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Compressive Creep of Fe₃Al-type Iron Aluminide with Zr Additions

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High-temperature creep of a Fe_3Al -type iron aluminide alloyed by zirconium was studied in the temperature range 873–1073 K. The alloy contained (wt.%) 31.5% Al, 3.5% Cr, 0.25% Zr, 0.19% C (Fe balance). It was tested in two states: (i) as received after hot rolling and (ii) heat treated (1423 K/ 2 h/air). Creep tests were performed in compression at constant load with stepwise loading: in each step, the load was changed to a new value after steady state creep rate had been established. Stress exponent and activation energy of the creep rate were determined and possible creep mechanisms were discussed in terms of the threshold stress concept. A rapid fall of the stress exponent and of the threshold stress with the increasing temperature indicates that creep is impeded by the presence of precipitates only at temperature 873 K. The results were compared with the results of long-term creep tests in tension performed recently on the same alloy.

Keywords: iron aluminides, creep, threshold stress.

Introduction. Iron-aluminides-based alloys are promising candidates for many industrial applications since they have excellent resistance to oxidation and sulfidation. One of their drawbacks is the insufficient high-temperature strength. This can be improved either through solid solution hardening or through precipitation hardening. The alloying by additions of zirconium is expected to be effective in the precipitation hardening due to low solubility and formation of phases (Fe, Al)₂Zr and (Fe, Al)₁₂Zr [1, 2]. This is in agreement with the review of existing studies of high temperature mechanical properties of iron aluminides [3, 4], which documented that the addition of Zr brings the best results. This fact initiated an investigation of quaternary alloy on Fe₃Al base with chromium and zirconium. The results of microstructural observations and of tensile creep tests at 873 K were published elsewhere [5]. The aim of the present paper is to report additional results of compressive creep tests of the same alloy performed in more extensive range of temperatures and to start discussion of the potential rate-controlling mechanisms.

Experimental. The composition of the alloy used for the experiment was as follows (wt.%): Al = 31.5; Cr = 3.5; Zr = 0.25; C = 0.19; Fe – balance. The alloy was prepared in the vacuum furnace and cast under argon in the Research Institute for Metals in Panenskä Brehany, Czech Republic. The casting (dimensions $400 \times 120 \times 38$ mm) was hot-rolled to the final thickness of 13 mm at 1473 K in several steps with 20% reductions for each pass. The rolled piece was heated after each second pass and the temperature did not decrease under 1273 K during the total rolling period. After the final pass, the slab was quenched from the temperature at least 1273 K into oil. One set of samples was additionally annealed at 1423 K/2 h and air cooled.

The specimens for compressive creep tests were prepared with the axis perpendicular to the rolling plane. The dimensions of samples were: diameter 4 mm, height 12 mm. Constant load compressive creep tests of the alloy were performed at temperatures from 873 to 1073 K. A stepwise loading was used: in each step, the load was changed to a new value after steady-state creep rate had been established. The terminal values of the true stress and the true strain rate were evaluated for the respective step.

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Protective atmosphere of dried and purified argon was used. During the test, temperature was kept constant within ± 1 K. Creep curves were PC recorded by means of special software. The sensitivity of elongation measurements was better than 10^{-5} .

Results. The applied stress dependence of the minimum creep rate in the rolled material is given in Fig. 1. The dependence can be described at a given temperature by the power function

$$\varepsilon = A\sigma^n, \tag{1}$$

where A is a temperature dependent factor and n is exponent. The values of exponent n are decreasing with increasing temperature: n = 12.2 at 873 K, 5.4 at 923 K, 4.9 at 973 K, 4.7 at 1023 K, and 4.6 at 1073 K. The apparent activation energy of creep can be obtained from the Arrhenius-type plot (Fig. 2). It is about 433 kJ/mol at 50 MPa, 407 kJ/mol at 80 MPa, and 375 kJ/mol at 100 MPa at temperatures from 923 to 1073 K, respectively, but it can be very high (greater than 700 kJ/mol) at lower temperatures.



Fig. 1. Applied stress dependence of creep rate at different temperatures. Fig. 2. Dependence of creep rate on reciprocal absolute temperature.

An example of creep results obtained on samples after heat treatment is given in Fig. 3. The heat treatment has positive effect on the creep resistance. The observed deceleration of the creep rate is less than one order of magnitude. The results of previous research of creep of the same alloy are also given in Fig. 3. A good coincidence of tensile and compressive creep data can be admitted.

Discussion. The values of stress exponent n in Eq. (1) are usually taken as an indication of potential creep controlling mechanisms. When dislocation motion controls creep deformation of pure metals and single phase solid solutions, the exponent n of about 3 to 5 is expected. The values of n can be substantially greater in alloys reinforced with particles of secondary phases. The creep behavior is then rationalized by means of the threshold stress concept: the stress dependence of the creep rate is rewritten as

$$\tilde{\varepsilon} = A' (\sigma - \sigma_{th})^{n'}, \qquad (2)$$

where σ_{th} is the threshold stress. The value of exponent n' should be close to the value of n observed in pure metals and single phase solid solutions. The value of the threshold stress can be determined in two different ways:

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(i) The method based on the additivity rule [6]. It is necessary to know the behaviour of corresponding single phase material.

(ii) The second method makes use of linearized plot of $(\dot{\epsilon})^{1/n^*}$ vs. applied stress. The method is sensitive to the choice of stress exponent n'.



Fig. 3. Comparison of results of creep tests in tension and in compression. Fig. 4. Dependence of threshold stress on temperature.



Fig. 5. Stress dependence of creep rate in present alloy and in two similar alloys from [8].

To enable a comparison with the previous application of the method to the results of creep in Fe–Al alloys – and also the alloy with Zr addition from [8], we have used the second method with the same value of n', i.e., n' = 4. The results are given in Fig. 4. Very good agreement is obtained at temperature 873 K. The values of the threshold stress in the present alloy are very rapidly decreasing with the increasing temperature and at temperature 923 K and above they are substantially lower than the values reported in [8]. This fact, together with the above given values of stress exponent n, indicates that creep is impeded by the presence of precipitates only at temperature 873 K. At temperatures greater than 923 K it could be expected that either the precipitates have only minor influence on the creep resistance of the investigated alloy or that they are dissolved. This can be further documented by comparing present data with the data published in [8] for

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temperature 973 K (Fig. 5). The slope of the stress dependence of present alloy is lower than that of the alloy Fe–20Al–Cr–Zr. Creep rates at low applied stresses are substantially slower in Fe–20Al–Cr–Zr than in the present alloy. On the other hand, creep properties of the present alloy are comparable with the properties of the binary alloy Fe–30Al without second-phase precipitation.

CONCLUSIONS

1. The uniaxial compressive tests of Fe–31.5Al–3.5Cr alloy with 0.25 wt.% of Zr at temperatures from 873 to 1023 K give the results comparable well with those of tensile creep tests.

2. The values of stress exponent n, activation energy Q, and threshold stress indicate a change of deformation mechanism within the above range of temperatures.

3. The applied amount of zirconium does not improve creep resistance efficiently at temperatures above 873 K.

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