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## STRUCTURE AND SOME MECHANICAL PROPERTIES OF Fe<sub>3</sub>Al-BASED CAST ALLOYS

### STRUKTURA I WŁAŚCIWOŚCI MECHANICZNE STOPÓW NA OSNOWIE FAZY Fe<sub>3</sub>Al

In the paper was studies the influence of chemical composition and temperature on the properties, microstructures, hardness and ordering process of intermetallics on the base closed to one of Fe<sub>3</sub>Al phase. The major problem restricting universal application of intermetallic phase base alloy from Fe-Al system is their low plasticity which leads to hampering their development as construction materials. The analysis were performed for two alloys i.e Fe28Al and Fe28Al-5Cr which were produced by casting and next were annealed for 8, 16 and 48 hours at 1000°C. Both alloys is closed to one of Fe<sub>3</sub>Al phase. Mechanical properties were measured by the uniaxial tensile test at room temperature and at 600°C. Some basic mechanical properties i.e. R<sub>0.2</sub>, R<sub>m</sub>, A<sub>5</sub>, Z were determined. Were obtained increasing ductility with a increase the temperature test in air condition for both alloys. Structural examination was carried out using light microscopy and scanning electron microscopy. There observed that in Fe28Al alloy without Cr phases rich in Fe and Al, which can correspond to FeAlC<sub>0.5</sub> were visible as well as phases rich in Zr. The precipitates rich in Cr and Zr were identified alloy with Cr which probably correspond to FeCr<sub>2</sub> ZrC and Cr<sub>2</sub>Zr phases. The fractographs of destruction surfaces after tensile tests shown, that for both alloy at room temperature are brittleness and intergranular. After the test at 600°C character of the fractures were changed (different). Were observed transgranular cleavage for both alloys.

The ordering process was analyzed by X-ray diffraction, Mössbauer spectroscopy. The studied alloys were prepared by melting in induction furnace under vacuum and next were gravitatively casted into cylindrical graphite moulds. After then samples were annealed at 1000°C for 8, 16 and 48 hours. Different behaviour of Fe-28Al and Fe-28Al-5Cr alloys after annealing were founded. Microstructures for both alloys was similar. Were indicated the presence of phases and precipitates in investigated alloys in the dendritic primary structure which wasn't changes after the heat treatment process. Were observed the influence the time of annealing on the hardness alloys. Increasing time of annealing from 8h to 48h at 1000°C influenced on the increasing of hardness in Fe-28Al alloy and decreasing of hardness in Fe-28Al-5Cr alloy. X-ray diffraction method were obtained that the Fe<sub>3</sub>Al phase of DO3 type structure was stated only in the sample of Fe28Al alloy annealed for 48 hours. The FeAl phase appeared to be the main phase in the other samples. All the informations including a some of mechanical properties, analysis of microstructure, long range ordering during the heat treatment of studied alloys could be helpfully for technological processing this materials.

*Keywords:* metal alloys, iron aluminides, static tensile test, microstructure

W pracy przedstawiono wpływ składu chemicznego na właściwości, mikrostrukturę, twardość oraz proces porządkowania stopów na osnowie fazy Fe<sub>3</sub>Al. Badania prowadzono dla dwóch stopów: Fe28Al i Fe28Al-5Cr, które zostały wytworzone metodą odlewania próżniowego, a następnie poddane wyżarzaniu przez 8, 16 i 48 godzin w temperaturze 1000°C. Właściwości mechaniczne obu materiałów wyznaczono na podstawie statycznej próby rozciągania w temperaturze pokojowej oraz w temperaturze 600°C. Wyznaczono podstawowe właściwości mechaniczne, takie jak: R<sub>0.2</sub>, R<sub>m</sub>, A<sub>5</sub>, Z. Badania strukturalne przeprowadzono wykorzystując technikę mikroskopii świetlnej oraz skaningowej mikroskopii elektronowej. Zaobserwowano, iż w stopie Fe28Al bez dodatku Cr w strukturze tworzą się fazy bogate w Fe i Al, które scharakteryzowano jako FeAlC<sub>0.5</sub>. oraz fazy bogate w Zr jako jeden z mikrodotyków. W stopie Fe28Al z 5% dodatkiem Cr ujawniono głównie tworzenie faz bogatych w Cr a także w Zr, takich jak FeCr<sub>2</sub> ZrC i Cr<sub>2</sub>Zr. Wykonane badania faktograficzne powierzchni zglądów powstałych po próbie rozciągania ujawniły, że charakter pęknięcia stopów badanych w temperaturze pokojowej jest łamliwy i międzykrystaliczny. Po próbie przeprowadzonej w temp. 600°C przelomy miały charakter transkrystaliczny dla obu stopów. Proces porządkowania analizowano wykorzystując dyfrakcję rentgenowską, oraz spektroskopię efektu Mössbauer'a. Wykazano, że faza Fe<sub>3</sub>Al typu DO3 występuje tylko w próbce Fe28Al, którą poddano wyżarzaniu przez 48 godzin. Fazę FeAl zaobserwowano w pozostałych próbkach.

