CREEP BEHAVIOUR OF THE INTERMETALLIC Fe-28Al-3Cr ALLOY

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The investigated material containing, compared to the basic composition Fe-28Al--4Cr (at. %), small amounts of Ce, C and Mn, was tested in creep at temperatures between 500 and 900 °C. The values of the activation energy Q and of the stress exponent n are compared to those obtained in experiments with materials containing Nb, Mo, Zr, C and B additives. The role of the heat treatments prior to creep testing is discussed.

K e y w o r d s: iron aluminides, creep properties

TEČENÍ ALUMINIDU ŽELEZA Fe-28Al-3Cr

Zkoumaný materiál, obsahující ve srovnání se základním složením Fe-28Al-3Cr (at. %) malé množství Ce, C a Mn, byl testován při tečení při teplotách 500 až 900 °C. Hodnoty aktivační energie Q a napěťového exponentu n jsou srovnávány s hodnotami, které byly zjištěny při experimentech s materiálem obsahujícím Nb, Mo, Zr, C a B. Je diskutován vliv tepelného zpracování před tečením.

1. Introduction

Iron aluminides are well known for their excellent resistance to oxidation and sulfidation. The main drawbacks are their bad workability at room temperatures and low high-temperature strength [1–4]. The influence of environmental effects was identified as the main reason for small plasticity of iron aluminides at room temperature. Several procedures were proposed to reduce this effect. One of the possibilities is alloying by chromium. First experiments with Fe_3Al aluminide are dated in the sixties, when they failed on the low ductility at room temperature (see e.g. review [3]). Only in recent years [2, 3] the improvement of the plasticity

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was achieved using both the off-stoichiometric compositions (e.g., 28 at. % Al) and the ternary additives (especially chromium, molybdenum and manganese) in combination with grain refinement agents such as TiB_2 and Ce. In contrast to the advantages, low ductility, brittle fracture and processing problems hampered the commercial use. Nevertheless, there already exist the first examples of application of Fe_3Al-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 about the creep of Fe_3Al-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_3Al are reported to be poor, which is attributed to the tendency of grain boundaries to crack under tensile stresses easily [5, 6]. Addition of the alloying elements [7–10] like Nb, Mo, Zr and W was shown to improve the creep rupture life and to reduce the minimum creep rate. In addition, it was shown in a complex alloy [10–12] that it is possible to improve the creep strength using changes of the heat treatment conditions. The improvement was also achieved through the presence of a second phase.

The most experiments concerning the creep of B2 structures were undertaken with alloys with compositions near to FeAl, mainly Fe-40at.%Al (see e.g. [13]). There exist only few creep investigations on materials with composition similar to Fe-28Al-4Cr (at. %) [10–12, 14, 15]. Very recent data are by Sundar et al. [14––15], who studied the creep behaviour of Fe_3Al-based alloys in both B2 and $D0_3$ phase fields. They used the creep impression experiments and had shown that a power-law creep is obeyed in the stress range from 100 to 500 MPa. They have investigated five types of materials on similar basis: Fe-27.6Al, Fe-28.7Al-2.5Cr, Fe-28Al-2Cr-0.04B, Fe-29.2Al-3.7Mn and Fe-27.2Al-3.6Ti (at. %). The values are hardly comparable to other data, because of non-standard creep conditions during the impression tests.

Recent most important creep measurements on Fe_3Al-based alloys are due to McKamey and co-workers [10–12], who studied the behaviour of a very complex alloy Fe-28Al-5Cr (at. %) with 0.5 Nb, 0.8 Mo, 0.025 Zr, 0.05 C and 0.005 B (Oak Ridge National Laboratory – ORNL – designation FA 180). They had shown that the creep rupture strength is strongly dependent on the microstructure. The creep rupture resistance of this alloy could be improved by solution annealing at 1150 ℃ followed by air cooling. The strengthening was due to a dispersion of fine Nb- and Zr-based MC precipitates in the matrix and along the grain boundaries. Even better results were obtained with samples, which were oil-quenched after the solution annealing. During their creep tests (200–250 MPa) performed at 600 and 650 ℃ the minimum creep rate (MCR) was between 10^{-8} to 10^{-10} s^{-1}. They got extreme values of the stress exponent n (10 to 20). The values of the activation energy Q were 350 and 590 kJ/mol.

