THE INFLUENCE OF HEAT-TREATMENT ON CREEP RESISTANCE OF Fe–Al ALLOYS WITH ADDITIONS OF NIOBIUM AND CARBON

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Abstract
The influence of annealing on high-temperature creep of a Fe₃Al-type iron aluminide alloyed by niobium and different additions of carbon was studied in the temperature range from 600 to 800 °C. The alloys contained (atomic %) (i) 27.6 Al, 1.15 Nb, 0.19 C and (ii) 27.1 Al, 1.11Nb, 0.76 C (Fe balance). Creep tests were performed in compression at constant load with stepwise loading. Creep rate is increased by annealing at 1000 °C and it is reduced by annealing at 1150 °C.

Keywords: creep, high temperature deformation, intermetallics (iron aluminides)

1 Introduction
Iron-aluminides-based alloys are ideal candidates for the development of new structural materials with improved performance in petrochemical, power-generation and aeronautical applications [1, 2]. They have excellent resistance to oxidation and sulfidation. Their density is about two thirds of the density of steels. Moreover, they have high electrical resistance. The input raw materials are relatively cheap due to their occurrence in the earth’s crust. The main drawbacks of these alloys are a bad workability at room temperature and low high-temperature strength. The low-temperature plasticity can be improved by the off-stoichiometric compositions, ternary additives (chromium, molybdenum and manganese) and grain-refining agents (TiB₂ and Ce). Improvements in the creep resistance are expected to be obtained through solid solution hardening, through precipitation of second phase particles or through dispersion of particulates.

Niobium is the element that has a potential for both solid solution hardening and precipitation strengthening. This potential was recognized at least two decades ago. Nevertheless, it seems that the promising results of the pioneering study of the influence of niobium additions on creep of Fe₃Al-based alloy [3] were not fully exploited. A more detailed inspection of the original data of McKamey et al. shows that the certain scepticisms may be due to several possible reasons: There exists a discrepancy between tabulated data and their graphical presentation (cf. Table III vs. Fig. 2 in reference [3]). This has to be probably solved in favour of the tabulated data. The reported creep rates are then two orders of magnitude slower than it follows from a sketchy observation of the results. Furthermore, zirconium and carbon were present as unintentional contaminants from melting and casting process. The contamination may result in formation of secondary phases (Fe₃Al)₁₂Zr and (Fe₃Al)₁₂Zr and carbides ZrC and Fe₃AlC.

Extensive study of niobium additions to Fe₃Al-based alloys was reported by Morris et al. [4-6]. The improvement of mechanical properties at elevated temperatures was disappointingly small.
On the other hand, it was shown by Yu and Sun [7] that addition of 0.5% (atomic percent is given throughout) of niobium to Fe-28% Al-4% Cr alloy increased creep-rupture life by one order of magnitude. A similar beneficial effect on high temperature strength and creep resistance was observed for a small (0.83 %) addition of niobium to Fe-16% Al-0.43% C alloy [8]. Contrary to this optimistic observation, the same author reported that addition of niobium up to 2 wt. % (about 1.1 at. %) to Fe-19% Al-3.65% C alloy “did not exhibit any significant improvement in either creep life or minimum creep rate” [9].

The influence of 9.5% Nb on Fe-26% Al alloy was studied in both directionally-solidified and as-cast states. Creep properties of directionally-solidified alloy were comparable to those of P92 steel [10]. The mechanical properties of Fe–15% Al and Fe–26% Al based alloys with alloying additions of Nb (2 and 4%) and C (1%) were investigated by Fallat et al. [11]. Promising properties for high temperature applications were confirmed; creep was not tested.

In view of the above discrepancies, it was decided to study creep properties of Fe$_3$Al-based alloy with niobium addition. Since the contamination by carbon is inevitable in industrial production of this type of alloys, the alloys with different content of carbon were examined. In the present contribution, the results of creep tests of alloys annealed at 1000 °C and 1150 °C are given and compared with the results reported previously for hot-rolled alloys [12].

2 Experimental
The alloys were prepared by melting in the vacuum furnace and cast under argon in the Technical University in Ostrava, Czech Republic. The composition of the alloys is given in Table 1. The castings were hot rolled to the final thickness of 11 mm at 1473 K in several steps with 20% reductions for each pass. Parts of the castings were annealed either at 1000 °C for 1 hour and air cooled (the same procedure as applied by McKamey et al. [3]) or at 1150 °C (reported as the optimum temperature by McKamey and Maziasz for the alloy Fe-28Al-5Cr-0.5% Nb, 0.8% Mo, 0.025% Zr, 0.05% C, and 0.005% B, designation FA-180 [13]) also for 1 hour and air cooled.

<table>
<thead>
<tr>
<th>Al</th>
<th>Nb</th>
<th>C</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>27.6/15.44</td>
<td>1.15/2.21</td>
<td>0.19/0.047</td>
<td>Balance</td>
</tr>
<tr>
<td>27.1/15.18</td>
<td>1.11/2.14</td>
<td>0.76/0.19</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The samples for uniaxial compressive creep tests were prepared with the axis perpendicular to the rolling plane. The dimensions of samples were: square cross section 5x5 mm$^2$, height 10 mm. Constant load compressive creep tests of the alloy were performed at temperatures from 600 to 800 °C. 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. 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$^{-5}$.

