are seen to be linear and can be described by Equation 1.

For the α-cobalt, the straight lines corresponding to various temperatures converge to the pole with the co-ordinates

$$\sigma_0^\alpha = 143 \text{ kgf mm}^{-2}, \log \dot{\epsilon}_0^\alpha = 10.5 .$$

The dependence of the activation volume, γ, determined by the slope of the straight lines ($\log \dot{\epsilon}$) versus σ, on temperature, is represented in Fig. 2. γ is seen to increase sharply at temperatures approaching 300°C, closest to the transformation interval; for lower temperatures γ is practically constant and equals $\gamma^\alpha = 4.42 \times 10^{-22}$ cm^3. From the relation $v_0 = \gamma\sigma_0$, the corresponding value of the activation energy $v_0^\alpha = 89$ kcal mol^{-1} can be obtained. The same value was also obtained by means of plotting the dependences $\log \dot{\epsilon}$ versus T^{-1} for various fixed σ by using the value of $\log \dot{\epsilon}$ obtained by extrapolation of the straight lines in Fig. 1 (see also Fig. 3). This also confirms that the points for 300°C fall outside the general trend.

Consider the results obtained for the β-cobalt. The activation volume proportional to the slope of the straight lines ($\log \dot{\epsilon}$) versus σ is determined fairly accurately. The corresponding values are represented in Fig. 2. The point for 500°C closest to the interval of transformation, also falls out here. The activation volume of the β-cobalt can, according to Fig. 2, be assumed to equal $\gamma^\beta = 2.90 \times 10^{-22}$ cm^3, this value is by 34% lower than that for the α-cobalt. For the co-ordinates of the pole of the straight lines of the β-cobalt in Fig. 1, treatment by the least-squares technique considering the errors both on the X-axis and the Y-axis [11], gave the following values: $\sigma_0^\beta = 176$ kgf mm^{-2}, $\log \dot{\epsilon}_0^\beta = 10.8$. The corresponding value of the activation energy $v_0^\beta = 72$ kcal mol^{-1} which is less by 19% than for the α-cobalt.

Fig. 3 illustrates the linear decrease in the activation energy v with increase in stress for the steady state creep for the α and β-cobalt. The values of the activation energy were calculated using the relation $v = 2.3\, kT. \log (\dot{\epsilon}_0/\dot{\epsilon})$ with the above values of $\log \dot{\epsilon}_0^\alpha$ and $\log \dot{\epsilon}_0^\beta$, and, also

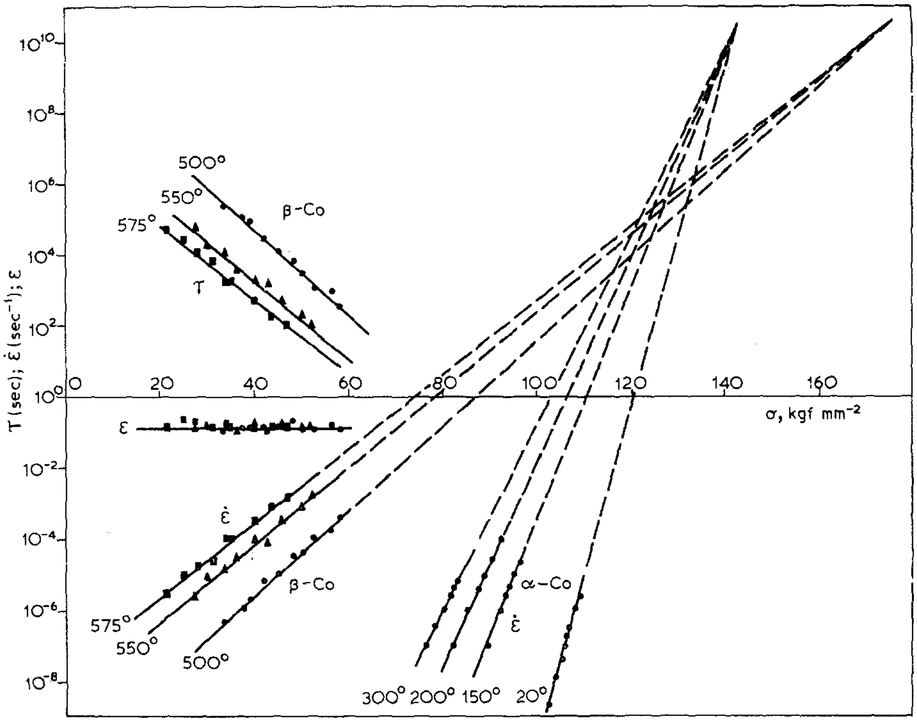

Figure 1 Life time τ, the rate of the steady state creep $\dot{\epsilon}$, and the steady state creep strain ϵ of cobalt crystals as a function of stress σ at different temperatures.

