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The influence of thermomechanical treatment on structure of FeAl intermetallic phase - based alloys

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Materials

ABSTRACT

Purpose: The major problem restricting universal employment of intermetallic phase base alloy is their low plasticity which leads to hampering their development as construction materials. The following work concentrates on the analysis of microstructure and plasticity of ordered FeAl (B2) alloy during cold and hot deformation and rolling process.

Design/methodology/approach: After casting and annealing, alloy specimens were subjected to axial-symmetric compression in the Gleeble 3800 simulator at temperatures ranging from 800, 900 and 1000°C at $0.1s^{-1}$ strain rate. In order to analyse the processes which take place during deformation, the specimens after deformation were intensely cooled with water. The process was conducted on the K -350 quarto rolling mill used for hot rolling of flat products. The process was conducted in some stages at temperature ranging from 1200-1000°C: Structural examination was carried out using light microscopy. The examination of the substructure was carried out by transmission electron microscopy (TEM).

Findings: The research carried out enabled the understanding of the phenomena taking place during hot rolling of the investigated alloy. which has been also confirmed in plastometric studies conducted in the form of hot compression tests. The microstructure analyses applying optic and electron microscopy have revealed the structure reconstruction processes occurring in FeAl alloys during cold and hot deformation.

Practical implications: The research carried out enabled the understanding of the phenomena taking place during deformation and annealing of the investigated alloy. The obtained sheets can be used as constructional elements working in complex stress fields, at a high temperature and corrosive environments. The results will constitute the basis for modelling the structural changes.

Originality/value: The obtained results are vital for designing an effective thermo - mechanical processing technology for the investigated FeAl alloy.

Keywords: Metallic alloys; Deformation; Hot rolling; Recrystallization

1. Introduction

Intermetallic compounds particularly aluminides, are emerging as materials for high temperature structural applications. They promise to bridge the gap between the operating temperature range of structural ceramics and nickel-based superalloys [1-4]. The position of alloys representing the Fe-Al system against other constructional materials is increasingly better as the research on the capacities for their fabrication and application advances [5-7].

The iron aluminides Fe_3Al and FeAl have been among the most widely studied intermetallics because of their low cost, low density, good wear resistance, ease of fabrication and resistance to oxidation and corrosion create wide prospects for their industrial applications, for components of machines working at a high temperature and corrosive environment [8-10]. The major problem restricting their universal employment is their low plasticity and their brittle cracking susceptibility, which leads to hampering their development as construction materials. Consequently, the research of intermetallic-phase-based alloys focuses on their plasticising [11-17].

The purpose of this work is to analyse the changes in the microstructure of the ordered alloys in Fe-Al system, based on Fe_3Al and FeAl intermetallic phases, following the high-temperature plastic strain ranging from 700 to 1200°C. The microstructure analyses applying optic and electron microscopy have revealed the structure reconstruction processes occurring in FeAl alloys. It has been shown that different mechanisms of the structural changes ensue from the thermal plastic strain in the investigated alloys, which influences their technological plasticity. The displayed results may form the basis for designing the technology of plastic working of the investigated alloy.

The results will be employed to develop the technology of shaping the structure of this materials group by means of hot plastic working. The obtained sheets can be used as constructional elements working in complex stress fields, at a high temperature and corrosive environments.

2. Research methodology

2.1. Research material

Material for the research consisted of bars cast from an alloys based on an FeAl intermetallic phase of a chemical composition shown in Table 1. The alloys was prepared by casting into grafite moduls. The following contents were used for smelting: ARMCO iron, aluminium 99.98% wt. minimum, amorphous boron and technically pure molybdenum powder compact. Ingots were obtained in the form of cylinders of dimensions: ø 14 mm and 120 mm in length.