3 Results
The applied stress dependences of the creep rate in both alloys in annealed states are given in Figs. 1 to 3. The dependences are as a rule described at a given temperature by the power function
\[ \dot{\varepsilon} = A \sigma^n, \]  

where \( A \) is a temperature dependent factor. The stress exponent \( n \) can be used to identify the rate-controlling mechanism. The values of \( n \) for both as-received and heat-treated states are summarized in Table 2. They are similar to those usually observed in iron aluminides (cf. Table 2 in ref. [14]). Note a significant decrease of the power \( n \) at lower creep temperatures resulting from the annealing at 1000 °C. Deviations of the experimental data from a power-law fit are clearly evident from figures. The convex-type deviations observed at lower temperatures can be in principle interpreted in terms of summation of creep rates resulting from diffusional mechanism and dislocation mechanism. An exact analysis is complicated by insufficient quantity of data at lower stresses. The concave-type deviations, visible at higher temperatures, can be described by means of the threshold stress concept [15]

\[ \dot{\varepsilon} = A'(\sigma - \sigma_{th})^{n'}, \]

where \( \sigma_{th} \) is the threshold stress and \( n' \) is the correct exponent for identification of the deformation mechanism.

![Fig.1 Applied stress dependence of creep rate in alloy with lower amount of carbon annealed at 1000 °C](image1)

![Fig.2 Applied stress dependence of creep rate in alloy with lower amount of carbon annealed at 1150 °C](image2)

<table>
<thead>
<tr>
<th>( \sigma ) [MPa]</th>
<th>10 (^{-5})</th>
<th>10 (^{-4})</th>
<th>10 (^{-3})</th>
<th>10 (^{-2})</th>
<th>10 (^{-3})</th>
</tr>
</thead>
<tbody>
<tr>
<td>600 C</td>
<td>0.19 % C</td>
<td>1.15 % C</td>
<td>2.20 % C</td>
<td>2.55 % C</td>
<td>2.90 % C</td>
</tr>
<tr>
<td>650 C</td>
<td>0.19 % C</td>
<td>1.13 % C</td>
<td>2.18 % C</td>
<td>2.53 % C</td>
<td>2.88 % C</td>
</tr>
<tr>
<td>700 C</td>
<td>0.19 % C</td>
<td>1.11 % C</td>
<td>2.16 % C</td>
<td>2.51 % C</td>
<td>2.86 % C</td>
</tr>
<tr>
<td>800 C</td>
<td>0.19 % C</td>
<td>1.09 % C</td>
<td>2.14 % C</td>
<td>2.49 % C</td>
<td>2.84 % C</td>
</tr>
</tbody>
</table>

Comparison of creep rates in as-received and heat-treated states is given in Figs. 4 and 5. For simplicity only lines following from power-law fitting are drawn. The annealing at 1000 °C evidently reduces the creep resistance. The lower the temperature and the applied stress the greater is the reduction of the creep resistance. On the other hand, the annealing at 1150 °C has a
beneficial influence on the creep resistance (the investigation of this annealing on the creep of alloy with 0.76 % C is in progress).

**Fig.3** Applied stress dependence of creep rate in alloy with greater amount of carbon annealed at 1000 °C.

**Fig.4** Comparison of creep rates in as received and heat treated alloy with lower amount of carbon.

**Fig.5** Comparison of creep rates in as-received and heat treated alloy with greater amount of carbon.

**Fig.6** Comparison of available creep data in Fe-Al-based alloys with niobium at 600 °C.

For comparison with previously published studies on niobium-containing Fe–Al alloys, **Fig. 6** shows creep data obtained at 600 °C for the present alloys as well as for two Fe–Al–Nb alloys with similar aluminium contents [3, 7]. The creep data reported for ferrite-based Fe-Al alloys with niobium additions are also included [8, 9]. The creep resistance of present low-carbon alloy is comparable to that of the McKamey’s material of similar Nb content. The data suggest that the creep resistance may be further improved by an additional heat treatment resulting in optimized particle sizes and distributions. The high-carbon alloy shows less creep resistance at
600 °C. This can be probably interpreted on the base of the high affinity of niobium and carbon. The formation of coarse niobium carbides reduces the amount of niobium available for precipitation in the form of the more stable Laves phase particles. The creep strength is then comparable with that of the alloy with lower niobium addition [7].

It follows from creep measurements that the present low-carbon alloy is more creep-resistant at lower temperatures, while the opposite is true at temperature of 800 °C. This can be interpreted in terms of possible occurrence of second-phase particles: In low-carbon alloy at lower temperatures the strengthening is due to Laves phase particles. The carbides are responsible for strengthening of high-carbon alloy at higher temperatures. This is in agreement with available section of Fe-Al-Nb-C system with constant contents of niobium and carbon [16].

4 Conclusions
The influence of annealing on creep of Fe–27Al-1Nb alloy with and without carbon additions was studied at temperatures from 600 to 800 °C. Creep rate is increased by annealing at 1000 °C and it is reduced by annealing at 1150 °C. Creep resistance of the low-carbon alloy is better at lower temperatures, while the opposite is true at higher temperatures. Strengthening by Laves phase is effective at temperatures below 700 °C, strengthening by carbides predominates at temperatures above 700 °C.

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References