



# Ordering process of Fe<sub>28</sub>Al and Fe<sub>28</sub>Al<sub>5</sub>Cr alloys

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## ABSTRACT

**Purpose:** The comparison of ordering process in Fe<sub>28</sub>Al and Fe<sub>28</sub>Al<sub>5</sub>Cr alloys annealed for 8, 16 and 48 hours at 1000°C was performed. The composition of studied alloys is closed to one of Fe<sub>3</sub>Al phase.

**Design/methodology/approach:** The studied alloys were melted in induction furnace under vacuum. Next the alloys were gravitatively casted into cylindrical graphite moulds. The alloy samples were annealed at 1000°C for 8, 16 and 48 hours. The ordering process was analyzed by X-ray diffraction, Mössbauer spectroscopy and positron annihilation methods.

**Findings:** Different behaviour of Fe<sub>28</sub>Al and Fe<sub>28</sub>Al<sub>5</sub>Cr alloys during annealing for 8, 16 and 48 hours at 1000°C was found. The Fe<sub>3</sub>Al phase of DO<sub>3</sub> type structure was stated only in the sample of Fe<sub>28</sub>Al alloy annealed for 48 hours. The FeAl phase appeared to be the main phase in the other samples.

**Research limitations/implications:** The applied investigation methods appeared to be useful in the studies of long range ordering process. Application of Rietveld refinement method enabled the verification of qualitative phase analysis and the determination of lattice constant parameters. Relatively great grain sizes in studied samples made the exact determination of long range ordering parameters difficult.

**Practical implications:** The information on the phase transformation during the heat treatment of alloys, including long range ordering, are of prime importance for technological processing. The structures with long range ordering significantly affect the properties of alloys with intermetallic phases.

**Originality/value:** Good correlation between the results of X-ray diffraction, Mössbauer spectroscopy and positron annihilation methods were obtained. Addition of chromium made the long range ordering process slower.

**Keywords:** Metallic alloys; X-ray phase analysis; Iron aluminides; Long range ordering; Rietveld refinement; Mössbauer spectroscopy

## MATERIALS

### 1. Introduction

Alloys with intermetallic phases are intensively studied now. Iron aluminides are an interesting class of materials. They offer a good combination of mechanical properties, good weight/strength ratio, corrosion resistance and relatively low material cost. It makes the alloys based on iron aluminides potential candidates for

the substitution of stainless steels in application at moderate to high temperature. Technical application of these alloys is restricted by poor ductility at low temperatures [1-4]. The possibilities of overcoming this restriction by introduction of additional alloy components are intensively studied [5-9].

The aim of the present paper is the analysis of chromium addition on the ordering process of Fe<sub>28</sub>Al alloy. The Rietveld

refinement method appeared to be very useful in the microstructure characterization of different materials with metallic and ceramic phases [10-19]. During Rietveld analysis a whole diffraction pattern is employed. Mössbauer spectroscopy was also applied to gain further insights into the structural evolution of the local environment around the iron atoms. Positron annihilation was used for the determination of vacancy concentration.

## 2. Materials and experiment

The studied alloys of nominal composition presented in Table 1 were prepared using the gravity casting technique. Melts were produced in induction furnace under a pressure of 1.0 MPa. Melted charge was homogenized and casted into graphite moulds. Such process was repeated three times with a mechanical cleaning between operations. As a result, the ingots in the form of rods were obtained with dimensions of  $\varnothing = 12$  mm and  $l = 120$  mm. The alloy samples were annealed for 8, 16 and 48 hours at 1000°C and cooled with furnace.

Table 1.  
Chemical composition of studied alloys (at%)

	Al	Mo	Cr	Zr	C	B	Fe
Fe28Al	28.0	0.20	-	0.05	0.1	0.01	71.6
Fe28Al5Cr	28.0	0.20	5.0-	0.05	0.1	0.01	66.6

X-ray diffraction patterns were collected using X-ray Philips diffractometer equipped with graphite monochromator on diffracted beam. The copper radiation was applied. During X-ray data collection the samples were rotated. 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  $^{57}\text{Co}/\text{Rh}$ . The spectra of the samples were measured at room temperature. The measurements of positron lifetime were made at room temperature using the conventional fast-fast spectrometer with time resolution of 270 ps for Co-60. The positron source of ~740 kBq activity covered by 5  $\mu\text{m}$  Ni foil was placed between two pieces of the investigated samples.

## 3. Results and discussion

X-ray diffraction patterns for Fe28Al and Fe28Al5Cr alloy samples are presented on Fig. 1 and Fig. 2, respectively.

