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Creep behaviour of intermetallic Fe–28Al–3Cr alloy with Ce addition

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Abstract

The creep behaviour of the intermetallic Fe–28Al–3Cr alloy with Ce additive was investigated at temperatures from 500 to 900 °C. The effect of the thermal processing was studied. The properties (e.g. time to rupture, minimum creep rate (MCR), the ultimate deformation) were improved by introducing small particles originating in the temperature interval from 600 to 800 °C.

Keywords: Intermetallics; Transmission electron microscopy; Creep

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]. First examples of application of Fe_3Al-type alloys are connected with the use of iron aluminides at high temperatures [2] (e.g. heating elements, furnace fixtures, catalytic converter substrates, etc.). Therefore, the creep testing of this material is very important.

Most of the creep studies were made in the B2 phase field (at temperatures over 560 °C). Creep rupture properties of binary Fe_3Al are poor, which is attributed to the easy tendency of grain boundaries to crack under tensile stresses [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 (MCR). In addition, it was shown in a complex alloy [10–12], that it is possible to improve the creep strength using heat treatment. 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–40 at.%Al (see e.g. [13]).

Important creep measurements on Fe_3Al-based alloys are due to McKamey et al. [10–12], who studied the behaviour of a complex alloy Fe–28Al–5Cr (at.%) with 0.5 Nb, 0.8 Mo, 0.025 Zr, 0.05 C and 0.005 B. They showed that the creep strength is strongly dependent on the microstructure. The creep rupture resistance of this alloy was improved by solution annealing at 1150 °C 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. During their creep tests (200–250 MPa) performed at 600 and 650°C the minimum creep rate was between 10^{-8} and 10^{-10} s^{-1}.

Very recent data are by Sundar et al. [14,15], who studied the creep behaviour of Fe_3Al-based alloys in both B2 and D03 phase fields. They used the creep impression experiments and obtained that power law creep is obeyed in the stress range from 100 to 500 MPa.

Also, Chen et al. [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 as in [10–12] to creep tests at 600 °C and 200 MPa. Neither the alloying nor the microstructure improved the creep strength to values obtained by McKamey and Masiasz [10], who used the same type of material (the difference was the addition of the high-temperature annealing near 1150 °C).

It is the purpose of the present paper to describe the creep properties of an iron aluminide alloy of the type Fe–28Al–4Cr (at.%) modified by Mn, Ce and C. The effect of high-temperature annealing comparable to [10] was also investigated.

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2. Experimental procedure

The alloy was prepared in a vacuum furnace and cast in an argon atmosphere by the Research Institute of Metals, Ltd. Panenské Břežany, which did the rolling of original ingots (thickness 40 mm) to the final sheets (13 mm) at 1100 °C as well. Finally the sheet was quenched into the oil. The composition of the studied alloy is (at.%): 28.4 Al, 2.6 Cr, 0.4 Mn, 0.16 C, 0.02 Ce. The samples for creep testing were prepared with the gauge length of 25 mm and the diameter of 5 mm. The experiments were performed in air under constant load conditions in temperature range from 500 to 900 °C.

Three series of samples were compared:

- A—without any thermal processing;
- B—annealed in air for 2 h at 1150 °C and cooled freely outside the furnace;
- C—annealed in air for 2 h at 700 °C and cooled freely outside the furnace.

The creep tests of individual series took place at following temperatures:

- A—500, 600, 700, 800 and 900 °C;
- B—500, 600 and 700 °C;
- C—600 °C.

The diamond saw was used both to cut the samples for transmission electron microscopy (TEM) to the thickness of 0.6 mm and to cut the samples for metalography (final polishing and etching was performed with an OPS-STRUERS etchant). Further thinning for TEM 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 microcsope.