Also Chen and co-workers [16] investigated the possibility to improve the high temperature properties of Fe_3Al based aluminides by alloying with Mo, Nb, Cr and some minor additives. Most data are related similarly as in [10–12] to creep tests at 600 ℃ and 200 MPa. Neither the alloying nor the microstructure enhanced the creep resistivity to values obtained by McKamey [10], who used the same type of material, but the application of high annealing temperature near 1150 ℃.

It is the purpose of the present investigations to perform first creep tests with an iron aluminide alloy of the type Fe-28Al-5Cr (at. %) modified mainly by Ce, Mn and C.

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 ℃. Finally, the sheet was quenched in oil. No further heat treatment was applied before the samples were creep-tested at different temperatures. The composition of the studied alloy was (at. %): 22.4 Al, 2.6 Cr, 0.4 Mn, 0.16 C, 0.02 Ce. The alloy was denoted as FA 282 [17]. The creep samples were prepared with the gauge length of 25 mm and the diameter of 5 mm. The creep testing was performed in the Research Center, SVÚM Běchovice, joint-stock comp., in air at constant load conditions in temperature range from 500 to 900 ℃. The range of loads was 5 to 100 MPa for 600 to 900 ℃ and 100 to 200 MPa for 500 ℃. The accuracy of the temperature control is given by the Czech standards ČSN 420351, i.e. $\pm$ 3 ℃ for testing temperatures up to 600 ℃, $\pm$4 ℃ for the range 600–800 ℃ and $\pm$6 ℃ for 800–1000 ℃. The deformation was measured by the analog extensometer with the accuracy of 10 μm.

A diamond saw was used to cut the samples for metallography. The final polishing and etching was performed with an OPS – STRUERS etchant. The observations of the structure were performed for both undeformed and deformed parts of the samples.

3. Results

Creep tests were conducted as a function of temperature and load in order to determine the activation energy of creep Q and the stress exponents n. The dependence of minimum creep rate (MCR) on the load at different temperatures is given in Fig. 1. The stress exponents determined for different temperatures are in Table 1. The activation energies of the creep Q evaluated from Fig. 2 are 500 and 320 kJ/mol for loads of 10 and 20 to 30 MPa, respectively. The type of the experimental results (creep under constant load) enables to use the evaluation for constant stress experiments. The reason is following: MCR was reached for deformation of approximately 3 %. For this value the load is very near to the

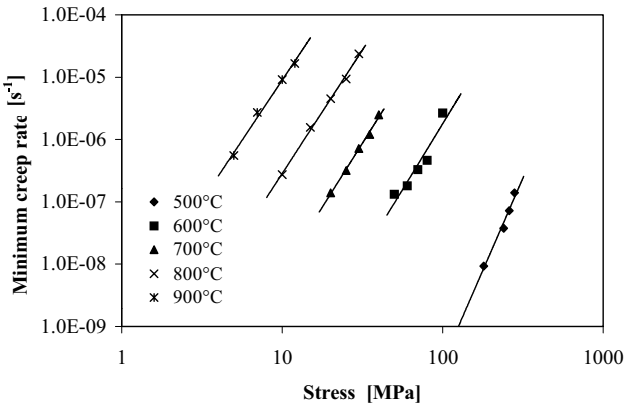

Fig. 1. Dependence of the minimum creep rate on stress at temperatures ranging from 500 to 900 °C.

T a b l e 1. Stress exponents as determined in the present work

Temperature [°C]		n
DO_3	500	5.92
B2	600	4.23
	700	4.10
	800	3.96
	900	3.86

stress, which differs only by a factor of 1.03. Thus, the values of n and Q can be compared with experiments using constant stress.

From the technical point of view, among the quantities, which determine the possibility to use the material at high temperatures, the important one is the time to rupture depending on load and temperature (Fig. 3). These values of the time to rupture cannot be directly compared to those of McKamey et al. [10], while the time to rupture for constant load experiments is approximately the half of that obtained during the creep experiments with constant stress. The studied material may be therefore taken as a material with properties comparable to that with Nb, Mo, Zr, C, and B (amounts are mentioned above) [10].