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## 1. Introduction

Some intermetallics like iron aluminides are known as materials useful for high-temperature structural applications. Their good corrosion resistance, high mechanical strength, and low density wide prospects for their industrial applications, for some components of machines working at a ambient and high temperature and corrosive environment. Unfortunately steel the major problem restricting universal application of iron aluminides is their low plasticity and brittle cracking susceptibility. Currently researches of intermetallics from Fe-Al base focuses on their plasticizing and increasing mechanical properties [1÷7]. It is well known that the properties (yield strength and hardness) are strongly dependent from the working temperature. The purpose of this work is the analyze of the influence of testing temperature on the mechanical properties and the destructing surface after the tensile tests at room temperature and at 600°C of the alloys from Fe-Al system, based on Fe<sub>3</sub>Al intermetallic phases. The scope of the present paper is the analysis of chromium addition and the thermal treatment parameters on the properties and ordering process of iron aluminides with a 28%at. Al. All using methods are known but their comparison is very useful to better analysis of the behaviour of this alloys.



## 2. Experimental procedure

Material for investigations constituted multicomponent alloys from Fe-Al system, the composition of which has been presented in Table 1. The Mo addition has been used for the purpose of improving high temperature resistance. In order to decrease grain sizes after the crystallization process, a modified in the form of zirconium has been used. The carbon addition has been used in order to increase resistance, and boron has been used in order to increase the resistance of grain boundaries. The objective introduction of Cr in the amount of 5% at. was increase plastic properties. The alloys were produced by vacuum induction melting. The obtained heat was melted twice and drop cast into graphitoidal moulds to obtain rods with 12 mm in diameter and 120 mm in length (Fig. 1).

Subsequently, the material was subjected to homogenizing annealing at the temperature of 1000°C for 8, 16 and 48h to eliminate coring. The alloys were furnace cooled after heat treatment.

TABLE 1

Chemical composition of the alloys

Chemical elements contents [at.%]						
Al	Mo	Cr	Zr	C	B	Fe
28,0	0,20	–	0.05	0.1	0.01	71.64
28.0	0.30	5.0	0.05	0.1	0.01	66.64

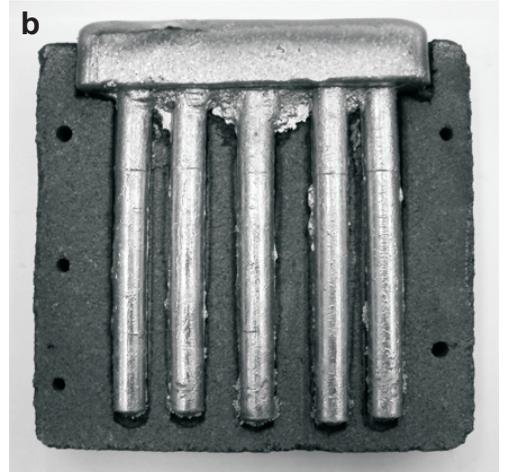


Fig. 1. Photographs of casting process (a) and rods modules after casting and crystallization in graphite forms (b)

Metallographic researches were carried out with use of light optical microscopy and scanning electron microscopy. The microanalysis of chemical properties of tested alloys after the process of homogenization was carried out with use of HITACHI S-3400N scanning

microscope which was equipped with Voyager System SIX roentgen microanalysis system together with EDS spectrometer.

The mechanical properties of presented alloys were determined during the static tensile test. The research

was conducted at the room temperature and at 600°C in air, with the use of cylindrical samples with the following dimensions of measuring part:  $d=6\text{mm}$  and  $l=30\text{mm}$ . The properties were determined for two samples of each of the alloys. Fracture surface were examined in the scanning electron microscope. Metallographic researches were carried out with the use of metallographic light microscope (LM). Hardness measurement were made with using the Vickers method (HV2) at a ZWICK tester.

X-ray diffraction patterns were collected using X-ray Philips diffractometer equipped with graphite monochromator on diffracted beam. Investigations of local Fe configurations formed in the studied materials were carried out by Mössbauer spectroscopy. The measurements of the  $^{57}\text{Fe}$  Mössbauer spectra were performed in transmission geometry by means of a constant spectrometer of the standard design. The 14.4 keV gamma rays were provided by a 50 mCi source of  $^{57}\text{Co/Rh}$ . The spectra of the samples were measured at room temperature.

### 3. Result of the research

The structure analysis were showed that the both tested alloys exhibit primary dendrite structure with interdendritic phases and precipitates. Microstructures of Fe-28Al alloy and Fe-28Al-5Cr alloy after homogenizing annealing with assumed parameters were presented in Fig. 2÷5. On the boundaries of dendrite grains and inside the grains, the phases of different size and form are observable. The detailed analysis of revealed phases which was carried out with use of scanning microscope with EDS analyser has proven that in Fe-28Al alloy without Cr the analysis the occurrence of the phases rich in Fe and Al, which can correspond to  $\text{FeAlC}_{0,5}$  (Fig. 4, 6, point A), as well as phases rich in Zr. The precipitates rich in Cr and Zr were identified in alloy containing Cr. The phases with Cr and Zr probably correspond to  $\text{FeCr}_2$  (Fig. 5, 6, point B),  $\text{ZrC}$  and  $\text{Cr}_2\text{Zr}$  phases.

