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INFLUENCE OF CUMULATIVE PLASTIC DEFORMATION ON MICROSTRUCTURE OF THE FeAl INTERMETALLIC PHASE BASE ALLOY

WPŁYW SKUMULOWANEGO ODKSZTAŁCENIA PLASTYCZNEGO NA STRUKTURĘ STOPÓW Fe-Al NA OSNOWIE FAZ MIĘDZYMETALICZNYCH

This article is part of the research on the microstructural phenomena that take place during hot deformation of intermetallic phase-based alloy. The research aims at design an effective thermo - mechanical processing technology for the investigated intermetallic alloy. The iron aluminides FeAl have been among the most widely studied intermetallics because their low cost, low density, good wear resistance, easy of fabrication and resistance to oxidation and corrosion. There advantages create wide prospects for their industrial applications for components of machines working at a high temperature and in corrosive environment. The problem restricting their application is their low plasticity and their brittle cracking susceptibility, hampers their development as construction materials. Consequently, the research of intermetallic-phase-based alloys focuses on improvement their plasticity by hot working proceses. The study addresses the influence of deformation parameters on the structure of an Fe38% at. Al alloy with Zr, B Mo and C microadditions, using multi – axis deformation simulator. The influence of deformation parameters on microstructure and substructure was determined. It was revealed that application of cumulative plastic deformation method causes intensive reduction of grain size in FeAl phase base alloy.

Keywords: FeAl alloys, Max Strain simulator, cumulative plastic deformation, microstructure

Prezentowana praca stanowi element prowadzonych badań nad możliwością kształtowania stopów na osnowie faz międzymetalicznych z układu Fe-Al drogą obróbki cieplno-plastycznej. Związki międzymetaliczne są traktowane jako przyszłościowe materiały do pracy w wysokich temperaturach i agresywnych środowiskach korozyjnych. Zainteresowanie tą grupą materiałową jest spowodowane dobrymi właściwościami użytkowymi: dobrą odpornością na utlenianie i korozję w środowisku wody morskiej, zużycie ścierne. Ponadto niskie koszty materiałowe stwarzają perspektywy do zastosowań jako alternatywę dla stali odpornych na korozję. Ograniczeniem możliwości szerokiego zastosowania intermetalików z układu Fe-Al, jest ich niedostateczna plastyczność, będąca czynnikiem hamującym ich dalszy rozwój jako materiałów konstrukcyjnych. Stąd badania nad możliwością poprawy plastyczności droga obróbki cieplno-plastycznej. W artykule analizowano wpływ parametrów odkształcania na mikrostrukturę stopu Fe-38% at. Al z mikododatkami cyrkonu, molibdenu, boru węgla. Z wykorzystaniem symulatora Max Strain, który umożliwa kumulację dużych odkształceń w materiale. Przeprowadzono badania mikrostuktury i substruktury wykazały istotny wpływ odkształcania tą metodą na rozdrobnienie ziarn w badanym stopie.

1. Introduction

Intermetallic-phase-based alloys FeAl, Fe_3Al of the Fe-Al system, also referred to as iron aluminides, due to their combination of attractive properties have recently been attracting attention of many research and industrial centres [1÷4]. They are treated as “the materials of the future”, constructional and for coating, for high temperatures applications and aggressive, corrosive environment. The interest in this group of materials is caused by their low cost, low density, good resistance to abrasive wear as well as resistance to oxidation and corrosion, which creates broad perspective for their application in power, chemical, and petrochemical, naval and food industry. One of the basic factors limiting application of alloys from the system of Fe-Al is their low plasticity and susceptibility to brittle cracking, which restrict their development as future construction materials [5, 6].

At the Department of Material Science of the Silesian Technical University, research related to study of microstructural phenomena taking place during hot plastic deformation as well as concerning development of hot-and-plastic working technology for the tested alloys of Fe-Al system is carried out. During the last couple of years, the increase of interest in deformation methods that enable grain size reduction in metals and their alloys by means of cumulative plastic deformation, was observed. These methods have been acknowledged because of the possibility to obtain ultra-fine-grained and nano-crystalline structure of the material with physical and chemical characteristics indicating new application range. The methods that enable to obtain ultra-fine-grained structure in-

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clude: High-Pressure Torsion (HPT), Equal – Chanel Angular Pressing (ECAE), Cyclic – Extrusion – Compression (CEC). One of such methods is also cumulative plastic deformation, performend on simulator Max Strain [7÷10].