The presence of  $\text{Fe}_3\text{Al}$  phase ( $\text{DO}_3$  type structure) is clearly seen for Fe28Al alloy sample annealed for 48h. The FeAl (B2 type structure) phase is the main one of the other samples. Because of relatively great grain size (~115  $\mu\text{m}$  and ~150  $\mu\text{m}$  for Fe28Al and Fe28Al5Cr alloys, respectively) of studied samples

the relation between the intensities of corresponding diffraction lines of FeAl and  $\text{Fe}_3\text{Al}$  phases are disturbed. This makes the estimation of the long range order parameter difficult. Lattice constant parameters determined by Rietveld refinement method show the tendency of their decreasing with the increase of annealing time. On Figures 3 and 4 the Rietveld plots with residual curves are presented for samples with main phase of FeAl (B2 type structure) and  $\text{Fe}_3\text{Al}$  ( $\text{DO}_3$  type structure), respectively.

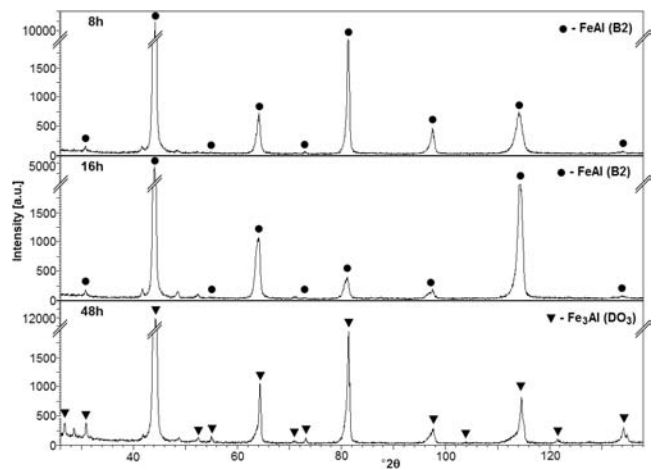


Fig. 1. X-ray diffraction patterns of Fe28Al alloy samples annealed at 1000°C for 8, 16 and 48 hours

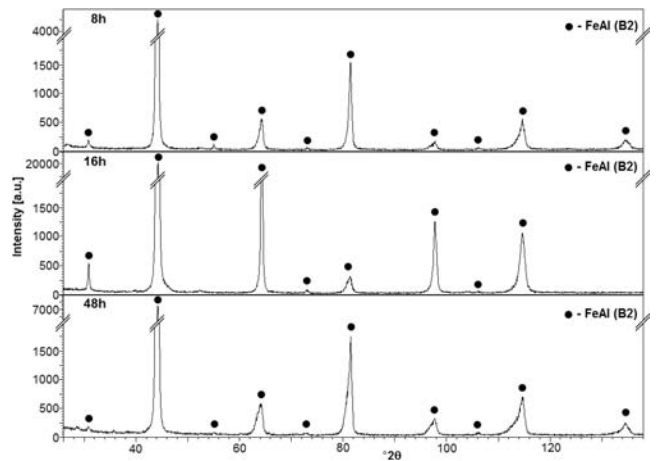


Fig. 2. X-ray diffraction patterns of Fe28Al5Cr alloy samples annealed at 1000°C for 8, 16 and 48 hours

The goodness of fit (GoF) parameter is often used to control the quality of fitting of calculated pattern to the experimental one, and is defined as a ratio of the weighted residual error,  $R_{wp}$  to the expected error,  $R_{exp}$ .  $GoF = R_{wp} / R_{exp}$

The best values of GoF parameter obtained for studied samples are of the order of 2.2 what seems to be satisfactory because of relatively great grain size of studied samples.

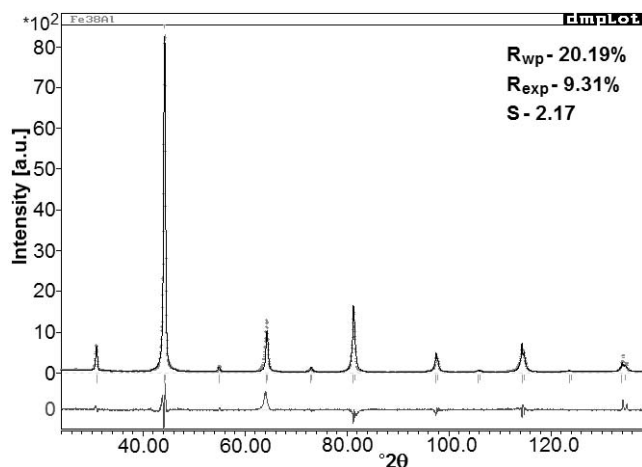


Fig. 3. Rietveld refinement plot for sample with FeAl (B2 type structure) main phase

