

TEM STUDY OF Fe₃Al BASED ALLOY AFTER CREEP DEFORMATION

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The microstructure of Fe-28Al-3Cr (at.%) intermetallic alloy with additions of Mn, Ce and C was studied by means of transmission electron microscopy. The observations are discussed with respect to previous results on creep behaviour at the temperature range from 600 to 900 °C. The Cr-Fe-C particles were observed in all samples except of the sample crept at 900 °C. Creep properties of the alloy are improved due to solid solution hardening and particle strengthening.

Key words: iron aluminides (based on Fe₃Al), microstructure, transmission electron microscopy

TEM STUDIUM SLITINY NA BÁZI Fe₃Al PO DEFORMACI V CREEPU

Pomocí transmisní elektronové mikroskopie byla studována mikrostruktura intermetalické slitiny Fe-28Al-3Cr (at.%) s příměsí Mn, Ce a C. Pozorovaná zjištění jsou diskutována s ohledem na předchozí výsledky deformace v creepu při teplotách od 600 do 900 °C. Ve všech vzorcích byly pozorovány částice typu Cr-Fe-C s výjimkou vzorku podrobenému creepu při teplotě 900 °C. Creepové vlastnosti slitiny jsou lepší v důsledku příměsového a precipitačního zpevnění.

1. Introduction

Iron aluminides based on Fe₃Al are well known as materials with excellent resistance to oxidation and sulfidation. Technical applications are limited by difficult workability at room temperature and decrease of strength at the temperatures above 600 °C [1–3]. Their low plasticity at room temperature is connected with the influence of the environmental effects on the deformation properties [4]. Recently,

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the improvement of the plasticity was achieved using off-stoichiometric compositions and addition of alloying elements, such as chromium, manganese, carbon, and molybdenum [1, 2].

The application of Fe₃Al type alloys at high temperatures (e.g. furnace fittings, heating elements, parts in automobile industry and elements used in melted salts environment) requires more knowledge about their high temperature behaviour. Most of the creep studies were carried out in the B2 phase region [5, 6]. The creep resistance and life to rupture of a binary alloy are poor, with frequently observed intergranular mode of fracture. It has been shown that addition of molybdenum and particularly niobium lead to considerable improvements in creep resistance and life [5–7]. These improvements have been attributed to solution hardening and particle dispersion strengthening. Similar behaviour was observed in the case of Fe-28Al-4Cr (at.%) alloy with additions of Mn, Ce and C [8–11].

This paper presents the transmission electron microscopy (TEM) study, which was carried out in order to explain creep behaviour of the Fe-28Al-3Cr (at.%) alloy with addition of Mn, Ce and C in the temperature range from 600 to 900°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. Original ingots (thickness of 40 mm) were rolled to sheets (thickness of 13 mm) at the temperature of 1100°C and then quenched into the oil bath. The composition of the alloy was 28.4 Al, 2.6 Cr, 0.4 Mn, 0.16 C, 0.02 Ce (Fe balance) in at.%.

Creep tests were performed at the temperatures of 600, 700 and 900°C [10]. In the case of deformation at 600°C, the specimen was also tested after preliminary heat treatment at the temperature of 1150°C for 2 h followed by cooling on air.

After the creep test the deformed part of the specimen was cut to discs with thickness of about 0.6 mm. Further thinning was done by mechanical grinding to thickness of 0.08 mm. The foils for TEM were prepared by electrolytic jet polishing in STRUERS Tenupol 2. The device was filled with 20 % solution of nitric acid in methanol and the conditions for polishing were: $T = -30^\circ\text{C}$, $U = 15\text{ V}$. Thin foils were observed by means of the analytical electron microscope JEOL 2000 FX operating at 200 kV.