Table 1.
The chemical compositon of the investigated alloys (at,-%)

	Al	Cr	Zr	C	B	Fe
FeAl	38.00	-	0.05	0.10	0.01	61.64

2.2. Compression test

After the casting, the samples were anealling at 1000°C for 48h with furnace cooling. Following the annealing, the material was used for the preparation of the samples for the compression test, which consisted of axisymmetrical strain on Gleeble 3800 simulator, with simultaneous structure-freeze by rapid quenching. The samples were cylindrical, measuring ϕ=10 mm and h=12 mm. The compression samples were conducted at temperatures ranging from 600-1200°C at a strain rate 0.01, 0.1, 1 and 10 s^{-1}, until the true strain values reached ε=1.0. The compression test results, such as the sample temperatures T [°C], stresses σ [MPa], forces [N] and strains ε, processed with the calculation sheet, provided the means for determining the flow curves in the stress σ - strain ε system. The methodology was described in papers [18-20].

The strain conditions, including the temperature and the strain rate for the r alloy were described with Zener-Hollomon parameter:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \qquad (1)$$

where:

- $\dot{\varepsilon}$ – strain rate, [s^{-1}];
- T – deformation temperature, [K];
- Q – activation energy of the plastic deformation process, [kJ/mol];
- R – molar gas constant [J/molK].

The activation energy for FeAl alloy was determined with ENERGY computer program using of data basecontaining stress values σ and and strain values ε, which had previously been extracted from the flow curves.

The cold compression tests was conducted in friction-free conditions, using teflon pads. The tests were performed on rolled specimens of an external diameter ϕ10 mm and height of 15 mm. For the evaluation of the nature of fracture, the specimens were also compressed until failure.

Based on the tests made, the real rolling reduction ε_h and the mean unit pressure, p_{mean} [MPa] were determined. After compression until strain ε = 0.4, the specimens were subjected to recrystallization annealing at a temperature of 600, 700, 800 and 900°C, with 1-hour soaking time. Measurement of microhardness was made in a "ZWICK" hardness testing machine by HV method, applying load of 200 g.

2.3. Hot rolling process

Cast specimens, 20 mm in height and 40 mm in width, were used as an initial material for the hot rolling process . The process was conducted at the Institute of Modelling and Control of Forming Processes in the Czech Republic on the K 350 quarto rolling mill used for hot rolling of flat products [21]. The process was conducted in the following stages:

- preliminary rolling at a temperature of 1200°C with the use of ferritic steel spacers separating cold rollers from the rolled material, with a 15% draft in each pass;
- rolling at a temperature of 1100°C in a ferritic steel shield separating cold rollers from the rolled material, with a 25% draft in each pass drafts and interoperation annealing;
- finishing rolling, forming the microstructure at a temperature of 1000°C, with applying 25% reverse drafts and interoperational annealing and cooling in oil after the last pass;
- annealing for 30 minutes at 800°C.

The calculated strain rate during rolling amounted to 10 s^{-1}.

2.4. Structure investigation

The metallographic investigation was performed on a light microscope in the range of magnifications 100-1000×.

The examination of the substructure was carried out by transmission electron microscopy (TEM) using a JEOL 100B transmission microscope operated at an accelerating voltage of 100 kV observation of fractures was carried out on a scanning Hitachi S-400 microscope.

3. Results

3.1. Flow curves and structure

The investigated alloys, based on Fe_3Al and FeAl intermetallic phases, having been cast (the diameter of the ingot: ϕ=12 mm, its height: 150 mm) and hot-worked, had coarse-grained one-phase structure, with diversified grain size (Fig. 1). Within the grains, as well as on their borders, dispersive particles of carbides and borides have been observed. The FeAl alloy is characterised by the size of the grains, whose average surface of the plane section equals $\bar{A}$=12600 μm^2 (d=115 μm) alloy and the variation index equalled ν($\bar{A}$)=152% [2-3].

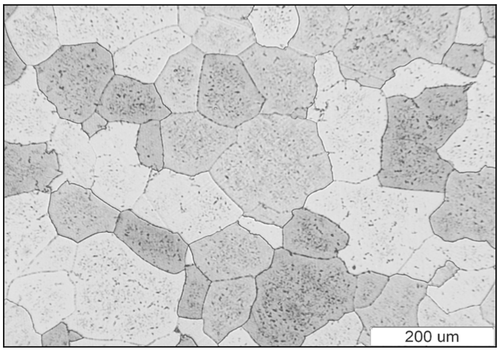

Fig. 1. Microstructure of the FeAl alloy after casting and annealing at 1000°C for 48 h, followed by cooling with furnace

The flow curves illustrating the influence of temperature and strain rate on the plasticity stress change as a function of the deformation of the investigated alloy during the hot compression were presented in Figure 2 and 3. The results indicate that the increase in the strain temperature ranging from 600-1200°C causes radical reduction in the peak stress σ_p, as well as the decrease in the corresponding strain ε_p.

