M. JABŁOŃSKA, I. BEDNARCZYK, K. RODAK, A. ŚMIGLEWICZ

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STUDY OF THE STRUCTURE OF INTERMETALICS FROM Fe - Al SYSTEM AFTER THE HOT ROLLING

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This paper presents the results of structure analysis of Fe - Al alloys after hot rolling deformation. Microstructure analysis were performed before and after deformation using a scanning transmission electron microscopy (STEM) technique. The detailed quantities research of the structures was conducted using scanning electron microscopy (SEM) equipped with the gun with cold field emission and the detector of electron back scattering diffraction (EBSD).

Key words: Fe - Al intermetallics, hot rolling process, substructure, microscopy, SEM/EBSD method

INTRODUCTION

The numerous investigations during the last decade were focused on the research for a new generation of materials. The most attractive advantage is that Fe - Al alloys exhibit excellent mechanical properties and corrosion resistance. More attention was paid to reduce of cost in industrial production, in order to increase of profitability [1 - 5]. Intermetallics are considered as the next generation of materials for use in high temperature. These alloys combine some characteristics typical for metal (deformability, i.e. a high range of plasticity) and for ceramics (strength, structural stability at high temperature, resistance to aggressive environments). Some of these properties can be controlled by heat treatment processes because they have a high sensitivity to the vacancies hardening [4, 6 - 8, 9, 10]. Intermetallics functional advantages make them one of the most attractive materials. Reduce in cost production should to gain more and more interest in Poland [2, 3, 6, 8, 11]. The development of this group of materials and implementation for industrial production and use as structural materials are dependent on improvement of casting properties, plasticity at room temperature [2, 4 - 8] as well as phase identification and their impacts during deformation [12]. For hot workability studies, very important is identification of safe processing domains for as - cast binary and ternary (Cr and Mn) alloys by using the processing maps [11, 14 - 18]. Production of semi-finished and finished products from intermetallic alloys requires the use of appropriate methods for their preparation, hot working and machining. The process of plastic deformation of Fe - Al alloys belongs to a quite complicated [2, 13 - 15]. This is due, inter alia, ordering of the structure, distribution of superdislocations and change in the slip direction [1, 4, 5, 9]. In addition, limited tendency to fragmentation of particles during the process of hot plastic deformation. The recommended area for hot forming of Fe - Al alloys occurs at 850 °C - 1 050 °C at strain rate of less than 0,1 s^{-1} and at 950 °C - 1 100 °C for up to 10 s^{-1} [18]. The literature does not provide sufficient information about the effects of hot plastic deformation parameters on the structure and substructure. For this reason more experimental work is apparently needed to detailed clarify and description of structural phenomena during hot plastic deformation. The results obtained from the study of technological plasticity and the analysis of changes in structure, occurring during hot deformation, can complement of existing knowledge about Fe - Al alloys. This paper presents an analysis of changes in the structure of the Fe - Al alloys after hot rolling process using X - ray diffraction patterns - XRD, scanning electron microscopy - SEM and scanning transmission electron microscopy - STEM techniques.

M. Jabłońska, I. Bednarczyk, K. Rodak, A. Śmiglewicz Silesian University of Technology, Faculty of Materials Science and Metallurgy, Institute of Materials Science Katowice, Poland

TEST MATERIALS AND METHODS

The research was carried out on alloys from Fe - Al system. The chemical compositions is given inTable 1.

Table 1 **Chemical composition of FeAl28Cr5 and FeAl38 alloys / wt. %**

Alloy	Chemical composition / wt.%		
	Al	Cr	Fe
FeAl28Cr5	15,88	5,47	78,65
FeAl38	22,85	-	77,15

The alloys are made by casting in a vacuum induction furnace. The ingots after casting process were subjected to heat by homogenization in temperature of 900 °C for 24 h with cooling in furnace. Hot rolling was carried out at a temperature of 1 200˚C in three passes. The rolling speed was 80 rpm. After rolling the samples were cooled in air. X - ray diffraction patterns were collected using X - ray Philips diffractometer equipped with graphite monochromator. Structural investigations were performed by SEM as well as STEM technique, using the thin foils. On the basis of the SEM / EBSD method the average equivalent diameter of the grains and distribution of grains were determined. The boundary between the grain and subgrain was determined on the basis of the misorientation angle measurement. Microstructure observation were carried out after heat treatment and after the hot rolling process.