3. Results and discussion

Creep tests were conducted as a function of temperature and stress in order to determine the activation energy of creep Q and the stress exponents n. The creep process was described by a formula for MCR:

$$\mathrm{MCR} = \left(\frac{\mathrm{d}\varepsilon}{\mathrm{d}t}\right)_{\mathrm{o}} = A\sigma^{n}\exp\left(\frac{-Q}{kT}\right), \qquad (1)$$

where Q is the activation energy for creep and A a constant. The dependence of minimum creep rate on the load at different temperatures is given for samples A, B and C in Fig. 1. These dependencies can be assimilated to those for constant stress experiments. The reason is that: MCR was reached for approximately 3% deformation. For this value the load

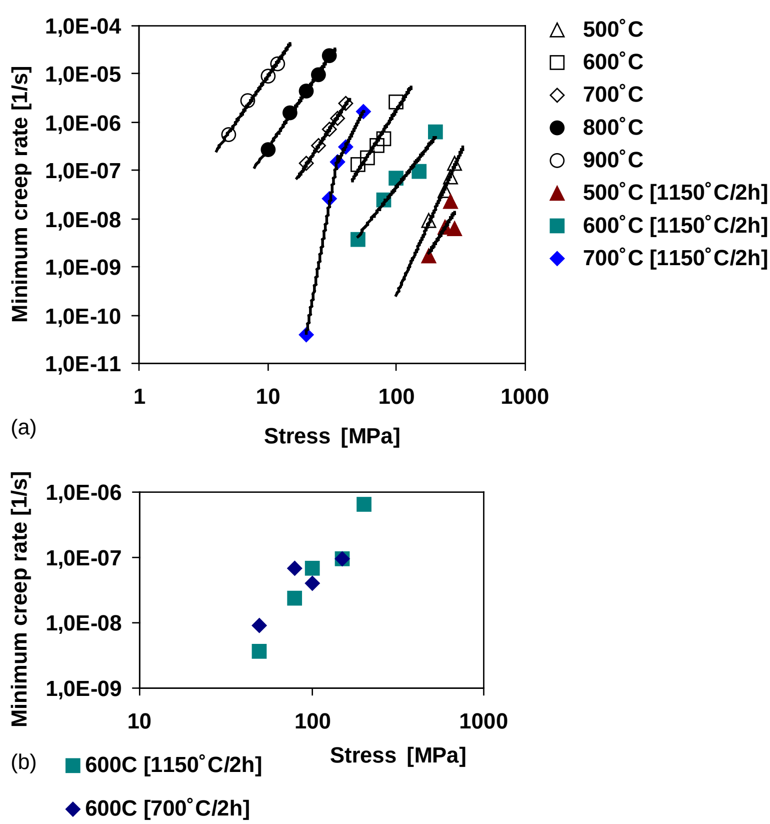

Fig. 1. (a) The dependence MCR—stress for different temperatures for 500, 600, 700, 800, 900 °C for samples A and for 500, 600, 700 °C for samples B; (b) the comparison of MCR—stress dependencies for B and C samples at 600 °C.