The growth of the strain rate from 0.01 to 10 s^{-1} leads to the higher maximal plasticity stress σ_{pp}, which results in the increase of the corresponding strain ε_p, this refers to strain rates ranging from 0.01 to 1 s^{-1} (Fig. 2). Further increase of the strain rate up to 10 s^{-1} leads to the decrease in the ε_p value.

Deformation with parameters: 600°C / 0.1 s^{-1} and 900°C/10 s^{-1} results in a loss of material cohesion - initially in the external zone of the barrel and then, towards the internal zone.

The technological plasticity indicators of the investigated alloy calculated on the basis of the plastometric torsion results are presented in Table 2. The activation energy for the hot deformation process of FeAl alloy was found to be equal 484.9 kJ/mol.

Correlations between parameters of the deformation process and mechanical properties defined with the plastometric test, are presented by Equations 2 and 3. They indicate that the influence of the Zener-Hollomon parameter on the peak flow stress σ_{pp} for FeAl can be presented in the form of a power function (Eq. 2):

$$\sigma_{pp} = 0.002 \times Z^{0.140} \qquad (2)$$

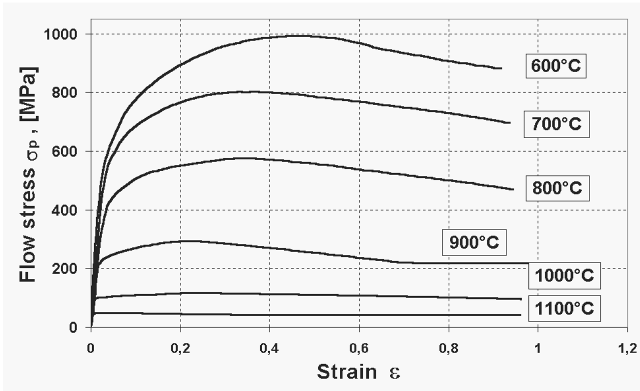

Fig. 2. Stress strain curves of FeAl alloy deformed at temperature 600 - 1200°C with a rate 0.1 s^{-1}

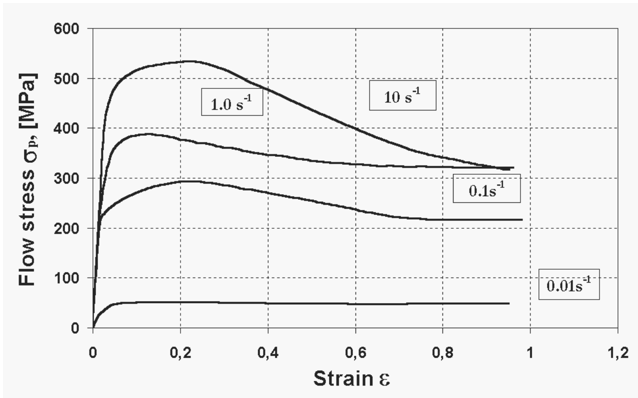

Fig. 3. The stress-strain curves of FeAl alloy deformed at the temperature 900°C with a rate 0.01-10 s^{-1}

The relation between the strain ε_p (deformation to maximum of peak flow stress) and the Zener-Hollomon parameter is expressed by a power function (Eq. 3).

$$\varepsilon_p = 0.024 \times Z^{0.195} \qquad (3)$$

The microstructures of FeAl alloy observed after heat deformation are presented in Figs. 4-7. The conducted hot deformation enabled the assessment of the microstructure of the investigated alloy, whose primary structure had been deformed and whose primary grains had been elongated. After compression at 600°C and 700°C in whole range of deformation rate the microstructure consists of deformed, elongated primary grain (Fig. 4).