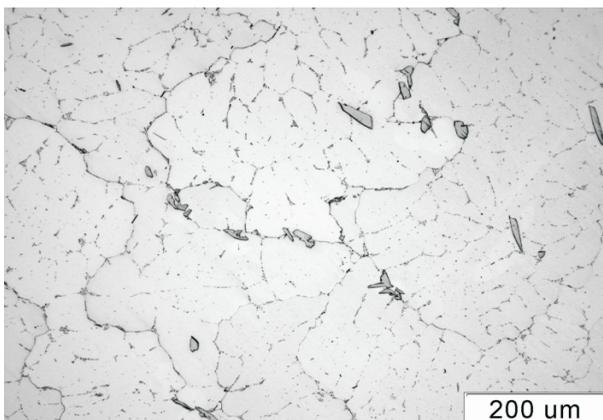


Fig. 2. Microstructure of Fe-28Al in the annealed conditions:

48h/1000°C. Precipitates and phases on the boundaries of grains and inside the grains

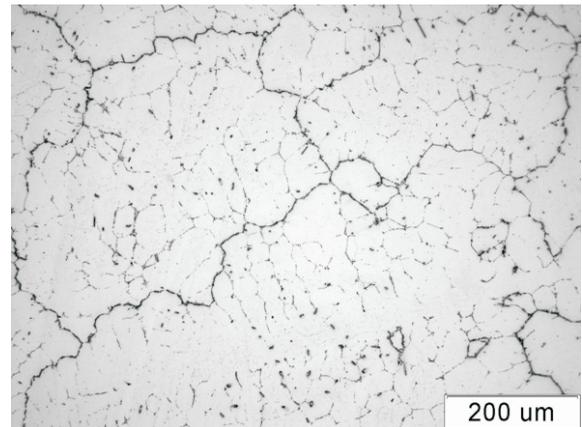


Fig. 3. Microstructure of Fe-28Al-5Cr in the annealed conditions: 48h/1000°C. Precipitates and phases on the boundaries of grains and inside the grains

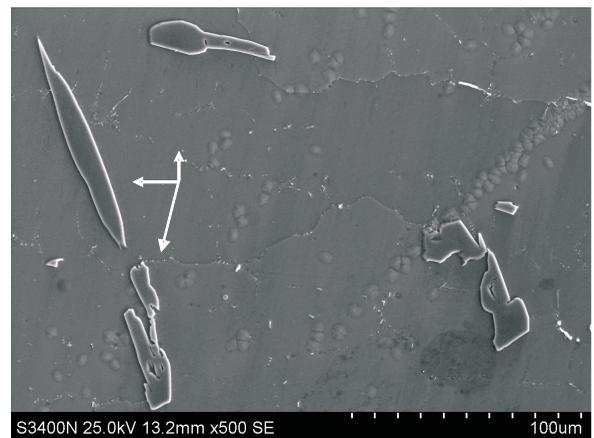


Fig. 4. SEM image of Fe-28Al after annealing (48h/1000°C)

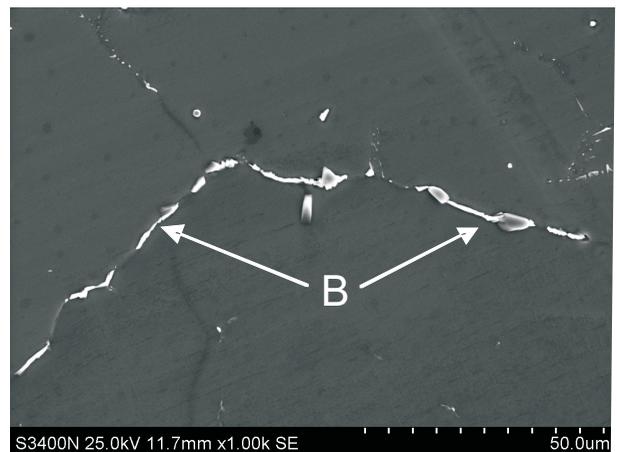


Fig. 5. SEM image of Fe-28Al-5Cr after annealing (48h/1000°C)