3. Results and discussion

The results of creep tests were recently published elsewhere [8–10, 12]. Therefore only two dependencies are presented to demonstrate improvement in creep properties in the present study. Figure 1 shows dependence of the minimum creep rate on the stress during creep at 600, 700 and 900°C. Comparing these results with those of the binary alloy [13], it is obvious, that the alloying elements improve the

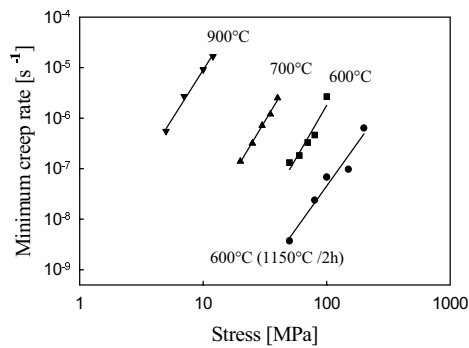


Fig. 1. Dependence of the minimum creep rate on the stress for Fe_3AlCr intermetallic alloy at various temperatures. Before the test, one of the samples crept at 600°C was heat treated for 2 h at temperature of 1150°C and then cooled on air.

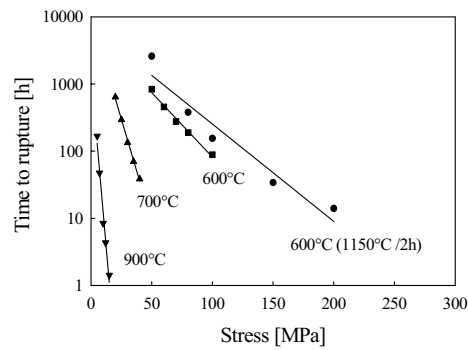
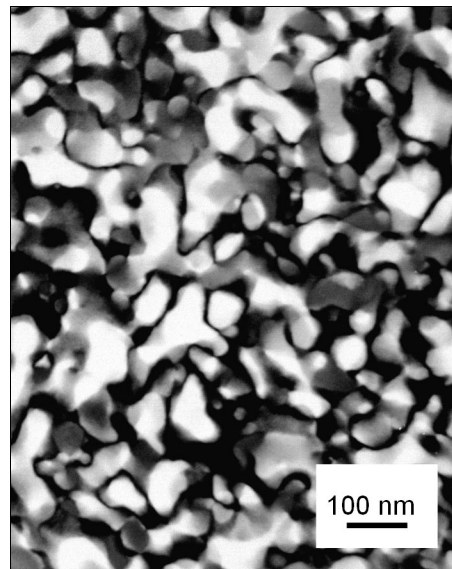


Fig. 2. Time to rupture for creep test of Fe_3AlCr intermetallic alloy at various temperatures. Before the test, one of the samples crept at 600°C (●) was heat treated for 2 h at temperature of 1150°C and then cooled on air.

Fig. 3. Dark field image of Fe_3Al domains in the sample after creep at 600°C and cooling on air.



creep properties. The preliminary heat treatment at 1150°C (compare results at 600°C – ●, ■) has positive influence as well. The same conclusions could be drawn in the case of time to rupture, as seen in Fig. 2. The values of stress exponent n ranged from 3.5 to 4.2, and the activation energy of creep Q was determined to be 320 kJ/mol [12].



Fig. 4. Microstructure of the sample after creep test at 600°C.

In all specimens, TEM revealed mixture of B2 ordered matrix (occurrence above approx. 550°C) and very fine ordered domains of Fe_3Al with D0_3 crystal structure (occurrence below approx. 550°C) (Fig. 3).

– *The microstructure after creep at 600°C.* Large particles (ranging from 200 to 500 nm in size) were found in material after hot rolling at the temperature of 1100°C and quenching into the oil bath. They have been irregularly distributed inside the grains and formed also clusters along the subgrain boundaries. The precipitates are composed of Cr, Fe and C. The X-ray energy dispersive (XEDS) analysis revealed the ratio of Cr to Fe 7 : 3 or 6 : 4. Some of the Cr-Fe rich particles are carbides with the carbon content ranging from 15 to 30 at.% C [14]. The dislocation structure has exhibited typical recovery features with parts of subgrain network and low density of dislocations inside the subgrains. In contrast to the dislocation substructure, the creep deformation at 600°C did not change the particles (Fig. 4). The parts of subgrain boundary networks were again observed but inside the subgrains the dislocation density was higher and the dislocations were partly pinned by the particles. The majority of dislocations were arranged as pairs of 2-fold dissociated superdislocations.