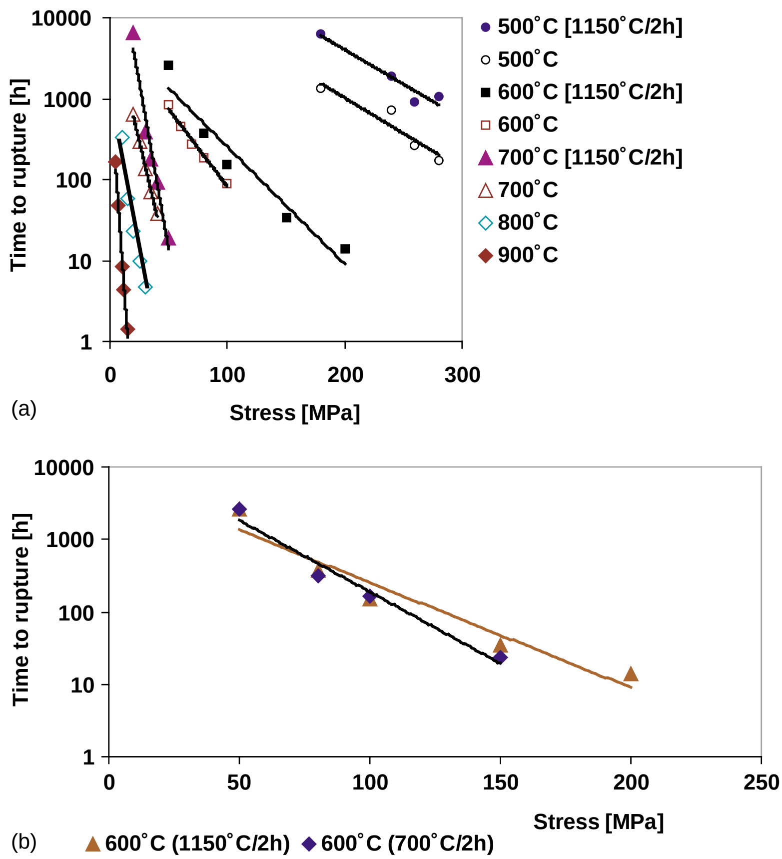

Fig. 2. (a) The time to rupture for different temperatures for 500, 600, 700, 800, 900 °C for samples A and for 500, 600, 700 °C for samples B; (b) the time to rupture at 600 °C compared for samples B and C.

is very near to the nominal stress, from which it differs only by a factor of 1.03.

From the technical point of view, time to rupture is the most important parameter, which determines the possibility to use the material at high temperatures (Fig. 2). The positive effect of the thermal treatment is obvious. This effect decreases with the increasing temperature but its favourable influence remains. The stress exponents *n* are in Table 1. The activation energies of creep (*Q*) was estimated to be 320 kJ/mol (stress 20–30 MPa) for the B2 structure (Table 2).

The structure of the unstrained specimen consists of grains elongated along the direction of rolling. Depending on temperature this substructure recrystallizes between 800 and 900 °C. The structure does not change during creep at temperatures ≤800 °C.

The samples, which were not annealed and those which were, differ substantially by the density of the observed precipitate (see Figs. 3 and 4). There exist no particles in the samples crept at 900 °C any more. This behaviour can be correlated with the estimates made by Karlik and Cieslar [17]. The particles visible in both the not annealed and annealed samples were identified by Karlik and Cieslar [17] as a Cr–Fe–C precipitate with hexagonal crystal lattice. The carbon content in the particles ranges from 15 to 30 at.%, the Cr:Fe ratio in the matrix and grain boundary is 7:3 and 6:4, respectively. However, Cr–Fe particles without carbon were also found. The stability of the mentioned precipitate was also studied [17], see Fig. 5. Annealing at 950 °C for 2 h is sufficient to dissolve Cr–Fe–(C) precipitate. In the so-

Table 1
The comparison of values of *n*

T (°C)	*n*				
	A	B	C	[14,15]	[10]
500	5.9	4.5	–	4.0–5.6[a]	
600	4.2	3.3	3.5	2.7–3.8[a]	10–20[b]
700	4.1	5.6–15.8	–	–	–
800	4.0	–	–	–	–
900	3.9	–	–	–	–

[a] The data correspond to the intervals 480–550 °C and 600–670 °C, respectively.
[b] Quenched and slowly cooled, respectively.

Table 2
The values of the activation energies *Q*

	Q (kJ/mol)		
	A (B, C)	[14,15]	[10]
B2	320	325–375	627
D03		217–285	–

Fig. 3. Structure of the not annealed sample (A) after creep at 600 °C.