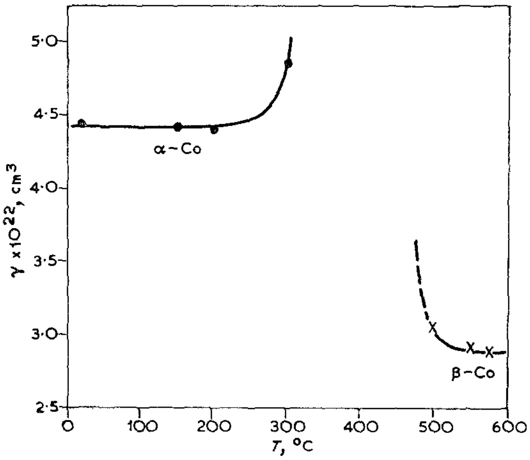

Figure 2 The activation volume γ versus temperature dependence.

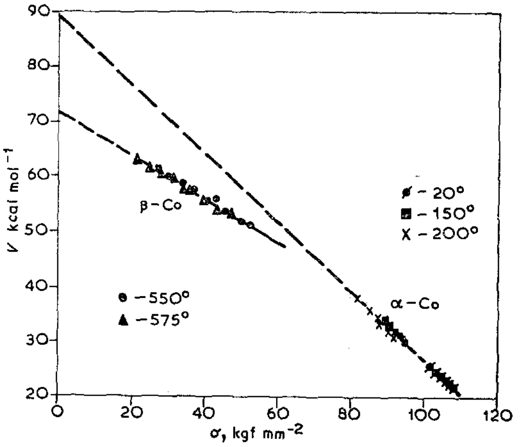

Figure 3 The activation energy, v, for the steady state creep of α and β-cobalt as a function of stress, σ.

using the temperature dependence of log $\dot{\epsilon}$. Note that the obtained values of σ_0^β, log $\dot{\epsilon}_0^\beta$ and v_0^β cannot be considered established with great accuracy (in contrast to the activation volume γ^β). This point will be discussed in the next section.

An electron microscope observation showed that in the initial state the α-cobalt was of a large grain structure with low dislocation density. Sometimes twins were found inside the grains and in local regions stacking faults were observed. The dislocation structure during primary creep was characterized by uniformly distributed dislocations of high density. During steady-state creep, a subgrain structure with tangled boundaries of high dislocation density and low dislocation density in the subgrain interiors were strongly pronounced which is in good agreement with the results obtained on other materials [5] whose steady state creep is subject to Equation 1. It can be assumed, therefore, that the processes regulating the rate of the steady state creep are similar for cobalt and for the metals investigated in other works [1-5]. Unfortunately, it does not seem possible to carry out an investigation of the dislocation structure of the β-cobalt in the process of creep.

4. Discussion and conclusions

From the results obtained, it follows that at temperatures below 300° C, creep in α-cobalt is subject to Equation 1. The presence of the plateau in the plot of $\gamma^{\beta}(T)$ for the temperatures of 550 to 575° C (Fig. 2) and the results obtained upon determining the co-ordinates of the pole being in agreement with graphical estimation of its location (Fig. 1), give one grounds to believe that in a certain temperature range ($\sim$550° C) Equation 1 is also valid for the β-cobalt. Constancy of the steady state creep deformation $\epsilon = \dot{\epsilon}\tau$ suggests that Equation 2 is also valid.

In accordance with the ideas expressed in [1] a comparison should be made between the activation energy of creep, v_0, and the available data on the sublimation energy. Two preliminary comments need to be made. Firstly, it is known from thermodynamics that the heat of sublimation is temperature dependent since thermal capacities of the condensed and vapour phases change with temperature. Therefore, in order to make the correct comparison between the activation energy of creep and the sublimation energy, the latter should be adjusted to the temperature range in which the creep tests were carried out. Note that the sublimation energy is determined at temperatures of 1000 to 1300° C which are markedly higher than those of the creep tests. It is known, however, that the temperature correction is insignificant and can be ignored [12]. Secondly, since the phase transformation in cobalt has a very small energy of transformation (0.1 kcal mol^{-1} [13]), as compared with the heats of melting and evaporation (3, 6 and 89 kcal mol^{-1}, respectively [12]), the expected difference in the heats of sublimation of the α-cobalt and the β-cobalt can amount to only $\sim$0.1 %.

The available data on the heat of sublimation in cobalt are of wide spread (from 82.7 to 101.7 kcal mol^{-1} [12, 14, 15]). The most reliable results are those obtained in [9], where it was found that the sublimation energy of cobalt was 101.6 kcal mol^{-1}. The values of the steady state creep activation energy of the α- and β-cobalt turned out lower than this and, furthermore, there was a noticeable difference in the activation energies for creep of the α- and β-cobalt.