– *The microstructure of samples crept at 600°C after heat treatment at 1150°C/2 h.* The microstructure developed in the specimen after hot rolling at 1100°C (oil quenching) followed by heat treatment at 1150°C for 2 h (cooling on

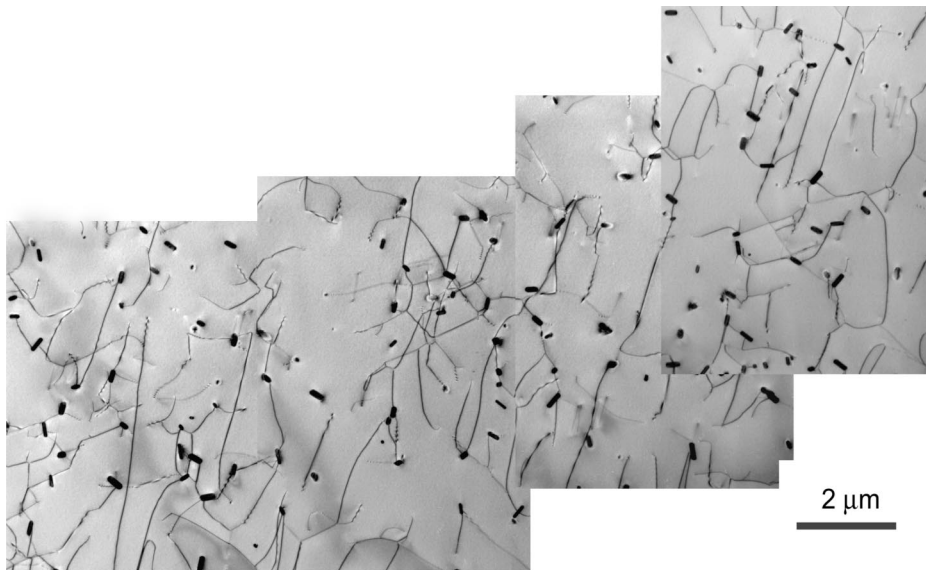


Fig. 5. Microstructure of the specimen after heat treatment at 1150°C for 2 h followed by cooling on air and creep test at 600°C.

air) and creep at 600°C is shown in Fig. 5. There are again the precipitates composed of Cr, Fe and C but the particles are smaller (100–200 nm) with higher frequency of occurrence in comparison to previous microstructure. These precipitates are nearly regularly distributed throughout the specimen without any significant preference in the case of subgrains and grain boundaries. This fact implies that the arrangement of the particles originated first and the dislocation substructure was generated afterwards during creep deformation. The dislocation substructure has homogenous character and consists of nearly straight single dislocations pinned at the particles or other dislocations.

– *The microstructure after creep at 700°C.* The microstructure of the alloy after creep test at temperature of 700°C has changed in comparison with features observed in previous two cases. Composition of the particles did not change but the distribution of the precipitates is more irregular (Fig. 6). The size of the particles ranges from 200 to 500 nm with lower frequency of occurrence. Precipitates are situated in rows at or near the subgrain boundaries. The dislocation substructure exhibits features of dynamic recovery – grains and subgrains with low density of dislocations or without dislocations inside.

– *The microstructure after creep at 900°C.* The microstructure of the sample after high temperature creep test at 900°C fully corresponds to the structure after

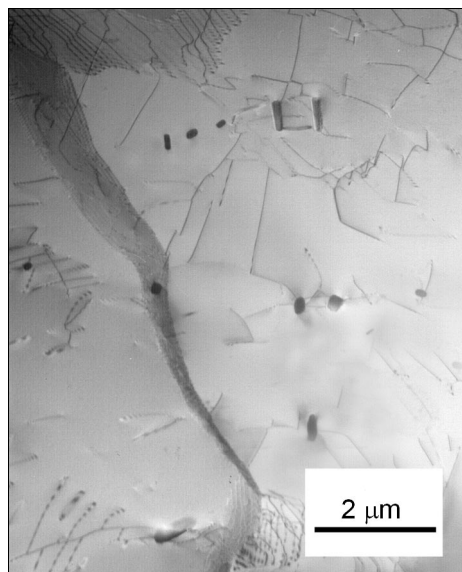


Fig. 6. Microstructure of the sample after creep test at 700 °C.