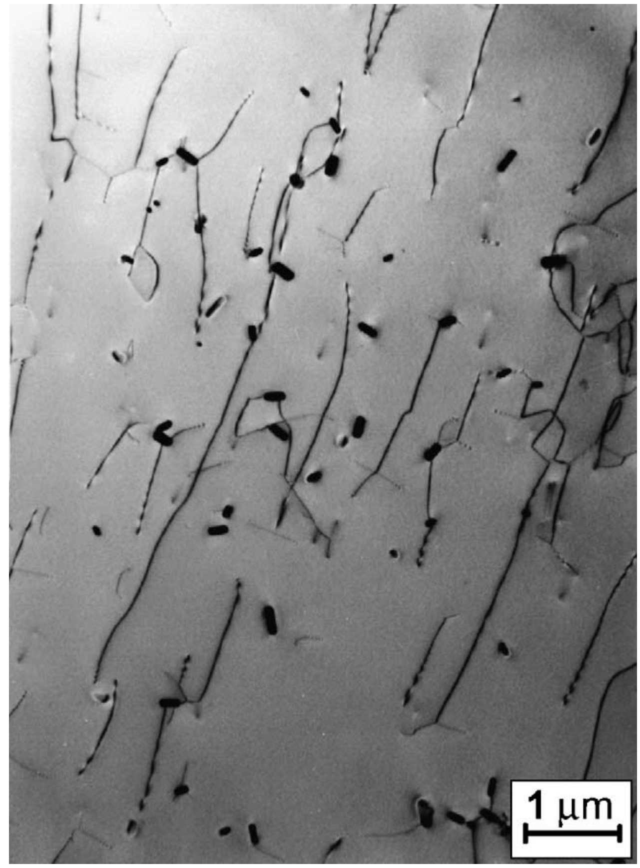

Fig. 4. Structure of the annealed sample (B) after creep at 600 °C.

lution treated and quenched alloy, the precipitate reappears during heating in the range from 300 to 800 °C.

The material used in this study contains little Ce, C and Mn in Fe–28 at.%Al–4 at.%Cr. Samples A, B and C are crossing during their cooling more or less the precipitation diagram (Fig. 5). Creep takes place for temperatures 600 and 700 °C certainly inside the nose. The comparison of our data with those of McKamey et al. [10–12] and Sundar et al. [14,15] is shown in Tables 1 and 2. The diferences of the stress exponent n are noticeable between B2 and D03 regions both in present study and in [14,15]. The values themselves in both cases do not differ as much. On the other hand the values obtained in B2 by McKamey and Masiasz [10] are extreme. This is connected [10] with precipitation and/or solid solution hardening due to the annealing processes employed by the authors of [10]. We were similarly able to enhance the values of the time to rupture as well as to lower MCR by similar processing (annealing at 1150 °C)—samples B. It is worth to mention, that the same effect was obtained in the case of samples C, which were annealed at 700 °C. The explanation of the behaviour of samples A, B and C is that the particles described in [17] form if the material crosses the nose in the PTT diagram. This takes place either during cooling from 1150 °C (B samples) or during annealing at 700 °C (C samples). During creep at 600 or 700 °C the conditions for the particle formation are fulfilled. TEM observation of the structure of samples crept at 900 °C even support the dissolution of particles as predicted in Fig. 5.

In spite of the fact that the effect of the annealing at 1150 °C was similar to that observed by McKamey and Masiasz [10], the situation is different. The results with samples C support the explanation, that the hardening by particles in our case is limited to the temperature region in which the particles do not dissolve again. The positive effect of the described process can be exploited only during the high temperature use of the material between 600 and 800 °C. For other cases composition must be changed so as

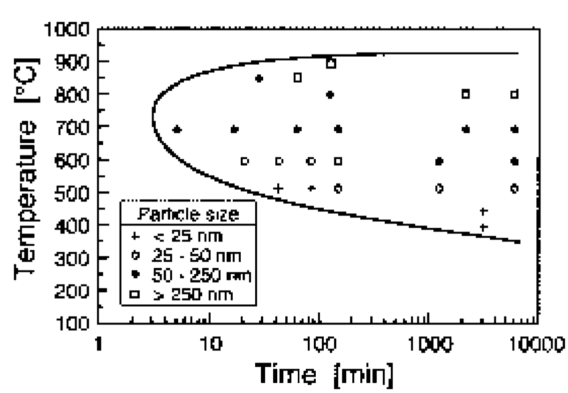

Fig. 5. PTT diagram of the studied alloy, after [17].

to enable the formation of carbides stable at temperatures as high as 800 °C.

The introduced obstacles lower the MCR and consequently the total deformation at rupture is lowered, which is a positive effect with respect to the change of the shape of potential products under such conditions.

4. Conclusions

Substantial improvement of the creep resistance was achieved by high-temperature annealing at 1150 °C and is due to the precipitates containing Fe, Cr and C. This precipitate is appropriate to enhance the creep strength of the studied material at temperatures from 600 to 800 °C.

The observed enhancement of creep properties (lowering of MCR, enhancing of the time to rupture, deformation to rupture) differs from that due to carbides ZrC, MoC [10].

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