Application of unconventional ways of deformation enables grain size reduction in alloys, leading to improvement of their therefore improved mechanical properties. In the course of previously carried out works connected with FeAl alloys, problems with obtainment of fine-grained structure were encountered e.g. under conditions of conventional rolling [11]. An advantage of the applied method is the possibility of deformation in the elevated and high temperature, which is especially important the case of alloys with B2 structure, that cannot undergo cold deformation due to their low formability.

The article presents the research mainly aimed at determining the influence of deformation parameters on microstructure of the alloys from Fe-Al system with 38% at. aluminium and with modifying additions of boron (B) and zircon (Zr), using the hot cumulative plastic deformation on the Max Strain multi-axis deformation simulator.

2. Test methodology and material

Material for the research consisted of cast bars from an alloys based on an FeAl intermetallic phase of a chemical composition shown in Table 1. Chemical composition was comparable to an alloy developed in Oak Ridge National Laboratory, designated as FA-385 [1]. To modify , microadditives of C, Zr, B were added to the alloy, and to improve strength – Mo additive. The cast was made an induction furnace (IS5/III type by Leybold-Heraeus) with in vaccuous application of compacted magnesite melting-pot (of $Al_2 0_3 \cdot MgO$ spinel). The alloy was prepared by means of gravity casting into cold graphite moulds. Obtained ingots were next subject to hot treatment in order to homogenize their microstructure. The process consisted of annealing in the temperature of 1000°C for 48 hours and cooling in the furnace.

TABLE 1

Chemical composition of tested alloy Fe-38Al (at %, wt %.)

	Al	Mo	Zr	C	B	Fe
at.-%	38.00	0.20	0.05	0.10	0.01	61.64
wt.-%	22.82	0.43	0.10	0.03	0.002	76.62

Model attempts of hot cumulative plastic deformation were made on simulator Max Strain at the Institute of Ferrous Metallurgy in Gliwice [13].The samples with unbounded ends were deformed what enabled their free elongation. During deformation, temperature was measured with of pyrometer. Attempts of deformation were made in two stages:

- preliminary deformation at 1150°C in order to remove the initial, dendritic microstructure caused by crystallization process;
- final deformation that formed the fine grain size at 1000°C, 900°C, 800°C and 700°C;

Deformation rate applied were 0.1 s^{-1} and 0.5 s^{-1} respectively, actual deformation in each cycle was ε =0.4 and 6, 8, 16 deformations were applied. The samples were placed in a rotary handle, and then they were subject to deformation in the direction perpendicular to the axis. Next, they were turned by 90° and subject to further cycles of deformation (Fig. 1).

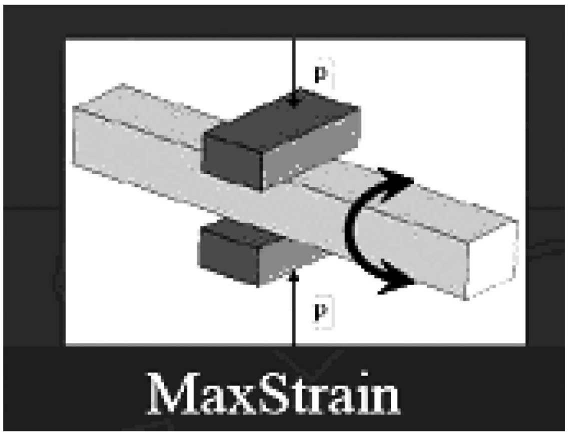


Fig. 1. The diagram of the applied cumulative deformation method on Max Strain simulator

The examination of the microstructure of the obtained martial were performs by means of light microscopy and transmission electron microscopy. Distribution of grain size was determined, by means of EBSD technology distribution of grain/subgrain misorientation. The tests were made on samples in initial condition and after deformation, on scanning electron microscope Inspect F with application of EBSD detector. Evaluation of size and shape of grain was made with application of image analysis software MetIlo [15].

3. Test results analysis

Microstructure of the alloy after casting and homogenizing hot treatment has been shown in Fig. 2. Tested alloy Fe-38Al has a coarse grain microstructure, average surface of grain plane section of $\bar{A}$=12600μm^2 (grain size 115μm) [16,17,19]. Obtained X-ray diffraction patterns for Fe-38Al alloy sample are presented in Fig. 3. The presence of FeAl phase (B2 type structure) is clearly seen for Fe-38Al alloy sample annealed for 48h, but some reflect from A2 phase were observed.