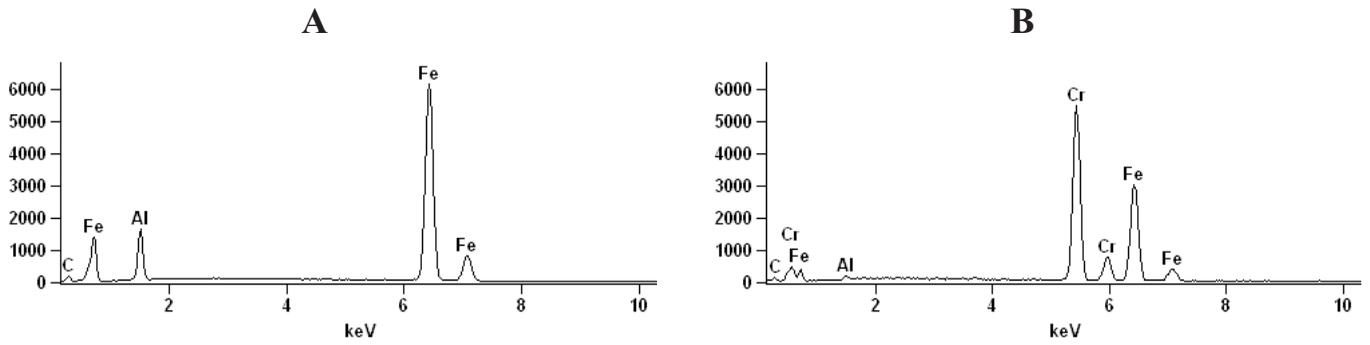


Fig. 6. Chemical analysis in microareas for investigated alloys

Microstructures of Fe28Al alloy without of Cr (Fe-28Al) and Fe28Al alloy with Cr (Fe-28Al-5Cr) after the process of homogenizing annealing with assumed parameters were presented in Fig. 7,8. Generally it wasn't found the most changes in microstructure with a increasing the time of annealing. In the tested materials microstructure, the grains of variable dimensions with characteristic residues of dendritic structure were found. For both alloys on the boundaries of dendrite and grains

as well as inside the grains, the phases of different size and form are observable.

The hardness of the investigated alloys after heat treatment at 1000° with variable holding time (8, 16, 48 hours) were presented in Fig. 9. We observed that the hardness of Fe-28Al had a higher level in compare with Fe-28Al-5Cr alloy. Indicated that in Fe-28Al alloy a hardness was increased with a extended time of the annealing and in the Fe-28Al-5Cr alloy tendentious is reverse.

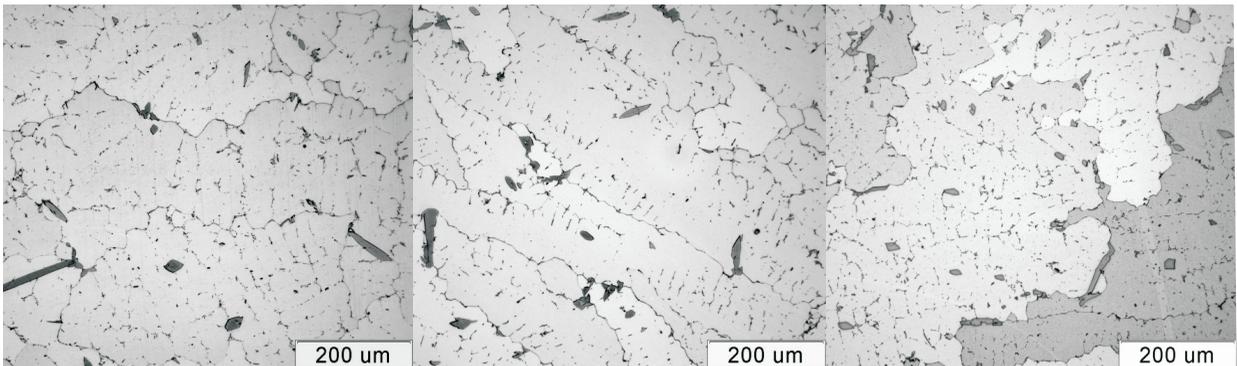


Fig. 7. Microstructure of Fe28Al after annealing at temperature 1000°C for: a) 8h; b) 16h; c) 48h

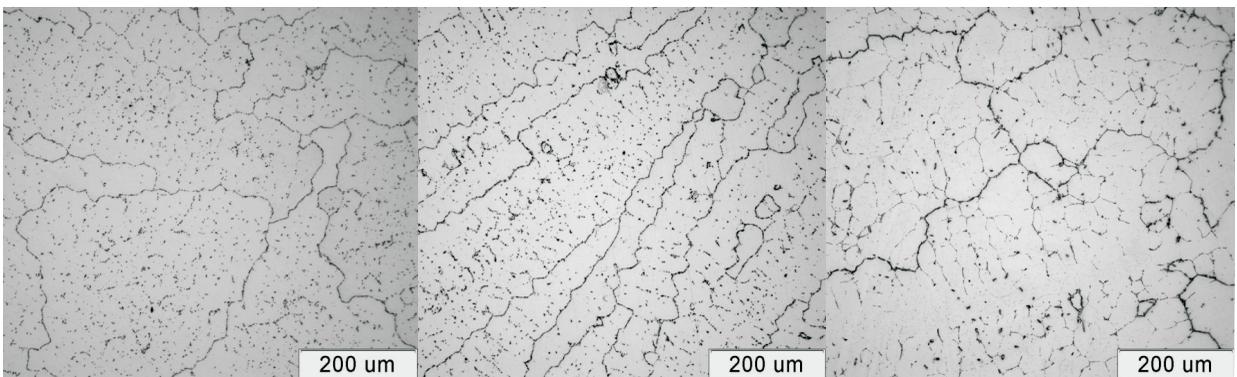


Fig. 8. Microstructure of Fe28Al-5Cr after annealing at temperature 1000°C for: a) 8h; b) 16h; c) 48h

