

## IMPROVEMENT OF CREEP RESISTANCE OF Fe-28Al-3Cr ALLOY BY THERMAL TREATMENT

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Fe-28Al-3Cr (at.%) alloy with small amounts of Ce, C and Mn additives was tested in creep under constant load ranging from 20 to 280 MPa at temperatures of 500, 600 and 700 °C. The effect of high temperature annealing (1150 °C) on creep characteristics (creep rupture life, creep rupture strength, minimum creep rate, etc.) was investigated. The improvement of creep properties is supposed to be due to the solid solution and/or precipitation hardening. The proportion of both is substantially enhanced by solution annealing and subsequent precipitation during cooling to room temperature.

**Key words:** iron aluminides, creep properties

## ZLEPŠENÍ CREEPOVÉ ODOLNOSTI ALUMINIDU ŽELEZA Fe-28Al-3Cr TEPELNÝM ZPRACOVÁNÍM

Byla testována creepová odolnost slitiny Fe-28Al-3Cr (at.%) s malými množstvími legur Ce, C a Mn při konstantním zatížení v rozsahu 20–280 MPa při teplotách 500, 600 a 700 °C. Doba do lomu, minimální rychlost tečení a ostatní veličiny popisující creepové vlastnosti byly zlepšeny vysokoteplotním žháním při 1150 °C. Vliv souvisí s rozpuštěním příměsí a s následnou precipitací jemných částic během chlazení. Efekt je navíc kombinován se zpevněním atomy rozpuštěnými v mříži.

### 1. Introduction

Iron aluminides are well known for their excellent resistance to oxidation and sulfidation. The main drawbacks are their bad workability at room temperature and low high-temperature strength [1–3]. The influence of environmental effects was identified as the main reason for small plasticity of iron aluminides at room temperature. First experiments with Fe<sub>3</sub>Al aluminide are dated in the sixties, when

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they failed on the low ductility at room temperature (see, e.g., the review [3]). Only in recent years [2, 3] the improvement of the plasticity was achieved using both the off-stoichiometric compositions (e.g. 28 at.% Al) and ternary additives (especially chromium, molybdenum and manganese) in combination with grain refinement agents such as TiB<sub>2</sub> and Ce. There already exist the first examples of applications of Fe<sub>3</sub>Al-type alloys. Most of them are connected with the use of iron aluminides at high temperatures (e.g. heating elements, furnace fixtures, catalytic converter substrates, etc.). Therefore, the creep testing of this material is very important.

The knowledge on the creep of Fe<sub>3</sub>Al-type alloys can be summarized as follows: Most of the creep studies were made in the B2 phase field. Creep rupture properties of binary Fe<sub>3</sub>Al are reported to be poor, which is attributed to the easy tendency of grain boundaries to crack under tensile stresses [4]. Addition of the alloying elements [5, 6] like Nb, Mo, Zr, and W was shown to improve the creep rupture life and to reduce the minimum creep rate (*MCR*). In addition, it was shown in a complex alloy [6], that it is possible to improve the creep strength using changes of the heat treatment conditions. The improvement was achieved by a combination of precipitation and/or solid solution hardening.

It is the purpose of the present paper to verify whether this method for improving of the creep properties can be applied to the relatively simple alloys with very little additives.

## 2. Experimental procedure

The alloy was melted in a vacuum furnace and cast in an argon atmosphere at the Research Institute of Metals, Ltd. Panenské Břežany. The rolling of the original sheet (thickness 40 mm) to the final one (13 mm) was performed at 1100 °C. Finally, the sheet was quenched into the oil. No further heat treatment was applied before the samples were creep-tested at different temperatures. The composition of the studied alloy is (at.%): 22.4 Al, 2.6 Cr, 0.4 Mn, 0.16 C, 0.02 Ce. The creep samples were prepared with a gauge length of 25 mm and diameter of 5 mm. The creep testing was performed in air at constant load conditions (20 to 280 MPa) in temperature range from 500 to 700 °C. The accuracy of the temperature control was ±3 °C for testing temperatures 500 and 600 °C, ±4 °C for 700 °C. The deformation was measured by the analog extensometer with an accuracy of 10 μm.

Two series of samples were compared: the first one, which is identical with that studied in [7, 8] and the other, which are the same samples annealed additionally in air for 2 hours at 1150 °C and cooled outside the furnace (the cooling rate approx. 0.15 K/s).