RESULTS AND DISCUSSION

X - ray diffraction patterns for FeAl28Cr5 and FeAl38 after heat treatment at 900 °C for 24 h with cooling in furnace, were presented in Figures 1, 2. The presence of FeAl phase (B2 type structure) is clearly seen for both alloys.

The Fe_3Al phase (DO3 type structure) has not been presented. This means that it may be caused by applying cooling rate of 3 °C / min, which may be insufficiently slow to produce DO3 ordering, which is characteristic for alloys with these aluminum content at room temperature. In order to maintain equilibrium DO3 ordering is needed cooling rate of about 1 °C / min. An example of the microstructures of the tested alloys after heat treatment are shown in Figures 3, 4. In analyzed

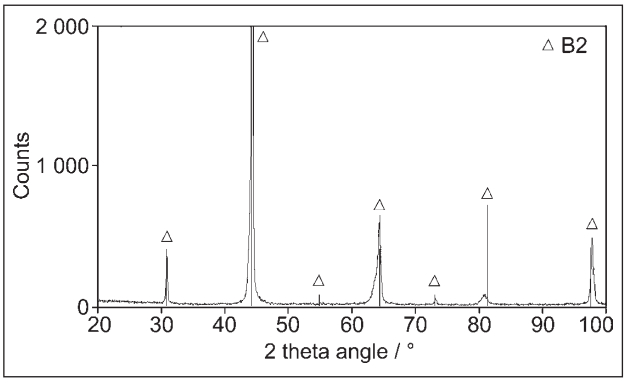


Figure 1 Diffraction pattern of FeAl28Cr5 alloy after heat treatment at temperature 900 °C for 24 h

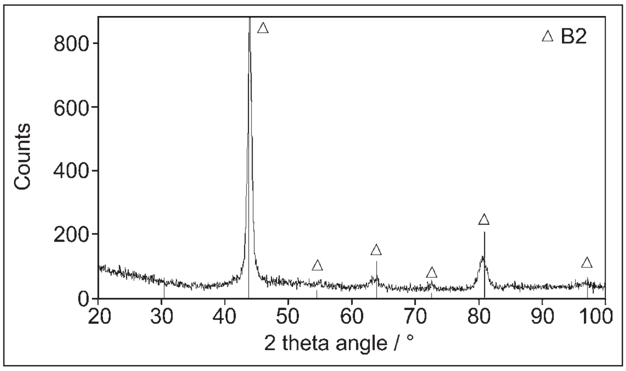


Figure 2 Diffraction pattern of FeAl38 alloy after heat treatment at temperature 900 °C for 24 h

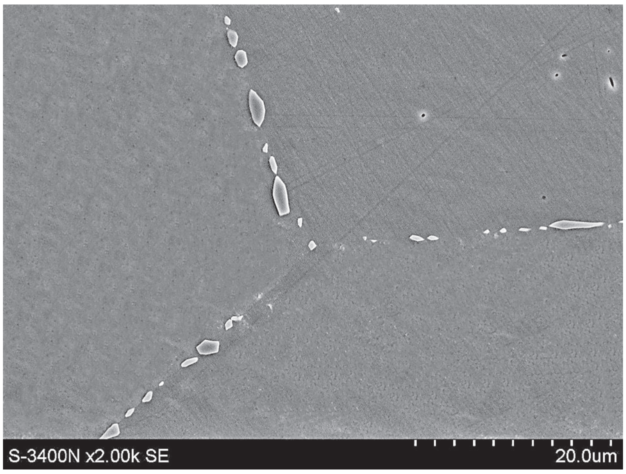


Figure 3 Microstructure FeAl28Cr5 of after annealing at temperature 900 °C for 24 h - SEM, phases on the grains boundaries

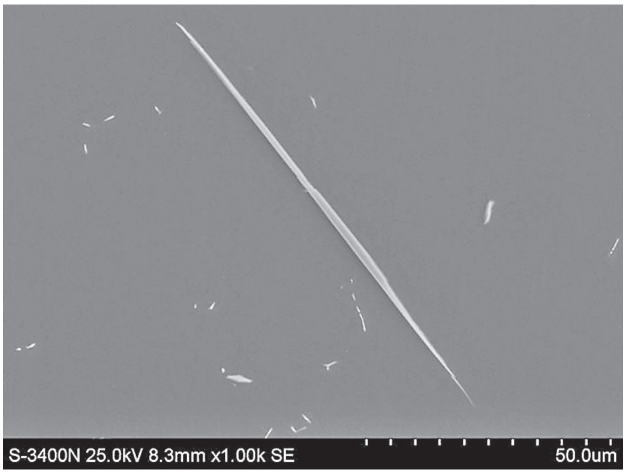
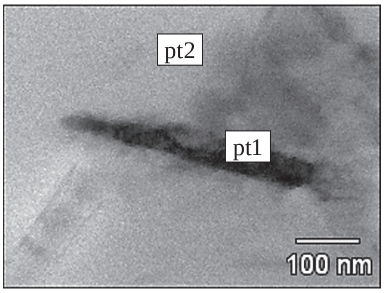