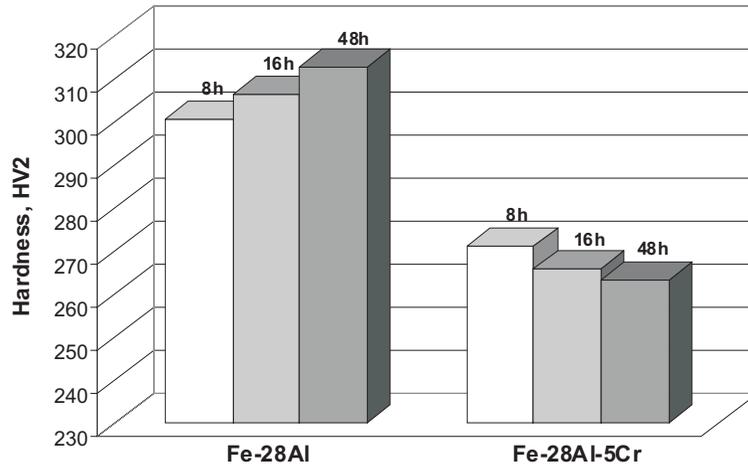


Fig. 9. Comparison of hardness values of the investigated alloys

X-ray diffraction patterns for Fe28Al and Fe28Al5Cr alloy samples were presented on Fig. 10 and Fig. 11.

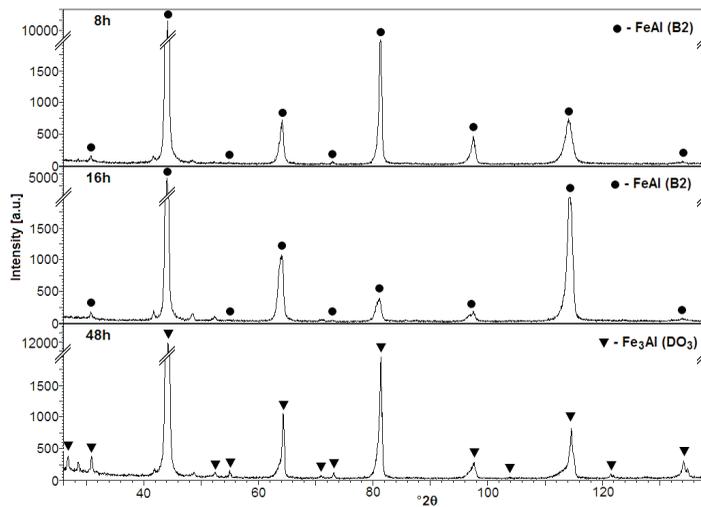


Fig. 10. X-ray diffraction patterns of Fe-28Al alloy samples annealed at 1000°C for 8, 16 and 48 hours

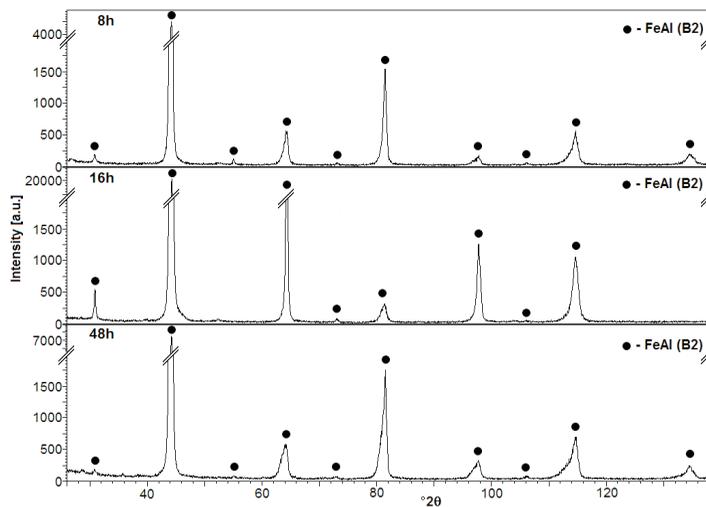


Fig. 11. X-ray diffraction patterns of Fe-28Al-5Cr alloy samples annealed at 1000°C for 8, 16 and 48 hours

The presence of  $\text{Fe}_3\text{Al}$  phase ( $\text{DO}_3$  type structure) is clearly seen for Fe-28Al alloy sample annealed for 48h. The FeAl, which had a B2 type structure, phase is the main one of the other samples.

Probably the observed difference in final phase composition of Fe-28Al and Fe-28Al-5Cr alloys after the same heat treatment could be explain by the low vacancy concentration in samples with chromium makes the atomic ordering process slower, which is investigated in work 2.