The strain of FeAl alloy at the temperatures of 800°C and 900°C results in the initiation of the dynamic recrystallization process, which is confirmed by the appearance of the chains of fine grains on the boundaries of the primary grains and the slip bands (Fig. 5). The clear intensification effect of the recrystallization process has been observed at the temperature of 900°C, as indicated by the singinficant area taken by the recrystallized grains on the boundaries of the primary grains as well as within these grains.

Table. 2.
The indicators which characterising the technological plasticity of FeAl alloy

Deformation temperature [°C]	Strain rate $\dot{\varepsilon}$ [s^{-1}]	Peak stress σ_{pp} [MPa]	Strain ε_p	Zener-Holloman Parameter Z [s^{-1}]
600	0.1	980	0.57	1.03×10^{28}
700	0.1	800	0.31	1.07×10^{25}
800	0.01	431	0.19	4.04×10^{21}
	0.1	558	0.23	4.04×10^{22}
900	0.01	186	0.18	3.92×10^{19}
	0.1	291	0.26	3.92×10^{20}
	1.0	388	0.13	3.92×10^{21}
	10	534	0.21	3.92×10^{22}
1000	0.01	59	0.06	7.90×10^{17}
	0.1	109	0.09	7.90×10^{18}
	1.0	193	0.08	7.90×10^{19}
	10	299	0,13	7.90×10^{20}
1100	0.1	109	0.09	2.80×10^{17}
	1.0	87	0.03	2.80×10^{18}
	10	161	0.07	2.80×10^{19}
1200	1.0	38	0.03	1.57×10^{17}
	10	75	0.04	1.57×10^{18}

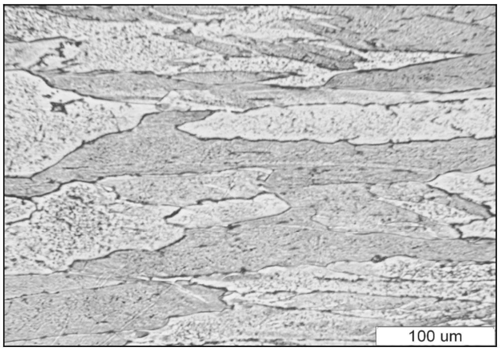

Fig. 4. Microstructure of the FeAl alloy after compression at 600°C with rate 0.1 s^{-1}. Deformation ε = 0.1

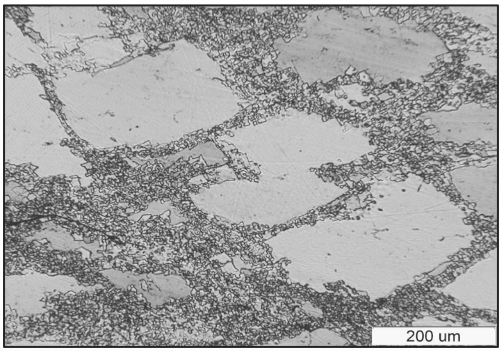

Fig. 5. The microstructure of the FeAl alloy following the thermal plastic strain at 800°C at the rate of 0.1 s^{-1}

In the substructure, the nuclei of recrystallization are formed, which spread towards the areas with very high dislocation density (Fig. 6). The upsetting of the strained alloy at 1000°C leads to the recrystallization of the structure, which is formed of finely shaped subgrains (Fig. 7).

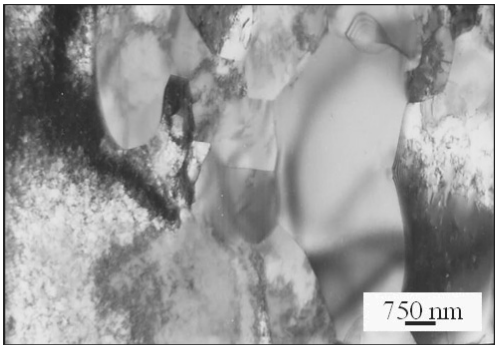

Fig. 6. The substructure of the FeAl alloy following the thermal plastic strain at 900°C at the rate of 0.1 s^{-1}