The optical microstructure after creep was determined by comparing the strained microstructure from the gauge region of the specimen to the unstrained microstructure from the heads of the specimen. The unstrained specimen is that obtained by rolling at 1100 °C followed by quenching in oil. The unstrained structure is composed of the grains elongated in the direction of rolling (Fig. 4). Depending on

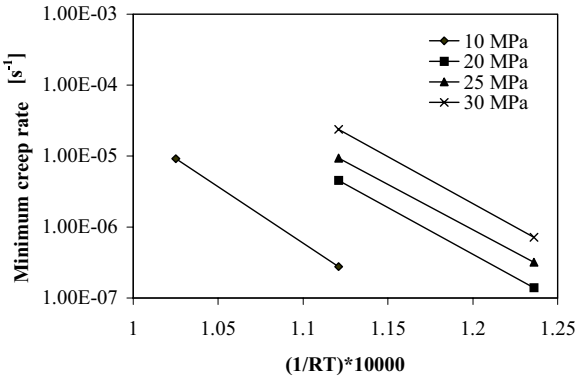

Fig. 2. Determination of the activation energy for creep Q for FA – 282.

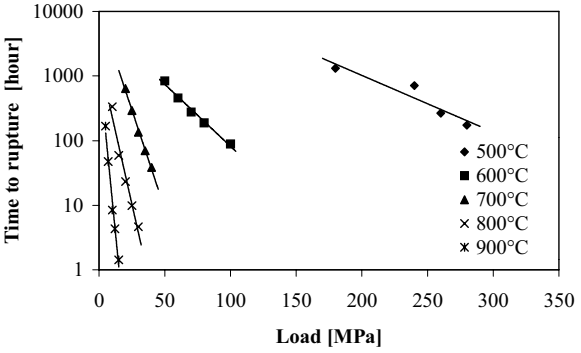

Fig. 3. Dependence of the time to rupture on load and temperature.

the creep temperature the sample (undeformed parts of the sample) recrystallizes between 800 and 900 ℃ (Fig. 5).

The following features are characteristic for the deformed parts after the rupture:

– shape of the grain boundaries (buckling) witnessing the dynamic or metadynamic recrystallization process at temperatures near to 800 ℃ (Fig. 6);

– polygonal recrystallized grains appearing as a result of the recrystallization process at 900 ℃ (Fig. 7);

– precipitates (size 2–10 μm) observed in the optical micrograph within the grains and along the grain boundaries. These were identified by Karlik et al. [18] as Ce containing particles.

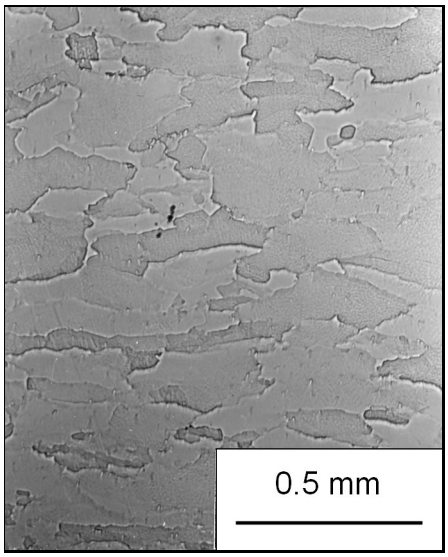

Fig. 4. Optical structure of the undeformed sample (after hot rolling at 1100 °C and quenching in silicon oil).

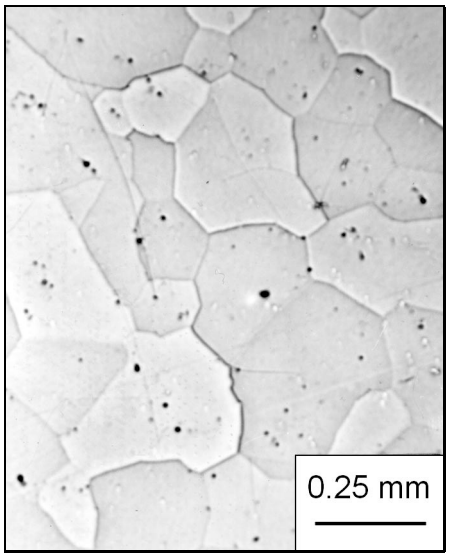

Fig. 5. Optical structure of the same sample as in Fig. 4 but annealed at 850 °C/2 h.

4. Discussion

The values of n and Q (of a given material) depend on the stress (load) and temperature, which influence the creep process. The higher n and (independently) Q are, the better is the creep resistance. MCR in the material having a good creep resistance is generally smaller compared to the material with the worse resistance. The possibilities to enhance the creep resistance (e.g. the stress to rupture and time for rupture) are the use of solid solution hardening and/or precipitation strengthening.