On the Fig. 12 were presents Mössbauer spectra of the samples annealed for 48h at  $1000^\circ\text{C}$  for both studied alloys. These spectra are the superposition of Zeeman sextets indicating the existence of magnetic phases with different degree of atomic ordering. In spectrum of Fe-28Al-5Cr sample (Fig. 12b) dominate the component corresponding to nonmagnetic phase [8]. Maximum value of hyperfine magnetic field ( $B_{\text{hf}}$ ) of  $^{57}\text{Fe}$  for Fe-28Al sample ( $\sim 300$  kGs) corresponds to the configuration of 8 iron atoms in the nearest surroundings of Mössbauer nuclide. This indicate that  $\text{Fe}_3\text{Al}$  phase of  $\text{DO}_3$  type structure is the main phase of Fe-28Al alloy sample annealed for 48h at  $1000^\circ\text{C}$  in accordance with X-ray diffraction analysis (Fig. 4). Mössbauer spectrum of Fe28Al5Cr alloy sample (Fig. 11b) is quite different from one of Fe-28Al sample (Fig. 11a). Maximum value of  $B_{\text{hf}}$  is much lower than 300 kGs what exclude  $\text{Fe}_3\text{Al}$  phase to be the main phase of Fe-28Al-5Cr alloy sample annealed for 48h at  $1000^\circ\text{C}$ . Central part of involved spectrum is described by component characteristic of nonmagnetic phase. This phase is FeAl one of B2 type structure according to X-ray diffraction analysis.

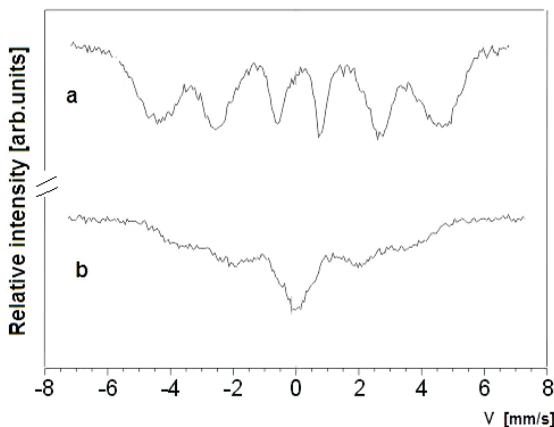


Fig. 12. The  $^{57}\text{Fe}$  Mössbauer transmission spectra for Fe28Al (a) and Fe28Al5Cr (b) alloys annealed for 48 h at  $1000^\circ\text{C}$

The alloys in the as-annealed state were subjected to uniaxial tensile test at room temperature and at  $600^\circ\text{C}$ . The charts on extension of alloy without Cr are presented in Fig. 13,14, result for alloy with Cr addition shows Fig. 15,16. Moreover, some basic mechanical properties of

investigated alloys (i.e.  $R_{0,2}$ ,  $R_m$ ,  $A_5$ ,  $Z$ ) were determined. Tables 2 and 3 present the effects evaluated by ambient temperature tension tests and  $600^\circ\text{C}$  temperature tension for Fe-28Al, Fe-28Al-5Cr appropriately.