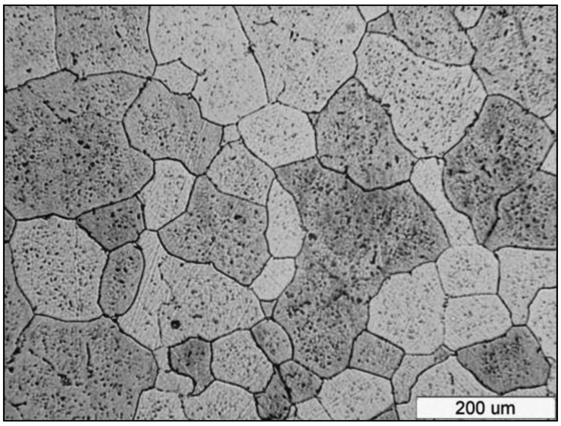


Fig. 2. Microstructure of Fe-38Al after homogenizing treatment at the temperature of 1000°C for 48h and furnace cooling

EBSD analysis enabled to determine distribution of grain size and misorientation angle of the tested Fe-38Al alloy (Fig. 4). After annealing, a small number of small-angle grain boundaries <5% was observed in the calculated maps (Fig. 4b). Once again, a great number of grain boundaries with the misorientation angle of >15° in the microstructure was detected. Such distriubution demonstrate a random orientation.

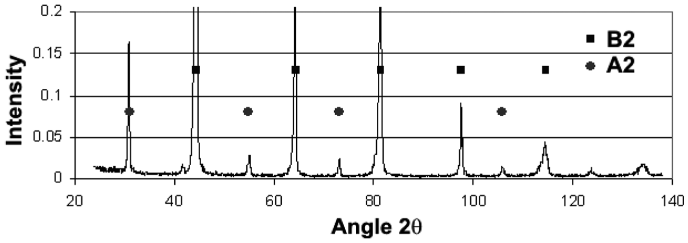
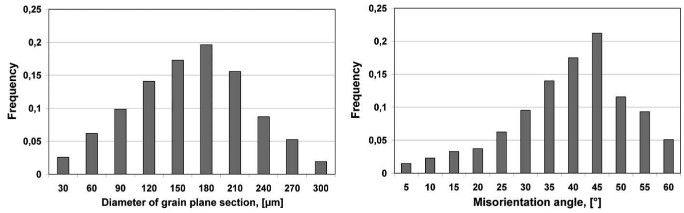


Fig. 3. X-ray diffraction patterns for Fe-38Al alloy sample



a) b)

Fig. 4. The distribution of grain sizes in initial state after casting (a), the distribution of boundary misorientations as measured by linear intercept (b)

The results obtained from the tests carried out on Max Strain simulator enabled to determine relation between average unit pressure and deformation. For example values of maximum unit pressure in the respective cycles of deformation for samples after preliminary deformation in the temperature of 1150°C (first 4 deformations) with next 12 deformations in the temperature of 1000°C and 900°C are shown in Fig 5. During deformation at the temperature of 1150°C, the value of maximum unit pressure was approx. 200 MPa. Decreasing deformation temperature up to 1000°C causes almost triple increase of pressure, and down to the temperature of 900°C increase to 900 MPa in first cycle. With further deformations decrease of hardening can be observed, which can be caused by intensive processes of structure rebuilding.

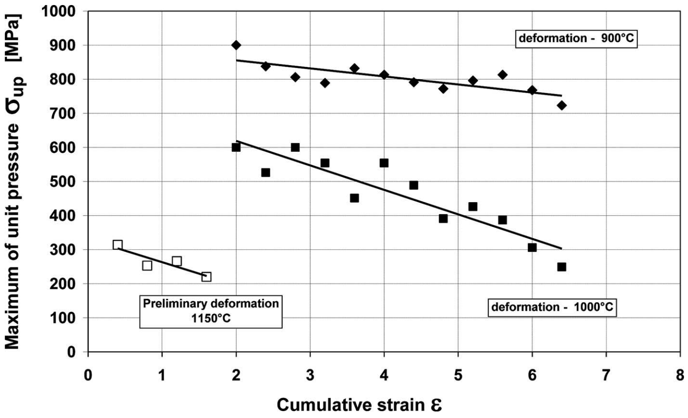
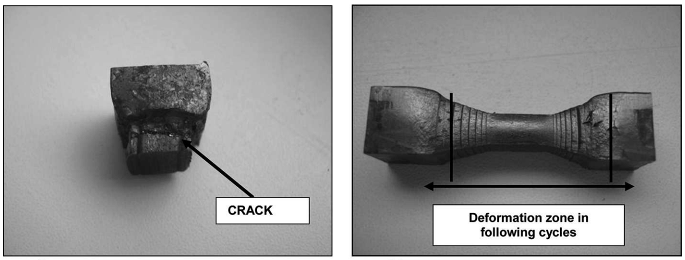