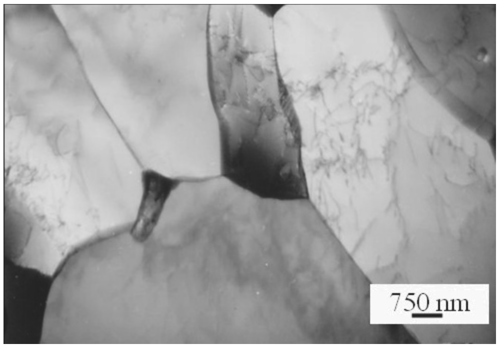

Fig. 7. The microstructure of the FeAl alloy followingthe thermal plastic strain at 1000°C at the rate of 0.1 s^{-1}

3.2. Hot rolling

Microstructures of the cast and heat treated alloys are shown in Fig. 8. The alloy composed of Fe-38% Al with an intermetallic FeAl phase matrix had a single-phase, coarse-grained structure.

The axisymmetrical hot compression tests made for the studied alloy determining the flow curves in the stress-deformation system (Fig. 9). A wide range of deformation strengthening with a diversified course was identified, depending on the temperature. The obtained compression tests results show that an increase of the deformation temperature in the range of 900-1200°C entails an intensive decrease in the maximum yield stress σ_p, causing at the same time a decrease in the corresponding strain ε_p, which, in consequence, leads to fast strengthening of the alloys. The alloy samples' compression at a temperature of 900°C and strain rate of 10 s^{-1} to a strain ε = 1 led to the sample's cracking on the lateral side of the barrel being formed for the alloy with a higher aluminium concentration.

Based on the structural studies carried out [2], the processes taking place in the orderly alloys' structure during high-temperature deformation were detected. The determination of the

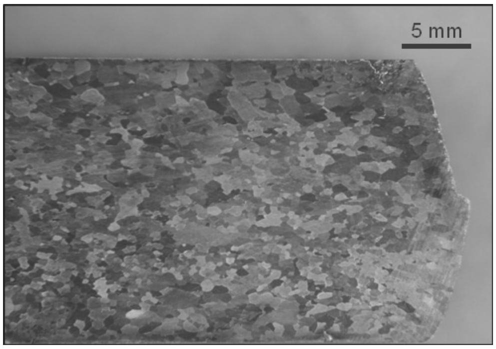

Fig. 8. Cross section of the ingot with FeAl alloy after casting (20 mm in height / 40 mm in width)

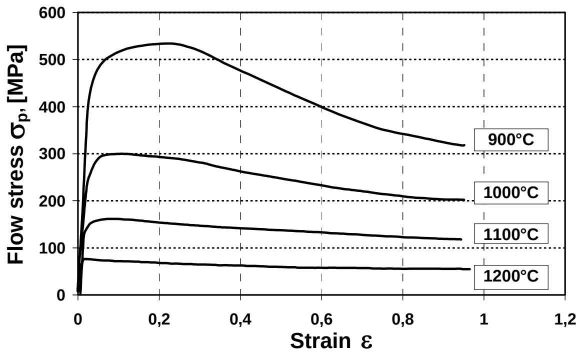

Fig. 9. The stress-strain curves of Fe-38%Al alloy deformed at the strain rate 10 s^{-1}

technological plasticity as well as structural studies enabled selecting the optimal rolling conditions.

Rolling on the quarto rolling mill proceeded in several stages. Both the studied alloys were subjected to the first stage of rolling, during which ferritic steel spacers were used. The application of the spacers was indispensable since first trials demonstrated a grid of cracks on the surface, even in the case of minor deformations (Fig. 10).

The spacers separated cold rollers from the alloys' surface. The first stage in a temperature range of 1200-1000°C, with applying two reverse drafts, enabled reducing the height by 50% (10 mm) 10 s^{-1}. At this stage of rolling, no cracks were identified on the external surface of the sheet.

Further deformation was performed using a complete shield made of ferritic steel sheets. The application of a lower process temperature (1100-1000°C) caused material cracking on its lateral surfaces, while longitudinal surfaces, protected by the shield, were free of any defects. The strips rolled at 1100°C with a 50% total draft (5 mm sheet) are shown in Figure 5. The effect of this stage of rolling for both the studied alloys were microstructural changes in the form of grain size reduction caused by the recrystallization process (Fig. 11).