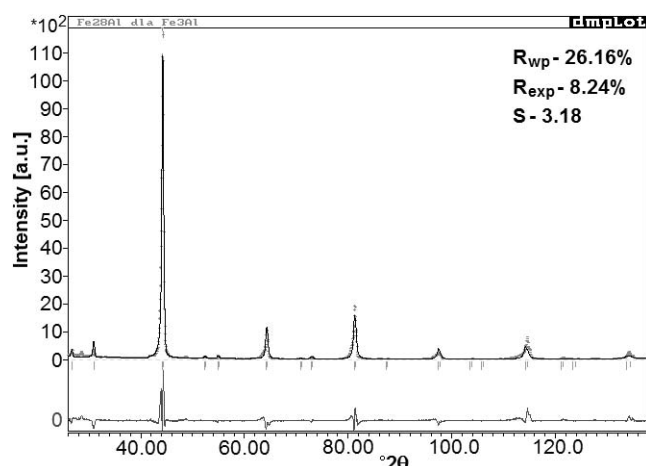


Fig. 4. Rietveld refinement plot for sample with Fe<sub>3</sub>Al (DO<sub>3</sub> type structure) main phase

Figure 5 presents Mössbauer spectra of the samples annealed for 48h at 1000°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<sub>28</sub>Al<sub>5</sub>Cr sample (Fig. 5b) dominate the component corresponding to nonmagnetic phase [20]. Maximum value of hyperfine magnetic field ( $B_{hf}$ ) of <sup>57</sup>Fe for Fe<sub>28</sub>Al sample (~300 kGs) corresponds to the configuration of 8 iron atoms in the nearest surroundings of Mössbauer nuclide. This indicate that Fe<sub>3</sub>Al phase of DO<sub>3</sub> type structure is the main phase of Fe<sub>28</sub>Al alloy sample annealed for 48h at 1000°C in accordance with X-ray diffraction analysis (Fig. 1). Mössbauer spectrum of Fe<sub>28</sub>Al<sub>5</sub>Cr alloy sample (Fig. 5b) is quite different from one of Fe<sub>28</sub>Al sample (Fig. 5a). Maximum value of  $B_{hf}$  is much lower than 300 kGs what exclude Fe<sub>3</sub>Al phase to be the main phase of Fe<sub>28</sub>Al<sub>5</sub>Cr alloy sample annealed for 48h at 1000°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.

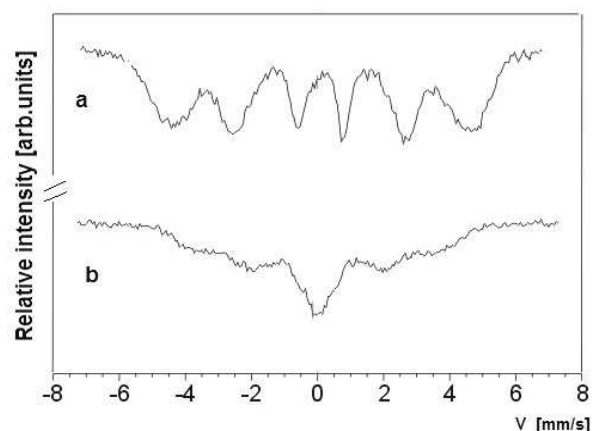


Fig. 5. The <sup>57</sup>Fe Mössbauer transmission spectra for Fe<sub>28</sub>Al (a) and Fe<sub>28</sub>Al<sub>5</sub>Cr (b) alloys annealed for 48 h at 1000°C

The studies of point defects performed by positron annihilation indicate the presence of monovacancies in iron sublattice. The concentration of monovacancies significantly depends on the chromium addition. The number of vacancies in Fe<sub>28</sub>Al alloy samples after cooling with furnace is almost twice higher than in Fe<sub>28</sub>Al<sub>5</sub>Cr alloy samples after the same heat treatment [20]. Such character of point defect structure can suggest that ordering process during slow cooling is mainly controlled by diffusion mechanism ongoing by vacancy concentration. Low vacancy concentration in samples with chromium probably makes the atomic ordering process slower what can explain the observed difference in final phase composition of Fe<sub>28</sub>Al and Fe<sub>28</sub>Al<sub>5</sub>Cr alloys after the same heat treatment.

#### 4. Conclusions

- Comparison of long range ordering process in Fe<sub>28</sub>Al and Fe<sub>28</sub>Al<sub>5</sub>Cr alloys was performed. Good correlation between the results of X-ray diffraction, Mössbauer spectroscopy and positron annihilation methods were obtained.
- Fe<sub>3</sub>Al phase of DO<sub>3</sub> type structure was found only in Fe<sub>28</sub>Al 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.
- Positron annihilation studies indicate lower concentration of vacancies in Fe<sub>28</sub>Al<sub>5</sub>Cr alloy samples in comparison with the samples of Fe<sub>28</sub>Al alloy. It can be the reason of slower long range ordering process in Fe<sub>28</sub>Al<sub>5</sub>Cr alloy.

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