Fig. 5. View of deformed sample made of Fe-38Al alloy a) cracking at 700°C, b) sound deformation at 900°C

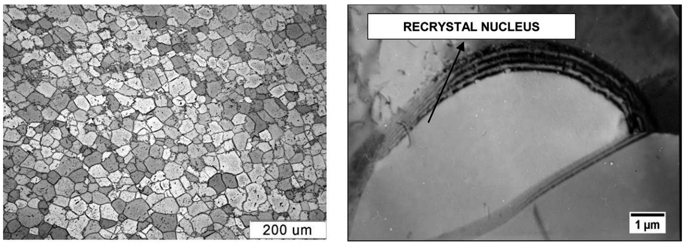
The analysis of deformation in the temperature of 800 and 900°C was also carried out. During accumulation of deformation macroscopic cracking in the zone between deformed and non-deformed material could be seen. It results from extremely high brittleness after casting and from homogenisation as well as from the increase of long-range order with the decrease of forming temperature (Fig. 6a). Cracks were extremely intensive in the zone between deformed and non-deformed alloy.

Higher deformation temperature increased substantially the deformability of alloy without cracking (Fig. 6b). After deformation at the temperature of 1000°C and after 8 cycles of deformation, grains with reduced size could be seen with average substitute diameter of grain plane section d=19,7μm. New grains have equiaxial shape and are approx. 6 times smaller in comparison with these observed before deformation (Fig. 7a). In the substructure of the tested alloy at that stage of deformation, the presence of well-developed subgrains with small dislocation density and of recrystallization nuclei was noticed (Fig. 7b). Process of recrystallization brings about creation of grains free of dislocation.



a) b)

Fig. 6. The maximum unit pressure in function of deformation. Frist 4 deformations at 1150°C, next 12 deformations at the temperature of 900 and 1000°C



a) b)

Fig. 7. Microstructure of Fe-38Al alloy after deformation ε =0.25 at temperature T=1150/1000°C with a strain rate of 0.1 s^{-1}, number of deformation cycles 4/4 (a) fine grains after deformation, (b) nuclei of recrystallization and migration of high angle boundaries

The purpose of the second variant of deformation was to cause further grain size reduction. It enabled decrease of deformation temperature and application of more deformations – 16 (4/12). After deformation at the temperature of 1150°C /1000°C and 900°C for Fe-38Al, the tests showed intensive process of grain size reduction (Fig. 8). The process of intensive grain size reduction in the structure of the tested Fe-38Al alloy was confirmed by the analysis of microstructure made by means of EBSD method. The results of the analysis allowed to make assumption that the process of such a strong grain size reduction of Fe-38Al alloy structure results from intensive dynamic recrystallization process. The obtained grains had substitute diameter of average grain plane section d = 12.5 and 7.3 μm after final deformation at 1000°C and 900°C respectively (initial average grain size after casting was d_o = 115μm). It seems, that depending on the degree of deformation (hardening) those both processes may lead to reconstruction of the structure. Presented bar chart proves that the size of the majority of subgrains/grains ranges from 2 μm to 14 μm, what makes over 90% of the analysed area (Fig. 9a). Bar chart of frequency rate of grains shows approximately equal content of low angle and high angle boundaries (Fig. 9b). After deformation at 900°C subgrains with diversified dislocation density (Fig. 10a) and recrystallization nucleus were found in substructure (Fig.10b).