Next deformation was performed at a temperature of 1000°C, with reducing the height to 2.5 mm. A flat bar of the FeAl alloy was subjected to further deformation at a temperature of 1000°C to a thickness of 2 mm. Consequently, a further grain size reduction was obtained.

The finishing rolling of the alloy forming the microstructure, was conducted at a temperature of 1000°C using 25% reverse drafts and interoperation annealing. The obtained final sheets (Fig. 12), 1 mm in thickness.

After rolling and recrystallization annealing in 800°C per one hour structure characterized by a fine-grain with average size 56 µm (Fig. 13).

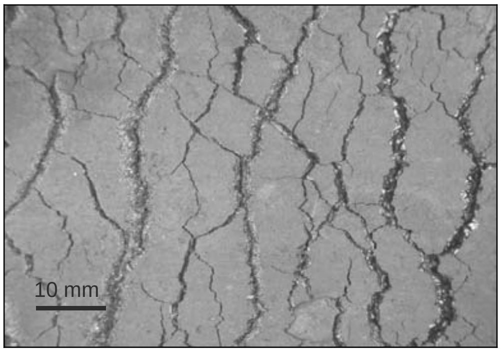

Fig. 10. A grid of cracks on the surface of hot rolled alloy at a temperature of 1200-1100°C at alloy without spacers

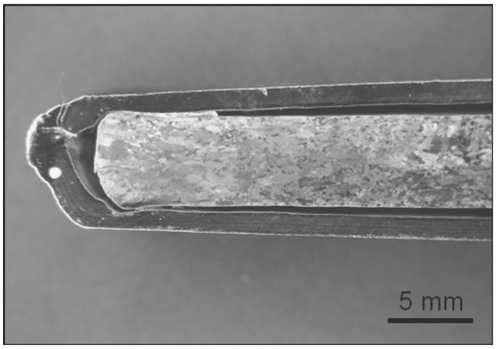

Fig. 11. Cross section of the flat with Fe-42% at. Al alloy after rolling with ferritic steel shield at temperature 1100-1000°C

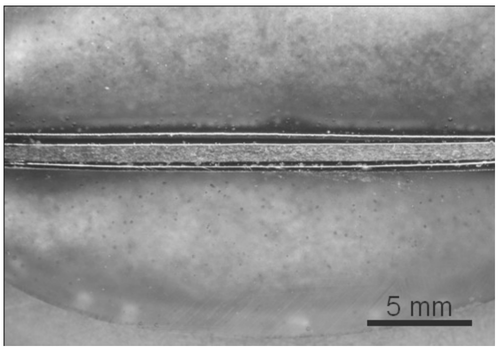

Fig. 12. Cross section of the flat with FeAl alloy after rolling with ferritic steel shield at temperature 1000°C

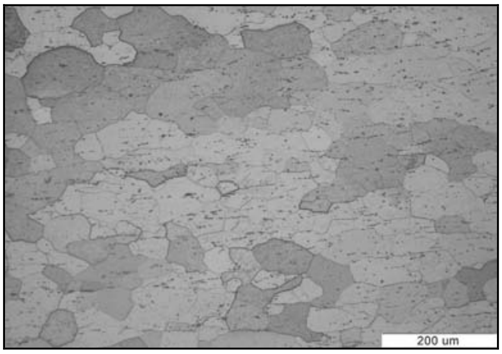

Fig. 13. Structure of the sample from Fe-38%at.Al alloy after rolling at 1000°C with annealing at 800°C per one hour

3.3. Structure after cold deformation and recrystallization annealing

The tested alloy is characterized by intensive consolidation during the cold compression test to ca. 1800 MPa at real draft of 0.4. Further deformation results in a loss of material cohesion - initially, in the external zone of the barrel and then, towards the internal zone.

Structure of the FeAl alloy specimens after cold compression at strain of ε = 0.4 and after annealing in the temperature range of 600-900°C, with soaking time of 60 min is shown in Figs. 14-17. The structure consists of deformed grain with visible slip bands (Fig. 14). In the substructure, wide slip bands and a large amount of structural defects were detected.