Figure 4 Microstructure FeAl38 of after annealing at temperature 900 °C for 24 h - SEM, phases elongated in shape

alloys, were observed phases with different shapes. In FeAl28Cr5 alloy phases were revealed mainly at grain boundaries. Chemical composition of these phases, showed that particles are enriched in chromium content (50 % at. - 75 % at. Cr) (Figure 5). In the case of the alloy FeAl38 chemical composition of particles performed by using SEM technique revealed presence of phases with chemical composition similar to the chemical composition of the matrix (Figure 6).

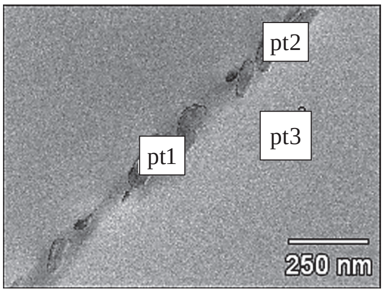
Qualitative analysis of chemical composition performed for phases by STEM method, confirmed that the phases were enriched in iron and aluminum. Because the distributions of elements in the areas examined by XRD revealed the presence of carbon, additional chemical analysis was performed to confirm the presence of this element in the FeAl38 alloy, in order to verify the hypothesis about the possibility in occurrence of carbide $AlFe_3C$ type. Carbon concentration was determined by measuring the absorption of infrared radiation (HFIR), which was amounted to 0,035 wt. %. In the Fe - Al alloys, of such a carbon content was not been observed in analyzed phases.

Has been also performed microstructure analysis using EBSD technique. The aim of this study was the



/ wt. %	Al	Cr	Fe
FeAl28Cr5 pt1	1,5	31,0	67,5
FeAl28Cr5 pt2	2,1	4.2	93,7
/ at. %	Al	Cr	Fe
FeAl28Cr5 pt1	3,0	32,0	65,0
FeAl28Cr5 pt2	4,2	4,4	91,3

Figure 5 Chemical composition of particles and matrix in FeAl285Cr alloy after heat treatment at temperature 900 °C for 24 h



/ wt. %	Al	Fe
FeAl38 pt1	23,9	76,1
FeAl38 pt2	23,2	76,8
FeAl38 pt3	24,4	74,6
/ at. %	Al	Fe
FeAl38 pt1	39,4	60,6
FeAl38 pt2	38,5	61,5
FeAl38 pt3	41,4	58,6

Figure 6 Chemical composition of particles and matrix in FeAl38 alloy after heat treatment at temperature 900 °C for 24 h

analysis of influence of heat treatment and hot deformation parameters on the distribution of grains / subgrains and misorientation angles between the grains / subgrains. In order to reveal the structural changes, which take place under the influence of hot plastic deformation, a study of the microstructure and substructure were performed. Figures 7, 8 shows the microstructure of FeAl28Cr5 and FeAl38 alloy after hot rolling process. In the microstructure of the FeAl28Cr5 alloy, the defragmentation process inside the primary grains was observed. Inside the primary grains formation of new grain / subgrain were found. In FeAl38 alloy were observed equiaxed grains with different shapes (Figure 8).

The microstructure maps obtained by EBSD technique for FeAl28Cr5 and FeAl38 alloys have been show in Figures 9, 10. For FeAl28Cr5 alloy there are grains having an average diameter of 500 μm. The grain / subgrains with an average diameter 1 000 - 1 100 μm in range were most observed in analyzed surface. There