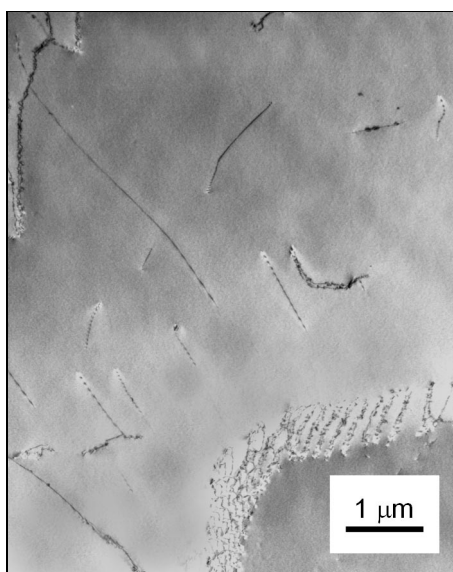


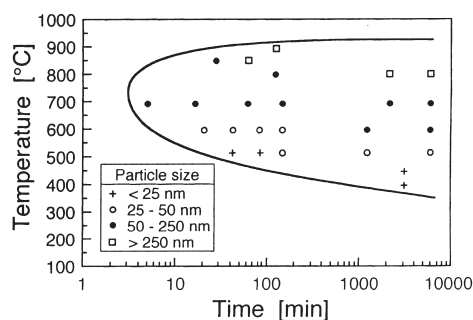
Fig. 7. Microstructure in a sample after creep test at temperature of 900 °C.

dynamic recovery and dynamic recrystallization (Fig. 7), i.e. large grains with a network of subgrain boundaries inside. The interior of subgrains is mostly without dislocations or the dislocation density is low. No particles were observed.

From micrographs (Figs. 4–7) it follows that two main features could characterize the microstructure evolution with respect to the creep test temperature. Firstly, the highest frequency of particle occurrence is observed at the temperature of 600 °C, and it decreases with increasing temperature. Secondly, the dislocation substructure changes with temperature due to dynamic recovery and dynamic recrystallization at high temperatures.

The structure analysis of particles has been recently carried out in order to determine the range of existence, symmetry and composition [14]. A schematic time-temperature precipitation diagram indicates that the quickest nucleation and growth of the precipitates occurs at temperatures ranging from 700 to 800 °C (Fig. 8). However, in our case, the temperature exposition took place under stress during creep tests and it is generally accepted that under these conditions the diffusion processes determining nucleation and growth are easier. On the basis of this fact we suppose that the nose in Fig. 7 is shifted a little towards lower temperatures. The time of exposition corresponds to the time to rupture (Fig. 2) i.e. the time of exposition decreases with increasing temperature (~ 1000 h at 600 °C and ~ 100 h at 900 °C).

Fig. 8. Schematic time-temperature precipitation diagram for Cr-Fe-C precipitates.



Development of dislocation substructure with increasing temperature could be described on the basis of dynamic recovery and dynamic recrystallization processes. At low temperature, the parts of subgrain boundary networks were observed and the dislocation density inside subgrains was higher. The dislocations are partly pinned by the particles (Fig. 4). The density of dislocations is becoming lower, subgrain boundary networks are more pronounced and the grains are larger with increasing temperature (Fig. 7). The above mentioned processes suppose the main role of lattice dislocations during creep deformation connecting with solid solution hardening and precipitation hardening. Also the values of the stress exponent n (about 4) and the activation energy of creep of $Q = 320$ kJ/mol suggest that the creep rate controlling mechanism is viscous glide of dislocations. In consequence, the interactions between alloying elements and dislocations improve the creep resistance of studied material. This finding is in agreement with results of previous investigations [5, 6, 7, 12, 13].

4. Conclusions

Description of the microstructure of Fe-28Al-3Cr (at.%) intermetallic alloy with additions of Mn, Ce and C after creep can be summarized as follows:

1. During creep the microstructure of the iron aluminide changes depends on temperature. The precipitates of CrFeC carbide form at the temperature of 600°C and their frequency of occurrence decreases with increasing temperature. No particles were observed at temperature of 900°C.
2. The dislocation substructure changes with the increasing temperature of creep deformation. The changes of dislocation structure can be described as consequence of dynamic recovery and dynamic recrystallization processes.
3. The enhancement of creep resistance is connected with solid solution and precipitation strengthening. The rate controlling mechanism during creep is a viscous glide of dislocations.

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