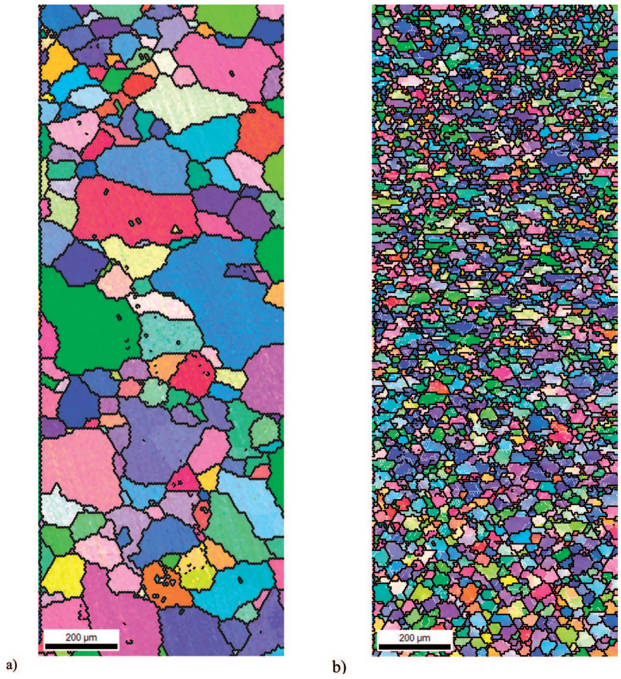


Fig. 8. EBSD map of a recrystallized specimen of Fe-38Al alloy after casting (a) and deformation at temperature T =1150°C/900°C with a strain rate of 0.1 s^{-1}, number of deformation cycles 4/12 (b)

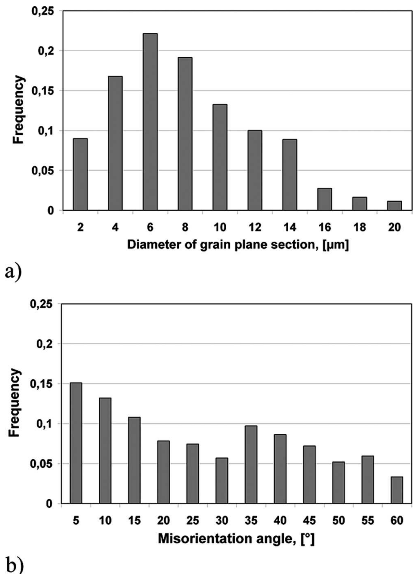


Fig. 9. The distribution of grain sizes in initial state after deformation (a), the distribution of boundary misorientations as measured by linear intercept (b) deformation parameters such as in Fig. 8

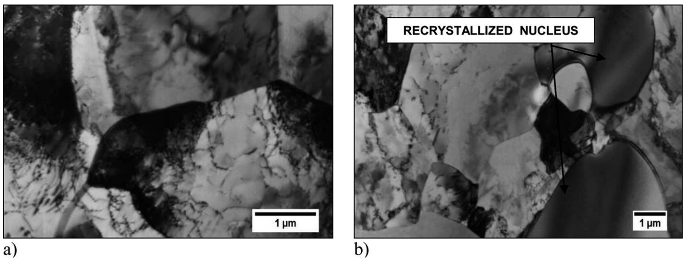


Fig. 10. Substructure of Fe-38Al alloy after deformation ε =0.4 at the temperature of 1150°C/900°C, number of deformation cycles 4/12 (a) subgrains with defected structure, (b) fine subgrains and nuclei of recrystallization

4. Summary

Improvement of the mechanical properties of cast alloys of Fe-Al systems can be enabled by controlled process of hot deformation, as it was shown in the study [17]. The applied methods of cumulative deformation on Max Strain simulator show possibility of intensive grain size reduction. Tested alloy is characterised by coarse grain structure after casting. Deformation performed on Max Strain simulator had a beneficial influence on the process of microstructure refinement. The changes in the microstructure of the alloy was caused by dynamic and static recrystallization. Suitable number of deformations (8, 16) and applied temperatures of the process (1150°C /1000°C, 1150°C /900°C) enable to obtain substantial grain size reduction of the tested material. Average grain diameter of d= 7.3 μm was obtained, that is 15 times the effect of grain size reduction. The substructure showed finely shaped subgrains, but secondary effect increasing dislocation density are disclosed. Nucleation of new grains and their growth in the process of dynamic recrystallization in the Fe-38Al alloy progresses through migration of high angle grain boundaries. EBSD method revealed that the content of narrow-angled and wide-angled boundaries was comparable. Applied deformation scheme does not influence to a large extent the creation of texture. It was proved, though, that decrease of the temperature below 900°C leads to cracking of the tested alloy. In further studies it is planned to use additional steel shields that will separate brittle intermetallic phase and cold dies. Decrease of processing temperature should lead to further decrease of grain size. It will enable effective processing of this alloy under conditions of cumulative plastic deformation.

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