The diamond saw was used to cut the samples for transmission electron microscopy to the thickness of 0.6 mm. Further thinning was mechanical grinding to 0.08 mm and final electrolytical jet polishing in 20% nitric acid in methanol at

−30°C. The observations of the structure of thin foils was performed using JEOL FX 2000 electron microscope.

### 3. Results and discussion

The relations between quantities characterizing the creep properties and deformation temperature and the applied load were estimated. The dependence of the time to rupture on the load for different temperatures is given in Fig. 1. The thin curves are those for the not annealed samples [7, 8].

The positive effect of the thermal treatment is obvious. E.g., for 500°C and the stress of 240 MPa, the enhancement of the time to rupture by the thermal treatment is threefold. This effect is decreasing with the increasing temperature but its favourable influence remains. As an example, the creep curves of two types of samples are compared for the load of 240 MPa and temperature of 500°C in Fig. 2.

The creep stress and minimum creep rate (*MCR*) as a function of load and temperature were also determined. The dependences of creep stress for 3% strain

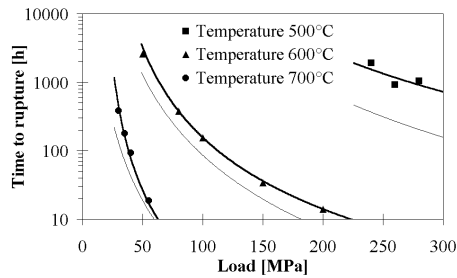


Fig. 1. Creep rupture strength (time to rupture) for both types of samples; thin curves correspond to the values reported in [7, 8].

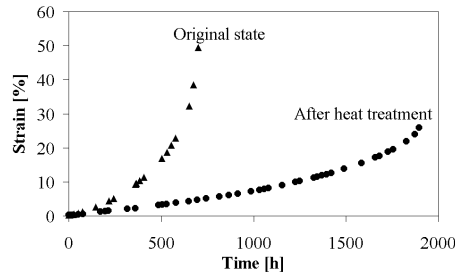
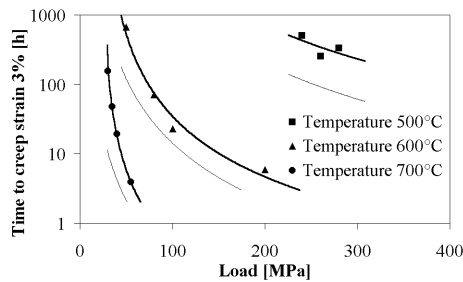


Fig. 2. The effect of thermal treatment on creep curves at 500°C/240 MPa.

Fig. 3. The effect of thermal treatment on creep stress for 3% strain (thin curves for not annealed samples).