Intensiveness of the recrystallization process depends on the annealing temperature applied. After annealing at 600°C, no new recrystallized grain is observed in the microstructure (Fig. 15).

Some fine, recrystallized grain, near primary grain boundaries, are detectable after annealing at a temperature of 700°C (Fig. 16). Increaing the annealing temperature to 800°C intensifies recrystallization (Fig. 17). The structure consists of fine recrystallized grain with a diamater of d=20 µm (mean equivalent diameter of the grain plane section). A further increase in the annealing temperature leads to a gradual grian growth after recrystallization, up to a mean size of 80 µm (Fig. 18).

Results of microhardness measurement in the initial state after cold deformatio and after recrystallization annealing are shown in Table 3. After cold compression, a significant increase of microhardness, by 240 µHV, took place, compared to the alloy after homogenization annealing at 1000°C with soaking time of 24 h, which testifies to considerable consolidation of the material.

After recrystallization annealing, a gradual decrease of microhardness is observed. Minimal microhardness is characteristic of specimens after annealing at a temperature of 800°C. A further increase of the annealing temperature results in an insignificant increase in microhardness.

Structure of the alloy specimens deformed until failure is presented in Figure 18. In some places, so-called ″leaf″ cleavage fracture and so-called “high-energy“ cleavage fracture were observed (Fig. 19).

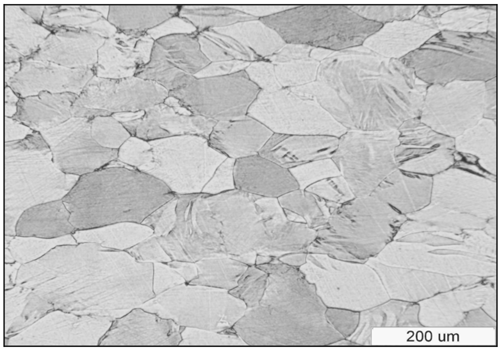

Fig. 14. Structure of the FeAl alloy after cold compression, real draft ε=0.4

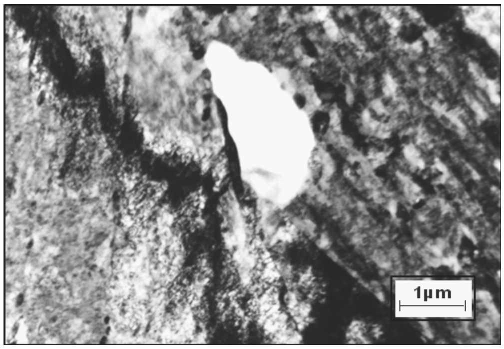

Fig. 15. Subructure of the FeAl alloy after cold compression and annealing at 600°C

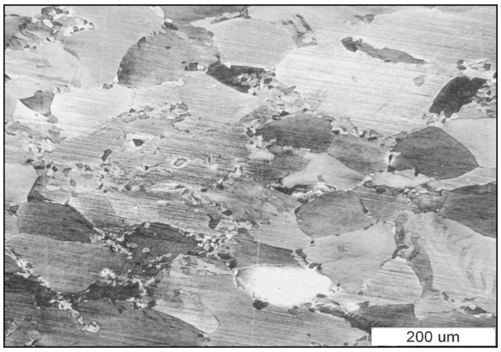

Fig. 16. Structure of the FeAl alloy after cold compression and annealing at 700°C

Table 3.
Results of microhardness measurement of the investigated alloy

	Microhardness, HV
after homogenization	300
after rolling	370
cold compression	540
annealing 600°C	420
annealing 700°C	350
annealing 800°C	340
annealing 900°C	345

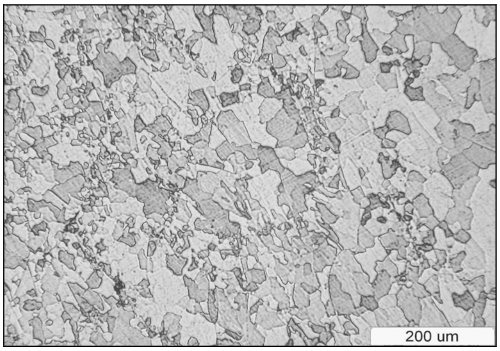

Fig. 17. Structure of the FeAl alloy after cold compression and annealingat 800°C