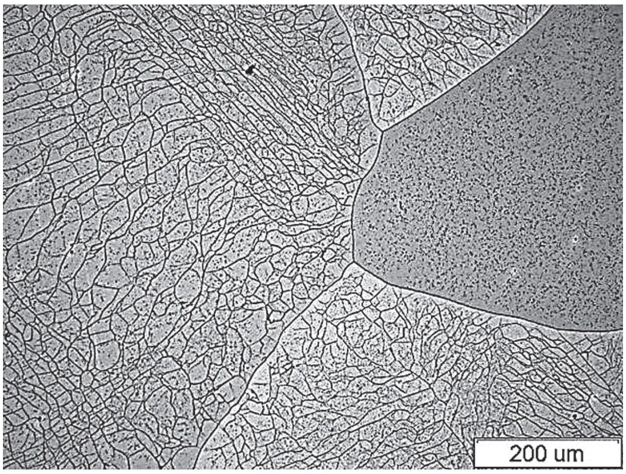


Figure 7 Microstructure of FeAl28Cr5 alloy after hot rolling process

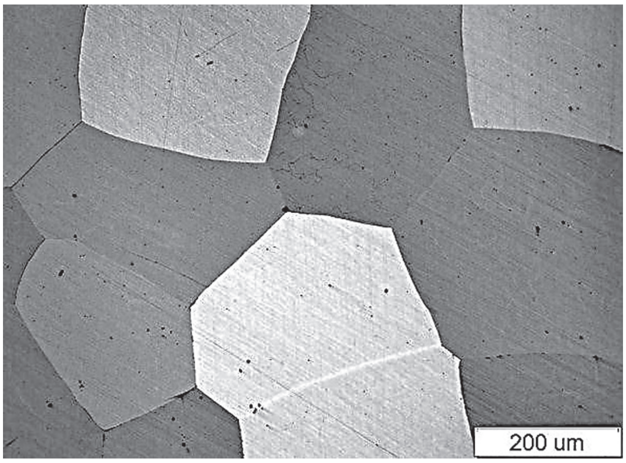


Figure 8 Microstructure of FeAl38 alloy after hot rolling process

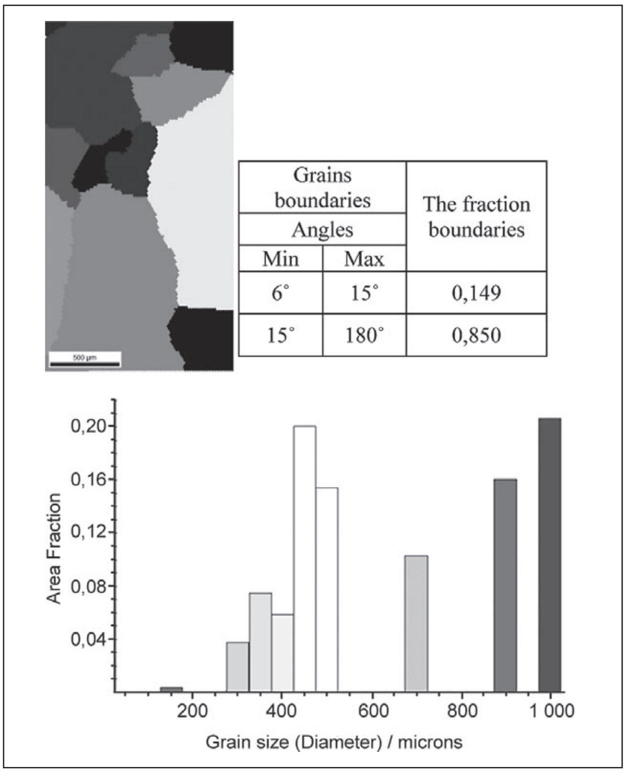


Figure 9 EBSD map, boundaries incidence and grain size distribution for FeAl28Cr5 after heat treatment and hot rolling