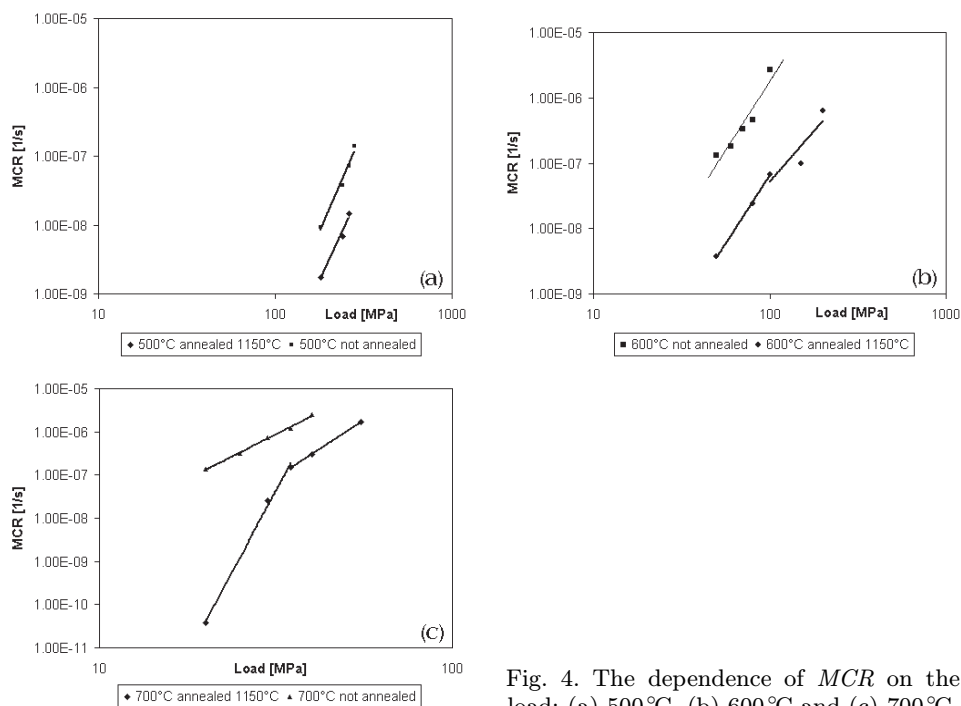


Fig. 4. The dependence of  $MCR$  on the load: (a) 500 °C, (b) 600 °C and (c) 700 °C.

(time to reach 3% strain) on temperature and load are given in Fig. 3. The dependence of  $MCR$  on the load for 500, 600 and 700 °C is given in Fig. 4.

The unambiguous effect of the thermal processing is shown in Fig. 3 and even in Fig. 4. Similarly to Fig. 1, the effect of the annealing decreases slightly with increasing temperature.

In order to improve the creep resistance, detailed investigation of the creep process study is necessary, e.g. the stress exponent  $n$ , which depends on the stress  $\sigma$  and temperature  $T$ . The higher  $n$  is, the better are the creep properties. The creep process can be described by a formula for  $MCR$ :

$$MCR = d\varepsilon_o/dt = A\sigma^n \cdot \exp(-Q/kT), \quad (1)$$

where  $Q$  is the activation energy for creep and  $A$  is a constant. The values of  $n$  up to 14 may be deduced at 600 and 700 °C depending on the range of loads, which is taken into account (see Fig. 4). Similarly, McKamey et al. [6] obtained  $n$  ranging from 10 to 20 in the iron aluminide Fe-28Al-5Cr alloyed mainly by Nb and Mo.

The data presented in Fig. 4 demonstrate the substantial decrease of  $MCR$  after thermal processing. This may result in the increase of the time to rupture. The increase of the creep resistance after high temperature annealing at 1150 °C



Fig. 5. Microstructure after creep at 600°C of the material annealed before deformation at 1150°C/2 h.

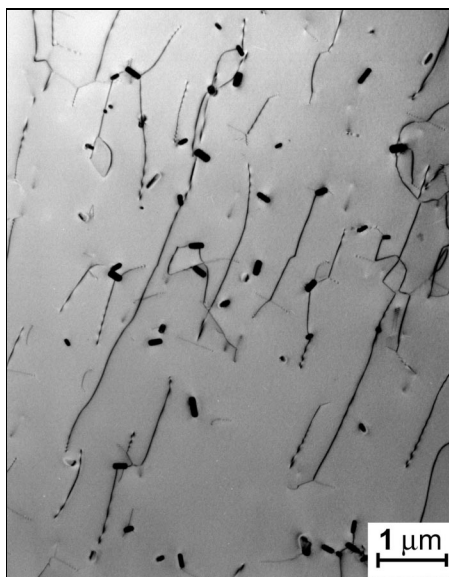


Fig. 6. The microstructure of the sample, which was not annealed prior to creep deformation.

followed by air cooling to room temperature is according to [6] connected with combined effects of solid solution hardening and/or precipitation strengthening. Similar explanation may be supported by our observation of the microstructure developed in our samples crept at 600°C after annealing at 1150°C (load of 100 MPa). Compared to the samples, which were not annealed (Fig. 6), the structure of the annealed samples (Fig. 5) obeys the arrangement of small particles pinning the dislocations. The composition of these particles was determined by EDAX analysis and the results are influenced by the composition of the matrix. The typical compositions are 50–77 Cr and 35–55 Fe (at.%). Approx. 5–10 at.% of C are present in some of the particles. The detailed structural analysis is necessary in the future.

#### 4. Conclusion

The thermal treatment consisting of the annealing at 1150°C/2 h followed by free cooling to room temperature improves the creep properties of the iron aluminide.

This improvement is connected with the formation of fine precipitates containing Fe, Cr and C. The contribution of solid solution hardening by other elements present in the alloy (Mn, C, Ce) is also probable.

The effect is decreasing with increasing creep temperature, but its favourable influence remains.

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