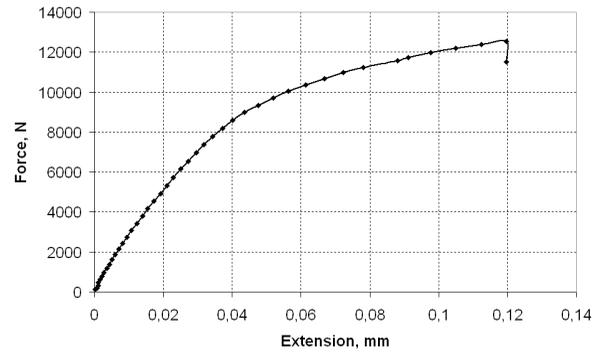


Fig. 13. The chart on Fe-28Al extension at room temperature

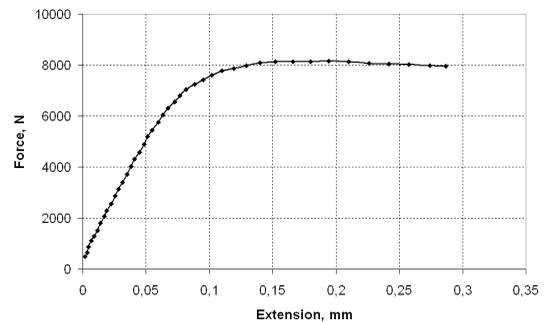


Fig. 14. The chart on Fe-28Al extension at  $600^\circ\text{C}$

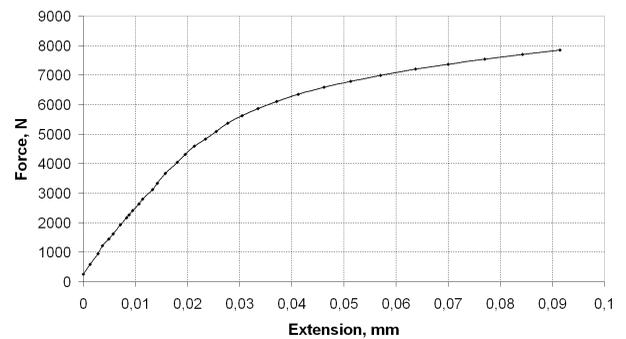


Fig. 15. The chart on Fe-28Al-5Cr extension at room temperature

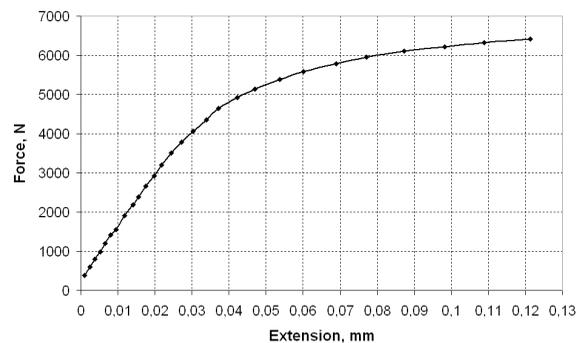


Fig. 16. The chart on Fe-28Al-5Cr extension at  $600^\circ\text{C}$

Mechanical properties of Fe-28Al

Alloy	R <sub>0,2</sub> [MPa]	R <sub>m</sub> [MPa]	A <sub>5</sub> [%]	Z [%]
room	414,7	463,6	1,2	–
temperature	449,2	471,6	1,3	1,7
600°C	309,8	311,5	27,4	77,3
	292,9	299,6	38,5	85,7

TABLE 2

Mechanical properties of Fe-28Al-5Cr

Alloy	R <sub>0,2</sub> [MPa]	R <sub>m</sub> [MPa]	A <sub>5</sub> [%]	Z [%]
room	271,6	285,3	2,5	–
temperature	284,6	327,6	3,3	–
at 600°C	239,9	266,7	32,3	84,0
	220,1	246,0	15,2	67,9

TABLE 3

ture ductility of both alloy without Cr addition and alloy with the contents of 5% at. of Cr. As an example, the extension of A<sub>5</sub> for specimen of Fe-28Al alloy accounts for 1,2%; 1,3% and for specimen of Fe-28Al-5Cr alloy is equal to 2,5%; 3,3%. Ductility investigated materials grows with increase test temperature. The extension A<sub>5</sub> determined during the static tensile test at 600°C is equal for Fe-28Al 27,4%; 38,5% and for Fe-28Al-5Cr accounts 32,3%; 15,2%. Simultaneously was observed the decrease of materials resistance properties (i.e. R<sub>0,2</sub>, R<sub>m</sub>).

Generally the Fe-28Al alloy had a higher strength, but the Fe-28Al-5Cr alloy had a smaller decreasing of strength with the changes of the temperature. Is important that in the tensile tests for the high temperature we obtained a Z [%] which will be impossible for the tests at room temperature.

Below on the Fig. 17 and 18 were presented the obtained fractographs for both analyzed alloys.

The results obtained indicate the low room tempera-

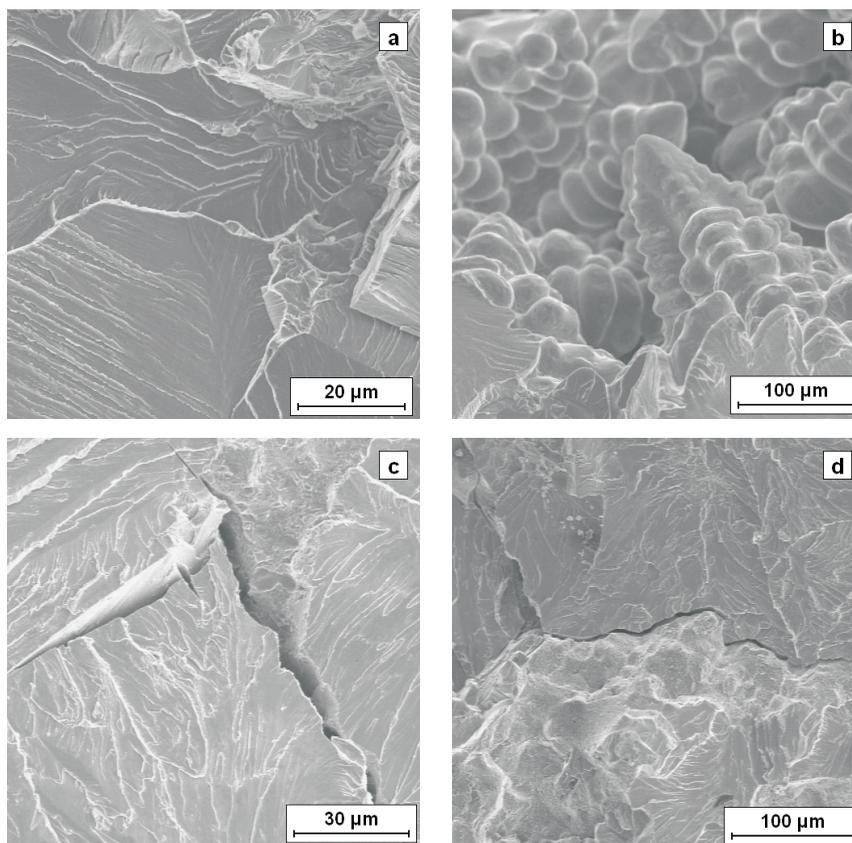


Fig. 17. SEM fractographs after tensile testing in air at room temperature: (a,b) Fe-28Al; (c,d) Fe-28Al-5Cr