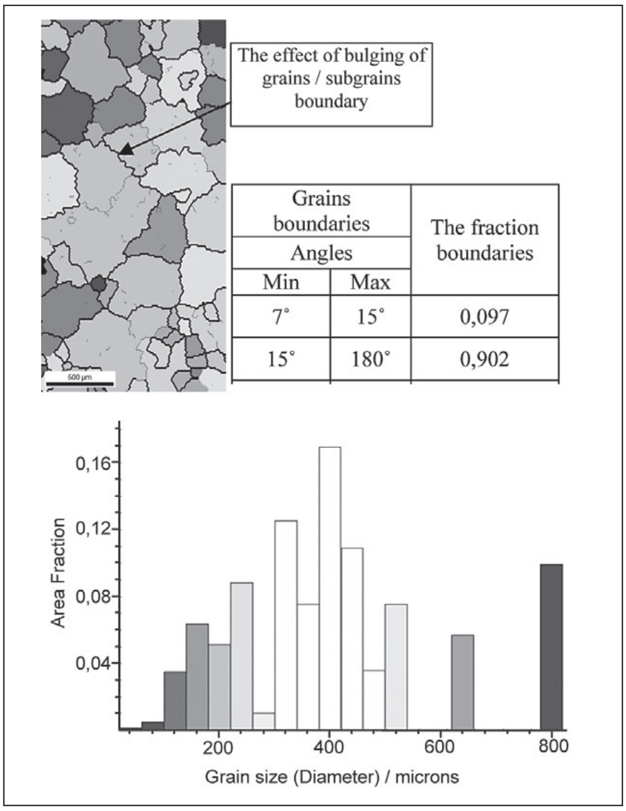


Figure 10 EBSD map, boundaries incidence and grain size distribution for FeAl38 after heat treatment and hot rolling

was a big fraction of grains / subgrains with misorientation angle above 15˚ (Figure 9). Hot rolling process performed for FeAl38 alloy results in formation of fine equiaxed grains / subgrains. The fraction of high angle boundaries is approximately about 90 % The grain size distribution of FeAl38 alloy and FeAl28Cr5 alloy is shown in Figures 9, 10. It can be noted that deformation process performed for FeAl38 alloy resulted in significant refinement structure. Figures 11, 12 shows a comparison of the microstructure using STEM analysis for FeAl28Cr5 and FeAl38 alloys after hot rolling process. STEM analysis confirmed the presence of secondary phases located in the privileged places as subgrain boundaries. These phases have different shape and different size.

CONCLUSIONS

Very often in the case of Fe - Al intermetallics we talk about the possibility of complex thermo mechanical processing. The behavior of these alloys during hot plastic deformation is primarily affected by the content of aluminum, which determines the plastic flow. Studied in this article alloys, subjected to annealing in the given parameters show the presence of precipitates existing especially at the grain boundaries and in the interior of grain. For FeAl28Cr5 alloy were revealed a high chromium phases. For FeAl38 alloy, were observed phases with chemical composition similar to the matrix. Performed researches for alloys indicated, that the rolling process cause formation of new grains and subgrains reflecting the processes of recovery and recrystallization. The microstructure of FeAl38 alloy revealed characteristic phenomenon grain "serration". This phenomenon indicates the geometric recrystallization process. In microstructure the bulging of grain boundaries / subgrains with misorientation angle above 15˚ were evident observed.

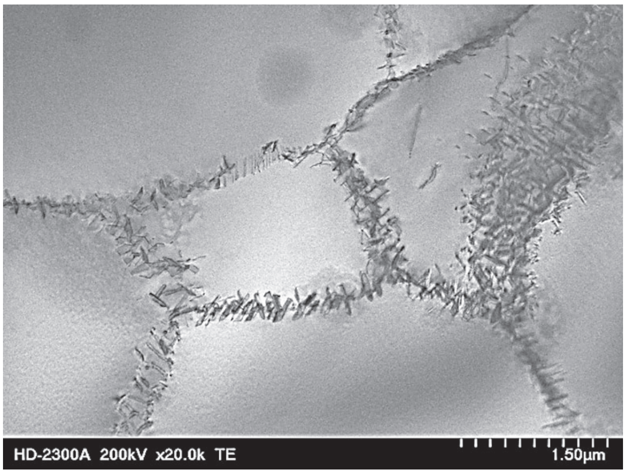


Figure 11 Substructure of FeAl28Cr5 alloy after hot rolling process. Secondary phases around the grains/ subgrains

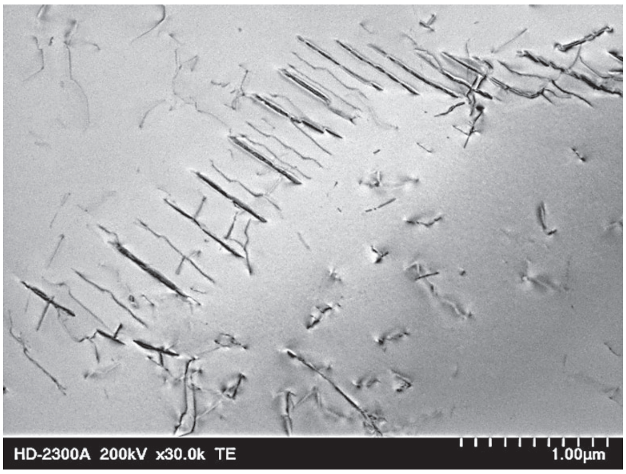


Figure 12 Substructure of FeAl38 alloy after hot rolling process. Dislocations and secondary phases

Acknowledgments

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