Comparing our data with those of McKamey et al. [10–12] and Sundar et al. [14–15] the following can be stated:

– The alloy was crept at temperatures, where it has B2 structure, with the exception of experiments at 500 °C.

– The values of Q (350 and 590 kJ/mol) and n (10 to 20) in [10] are due to the high alloying (combined effects of solid solution and precipitation strengthening after high temperature annealing at 1150 °C followed by air cooling to room temperature). The material studied in the present work contains small amounts of Ce, C and Mn (in the basic composition Fe-28Al-4Cr (at. %)). The material was hot rolled at 1100 °C and hold at this temperature one hour before the rolling. This might be taken as comparable to solution treatment at 1150 °C. On the other hand,

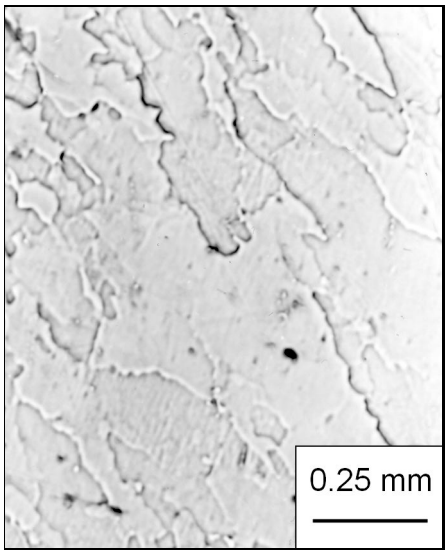

Fig. 6. Optical structure of the sample as in Fig. 4 after the creep experiment at 600 °C (load of 30 MPa).

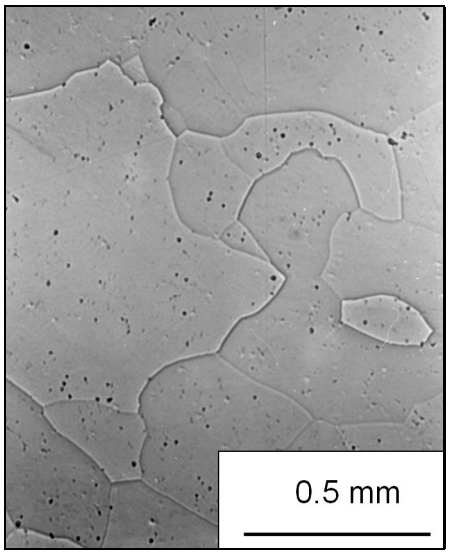

Fig. 7. Optical structure of the sample as in Fig. 4 after the creep experiment at 900 °C (load of 10 MPa).

the high temperature creep at 800 and 900 °C was employed here too. It cannot be expected that the effect of holding at 1100 °C will be also effective at temperatures substantially higher than 600 °C (800 and 900 °C).

– At temperatures in the $D0_3$ structural range the stress exponent n for series of materials ranged from 4.0 to 5.6 [14, 15] in comparison with our $n = 5.9$. Full comparison with our data is not possible because of higher stresses and different experimental procedure (hot indentation creep tests). Activation energies for creep Q ranging from 217 to 285 kJ/mol in [14, 15] are (compared to our results) lower.

– For structural range B2 the values of n were determined to be from 2.7 to 3.8 and Q from 325 to 375 kJ/mol in [14, 15]. We got higher n and similar Q. It is possible to find the reason for our higher values of n and Q: the sheets, from which the creep samples were machined, had been hot rolled at 1100 °C and then slowly cooled down to room temperature. This is similar to that used by McKamey et al. [10] – combined effect of solute and precipitates. Most creep curves in [10] have been taken at constant stress of 200 MPa at 600 °C.

– We were not able to reach the creep resistivity described by $n = 10-12$, which corresponds to 1150 °C annealing and to completely other chemical composition of alloy. Nevertheless, the method may be used in future to prepare better creep resistant materials.

5. Conclusions

The iron aluminide Fe-28Al-3Cr (at. %) with Ce, C and Mn as small amount additives (FA 282) was presented as a material with promising creep properties.

A modified thermomechanical processing is expected to improve the creep properties of the investigated material.

The use of other combination of additives can enhance the solid solution and precipitation strengthening of the studied material.

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