It must be noted that the pole position for the β-cobalt in Fig. 1 is found as the point of intersection of the straight lines converging at a small angle. Though the co-ordinates of the pole are found by the least-squares technique and, in this sense are objective, it is quite possible that further investigations might suggest a shift in this pole position and, therefore, in the activation energy value v_0^{β}. Moreover, the least-squares technique itself can, in the case under consideration, result in a marked error. Therefore, it should be emphasized again that the co-ordinates of the pole σ_0^{β}, log $\dot{\epsilon}_0^{\beta}$ and, hence, the numerical value of the activation energy v_0^{β} for the β-cobalt cannot be considered precisely established. Plotting of the dependences log $\dot{\epsilon}$ versus T^{-1} corresponding to the fixed stresses in order to find the parameter v_0 only on the basis of the straight lines (log $\dot{\epsilon}$) versus σ for 550 and 575° C does not permit an accurate determination of the activation energy, v_0^{β}, since on these dependences we shall have only two by two closely located points.

If one assumes that $v_0^{\beta} \approx v_0^{\alpha} = 89$ kcal mol^{-1}, then the pole position in Fig. 1 for the β-cobalt must be different. Let us assume that the pole displaces along the straight line log $\dot{\epsilon}$ versus σ corresponding to the temperature of 550° C (it can easily be seen that the choice of the other straight lines will not change the above considerations). Since such a displacement insignificantly changes the slope of the straight lines, the activation volume remains practically the same, and for all the varied pole positions one can assume $\gamma_{\beta} = 2.90 \times 10^{-22}$ cm^3. At $\sigma_0^{\beta} = 218$ kgf mm^{-2} (and, correspondingly log $\dot{\epsilon}_0^{\beta} = 15.2$) we would have $v_0^{\beta} = v_0^{\alpha} = 89$ kcal mol^{-1}. Then, assuming that log $\dot{\epsilon}^{\beta} = 0.85$ (see Fig. 1) we obtain for this case log $\tau_0^{\beta} =$ log $\epsilon^{\beta}/\log \dot{\epsilon}_0^{\beta} = -16$. At the same time, assuming $\epsilon^{\alpha} \approx \epsilon^{\beta}$, we obtain log $\tau_0^{\alpha} \approx -$ (11 to 12) for α-cobalt. Therefore if during the $\alpha \leftrightarrow \beta$ transformation the activation energy of creep is considered unchanged, while the activation volume and the pre-exponential term $\dot{\epsilon}_0$ change, it should be admitted that the parameter τ_0 decreases by 4 to 5 orders. In terms of the conceptions in [1], based on Equations 1 and 2, this decrease in the parameter τ_0 is impossible to understand since by this conception, the times of the order of 10^{-16} sec, less than the period of atom vibrations in the materials, are of no physical sense.

Despite this alternative, the following result is unquestionable: the straight lines log log $\dot{\epsilon}$ versus σ in Fig. 1 for the α-cobalt and the β-cobalt cannot be definitely attributed to the same line. This means that the whole totality of the straight lines by Equations 1 and 2 with the analogous parameters v_0 and $\dot{\epsilon}_0$ (or τ_0) for the two modifications is impossible to describe. Hence, the above alternative arises: if the equality $v_0^{\alpha} \approx v_0^{\beta}$ is admitted, then admissible (accordding to [1]) change in τ_0 at the α-β transition is obtained; the contrary assumption also results in contradiction with the suggestions in [1]. This conclusion is valid for the intermediate cases too. Evidently a change in the activation volume during the α-β transformation, can be related to difference in geometry of dislocation glide in hcp and fcc crystals.

The Dorn relation [2]

$$\dot{\epsilon} = A \exp\left(\beta\sigma - \frac{\Delta H}{KT}\right) \qquad (3)$$

(A, β are the constants, ΔH is the activation enthalpy) is not valid for describing the behaviour of the β-cobalt, since it gives the system of parallel straight lines log $\dot{\epsilon}$ versus σ. For the β-cobalt in the temperature range under consideration, non-parallelity of these straight lines is obvious and, therefore, one can assert that Equation 1 is in better agreement with the experimental data than Equation 3. However, Dorn and co-workers have asserted that Equation 3 is valid at temperatures $T > 0.5\, T_m$ whereas, in accordance with our intentions such experiments were not carried out in our investigation.

Acknowledgements

The authors express deep gratitude to V. L. Indenbom on whose suggestion, partially, the present experiments have been performed and for his continuous helpful advice and discussions. They are also grateful to M. N. Peregudov who carried out the X-ray investigation referred to in Section 3.

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Received 12 February and accepted 27 November 1973.