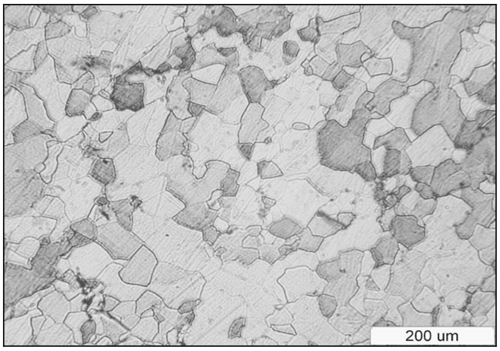

Fig. 18. Structure of the FeAl alloy after cold compression and annealing at 900°C

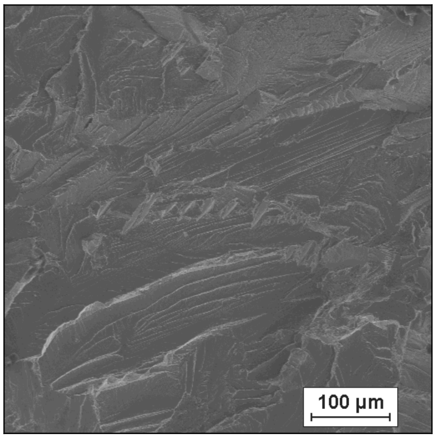

Fig. 19. Fracture of a FeAl alloy specimen deformed until failure

4. Conclusions

In their initial state (and following the introductory heat treatment), the FeAl intermetalic-phase-based alloys had coarse-grained single-phase structure with diversified grain size. A significant influence of the chemical content on the structure reconstruction processes occurring during thermal plastic strain in the investigated alloys in the FeAl system. The former determines the plasticising stress, a number of times higher for the FeAl alloy. In the alloy, fine recrystallised grains forming on the boundaries of the primary grains and slip bands are perceptible in the microstructure, and their number increases with the rise in the process temperature. A considerable increase in the defect density and the nuclei of new grains, growing towards the defected areas, have been noted to appear in the substructure. The high level of defectation led to the high plasticising strain level. A started of recrystallization process takes place at a temperature of 800°C.

In alloy specimen deformed at 900°C and higher temperatures, after exceeding strain ε_p, the stress becomes steady at σ_s. After deformation at 1100-1200°C the structure is completely recrystallized and the effect new grain growth becomes visible.

The hot rolling have shown that it is possible to form the alloys through thermomechanical processing only where ferritic steel shields of appropriate thickness are applied. Rolling of the alloys without shields led to the occurrence of a grid of cracks. An alloy with a lower aluminium concentration can be plastically formed at a temperature of up to 1000°C, which has been also confirmed in plastometric studies conducted in the form of hot compression tests. Thus, there seems to be a capacity for improvement of the studied alloy's properties by means of thermoplastic treatment.

The obtained 1 mm thick sheets can be used as constructional elements working in complex stress fields, at a high temperature and corrosive environments.

The structure after cold deformation consists of deformed grain. High density of defects was detected in the substructure. After annealing of the cold deformed specimens, static recrystallization is observed. At temperatures of 600 and 700°C, recrystallization proceeds at low intensity. Fine recrystallized grain is observed at primary grain boundaries as well as recrystallized grain nuclei in the substructure. Optimal annealing conditions in the investigated range of parameters‘ variability, as regards the structure (grain size d=20 µm) and the lowest micro-hardness, are: temperature of 800°C and 1h soaking time. A further increase of the recrystallization temperature induces a growth of new grain.

The research carried out enabled the understanding of the phenomena taking place during hot rolling of the investigated alloy, which has been also confirmed in plastometric studies conducted in the form of hot compression tests. The microstructure analyses applying optic and electron microscopy have revealed the structure reconstruction processes occurring in FeAl alloys during cold and hot deformation.

The results obtained are vital for designing an effective thermo - mechanical processing technology for the investigated FeAl alloy.

Acknowledgements

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