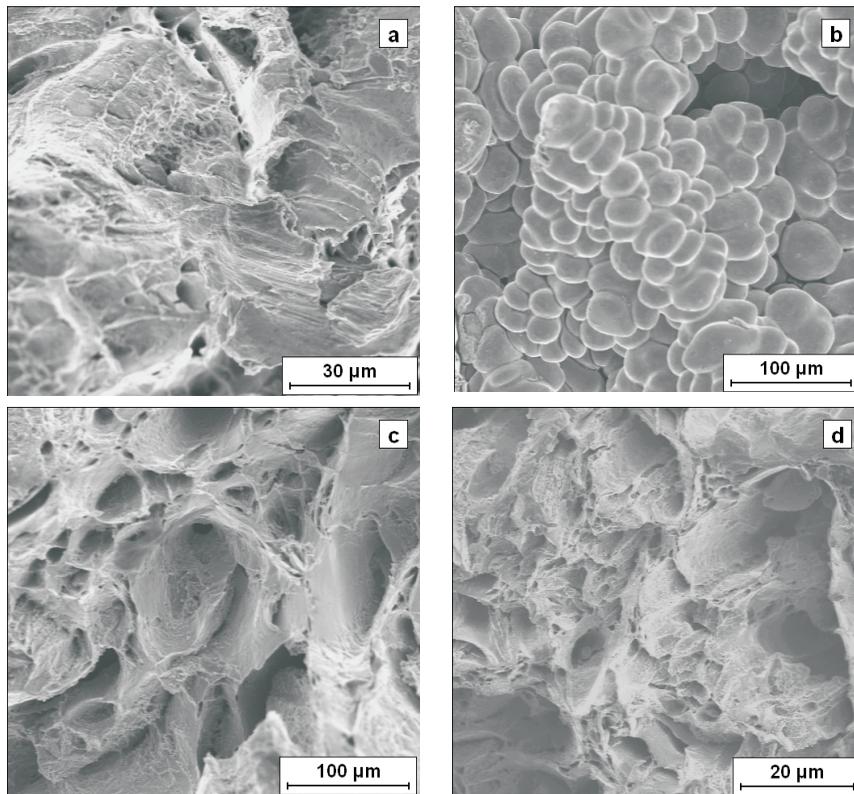


Fig. 18. SEM fractographs after tensile testing in air at 600°C temperature: (a,b) Fe-28Al; (c,d) Fe-28Al-5Cr

The fractographs (destructing surface) shown that for both alloy investigated at room temperature the fractures were brittleness intergranular character with very small tenacity areas for Fe-28Al-5Cr alloy.

After the test at 600° character of the fractures were changed. We observed mainly transgranular cleavage occurs in both investigated alloys. Cracking of alloys had intercrystalline character and occurred along or dendritic crystals boundaries.

#### 4. Conclusions

The paper deals with an evaluation, of the  $Fe_3Al$  intermetallic phase based alloys with and without the Cr addition, of microstructure, hardness, X-ray diffraction and Mössbauer spectroscopy after the heat treatment with different parameters.

The microstructure of both alloys isn't depended on time of the annealing process. Were not found a significant difference between Fe-28Al and Fe-28Al-5Cr alloy.

The hardness level of Fe-28Al-5Cr were almost 35HV2 lower in the compare with Fe-28Al alloy.

Comparison of long range ordering process in Fe-28Al and Fe-28Al-5Cr alloys were presents. Were obtained a good correlation between the results of X-ray diffraction, Mössbauer spectroscopy and positron anni-

hilation.  $Fe_3Al$  phase of  $DO_3$  type structure was found only in Fe-28Al alloy sample annealed at 1000°C for 48h. FeAl phase of B2 type structure appeared to be the main phase of the other studied samples. Were indicated that the long range ordering process is slower in Fe-28Al-5Cr alloy.

Because potential using of iron aluminides was in high temperatures application  $Fe_3Al$  – based alloys with multicomponents composition were investigated with respect to microstructure, mechanical properties at room temperature and at 600°C and surface fracture after a tensile tests. It was found that the micro-addition of Zr, B and Cr were located as a phases and precipitates on the boundaries of dendrite grains and inside the grains for of both tested alloys.

Were indicated that the increasing a plasticity influenced to a small decreasing of strength. Tensile tests of alloy with Cr addition were provided lower strength circa about 100MPa in comparison of alloy without Cr addition. On top of that alloy with Cr characterize greater decreasing with a change the temperature of test. The of elongation  $A_5[\%]$  and necking down  $Z[\%]$  were similar for both studied alloys. The fractures of alloys confirmed modify change of a character of creep from intergranular to transgranular.

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