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Defect structure of Fe-Al and Fe-Al-Ni metallic powders obtained by the self-decomposition method and intensive grinding in an electro-magneto-mechanical mill

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Abstract. The Fe-Al and Fe-Al-Ni metallic powders produced by the self-decomposition method and then intensive grinding in an electro-magneto-mechanical mill with Fe and Ni additions were examined by X-ray powder diffraction, scanning electron microscopy and Mössbauer spectroscopy. The concentration of vacancies and Fe atoms occupying Al positions (Fe-AS atoms) was determined from the Mössbauer spectra analysis connected with distinct Fe environments. The results show that nickel addition causes both an increase in vacancy concentrations in comparison with values found for Fe-Al metallic powders and a significant increase in the antisite Fe-AS atoms concentration. Intensive high-energy grinding in the electro-magneto-mechanical mill modifies the phase composition of the studied materials and changes the concentration of point defects.

Key words: iron aluminides • defects • point defects • Mössbauer spectroscopy • Fe-Al • Fe-Al-Ni

Introduction

Iron aluminides represent an intriguing class of materials; they offer a good combination of mechanical properties, specific weight/strength ratio, corrosion (and oxidation) resistance and low raw material cost [10], which makes them potential candidates for the substitution of stainless steel in applications at moderate to high temperature. It is well known that upon rapid quenching from elevated temperatures iron aluminides retain a high concentration of thermal vacancies which, frozen, increase their yield strength and hardness at room temperature [8]. However, the technical application of these alloys is, at present, restricted by poor ductility at low temperatures and low fracture toughness [9]. The development of new, more ductile, Fe-Al alloys depends on a thorough understanding of their properties implicating a better comprehension of the properties and behavior of defect in these materials. Experimental as well as theoretical studies suggest that iron aluminides present complex point defect, especially triple defect [7–10]. It is expected that the concentration of vacancies can be strongly changed in the aluminides with the variation of heat and mechanical treatment, together with the composition modification of the aluminides by transition metal ternary additives [7]. Alloying additions such as Ni, Cu, Mn increase the equilibrium hardness and only slightly affect the concentration of thermal vacancies [7–9]. Munroe [11] has studied the effects of Ni additions on FeAl mechanical properties for the composition $(\text{Fe}_{55-x}\text{Ni}_x)\text{Al}_{45}$ (where x is equal to 0.1–10 at.%) and has found that Ni affects the softening behavior of FeAl alloys by increasing the

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Table 1. The chemical and phase composition of the investigated metallic powders determined by qualitative and quantitative structural analysis with XRD and SEM (A – produced by the self-decomposition method; B – intensive grinding in an electro-magneto-mechanical mill with Fe and Ni additions)

Contents (at.%)	Fe-Al		Fe-Al-Ni					
	A	B	A			B		
			Contents (at.%)	Estimated phase composition	Percentage phase content in a sample (%)	Contents (at.%)	Estimated phase composition	Percentage phase content in a sample (%)
Fe	46.1	48.3	13.5	$\text{Fe}_{12}\text{Al}_{53}\text{Ni}_{35}$	65	26.3	$\text{Fe}_{36}\text{Al}_{52}\text{Ni}_{12}$	55
Al	53.9	51.7	53.8			54.2		
Ni			32.7	$\text{Fe}_{10}\text{Al}_{53}\text{Ni}_{37}$	35	19.5	$\text{Fe}_7\text{Al}_{54}\text{Ni}_{39}$	45

equilibrium hardness after annealing at low temperature (400°C, 120 h). The work of Schneibel, cited by Jordan in Ref. [8], has confirmed the increase in the equilibrium vacancy concentration by the addition of nickel. Schneibel has found that nickel concentrations of 2 vol.% can cause softening in FeAl at high temperature anneals by reducing the vacancy hardening and proposed two mechanisms: (i) formation of nickel/vacancy complexes, (ii) presence of small voids in FeAl with Ni. Pike *et al.* (also cited in [8]) have also studied the effect of nickel additions on the vacancy concentration in FeAl and have found that hardness and, therefore, vacancy concentration of $\text{Fe}_{45}\text{Al}_{1-x}\text{Ni}_x$ alloys increase with increasing temperature and nickel concentration, except for the initial decrease at low nickel concentrations for the 700 and 950°C samples.

In this paper, the Fe-Al and Fe-Al-Ni metallic powders produced by the self-decomposition method [1] and modified by intensive grinding in an electro-magneto-mechanical mill [2] with Fe and Ni additions were studied by Mössbauer spectroscopy, X-ray powder diffraction (XRD) and scanning electron microscopy (SEM). The ^{57}Fe Mössbauer spectra were analyzed by using a model according to which point defects in atomic shells close to the probe atom induce changes in the isomer shift and quadrupole interactions. The concentration of the vacancies and the Fe-AS antisite atoms was determined from the intensities of the corresponding sub-spectra in the Mössbauer analysis related to distinct Fe environments.

The results of solute nickel addition and intensive grinding with Fe and Ni additions on the defects structure in the investigated metallic powders Fe-Al-Ni are presented and discussed.

Experimental details

The investigated metallic powders Fe-Al and Fe-Al-Ni with an average grain size of about 50 μm were made by the self-decomposition method of the Fe-Al based alloys. Technical details of this method are described in [1]. The results of the chemical analysis presented in Table 1 indicated that samples are characterized by the aluminum concentration of over 50 at.%. X-ray powder diffraction and scanning electron microscopy revealed

that the Fe-Al samples are a single phase material with B2 structure, whereas the Fe-Al-Ni samples are a two-phase system in which the main phase is of FeAl(Ni) type and the second phase NiAl(Fe) contains iron as an impurity [5]. Then, the Fe-Al and Fe-Al-Ni powders were intensively ground in an electro-magneto-mechanical (EMM) mill [2]. In the EMM mill the ground powders are treated in a very specific and intensive way owing to several field forces operating simultaneously. The rotating magnetic field and the ferromagnetic needles as the grinding medium are the principal factors in the intensive grinding process. Several high-energy fields, i.e., electric, magnetic, acoustic and mechanical, operating simultaneously make the grinding process very intensive. The efficiency of the grinding type of mill is higher by tens to hundred times in comparison with ordinary mills. The grinding energy is concentrated in the space of the rotating grinding medium which is subjected to local pressure as high as 1 GPa [2]. In the paper, the 3.2 wt% Fe and 19.6 wt% Ni additions were added to samples obtained by the self-decomposition method and then intensive grinding in EMM mill with 20 s time. The grain size of ground powders determined as 60 μm was measured with laser analyzers of the Coulter type. The results of the chemical analysis, X-ray powder diffraction and scanning electron microscopy presented in Table 1 indicate that alloying additions (Ni, Fe) and intensive high-energy grinding in the EMM mill modified the chemical and phase composition of the Fe-Al and Fe-Al-Ni samples. All the phases that exist in the milled material contain high concentrations of aluminum (over 50 at.%) and have the ordered B2 structure. It is worth noticing that EMM milling produces only paramagnetic materials because ferromagnetic phases are trapped by the magnetic elements of the mill construction. For this reason, no equilibrium of the amount of Fe_3Al , Ni_3Al , FeAl_2 and Fe_2Al_3 intermetallic phases was detected. The measurements of the ^{57}Fe Mossbauer spectra were performed in transmission geometry by means of a constant spectrometer of the standard design. The spectra of the samples immediately after the process of self-decomposition and intensive high-energy grinding in the EMM mill were measured at room temperature. Some selected spectra which were obtained are presented in Fig. 1.

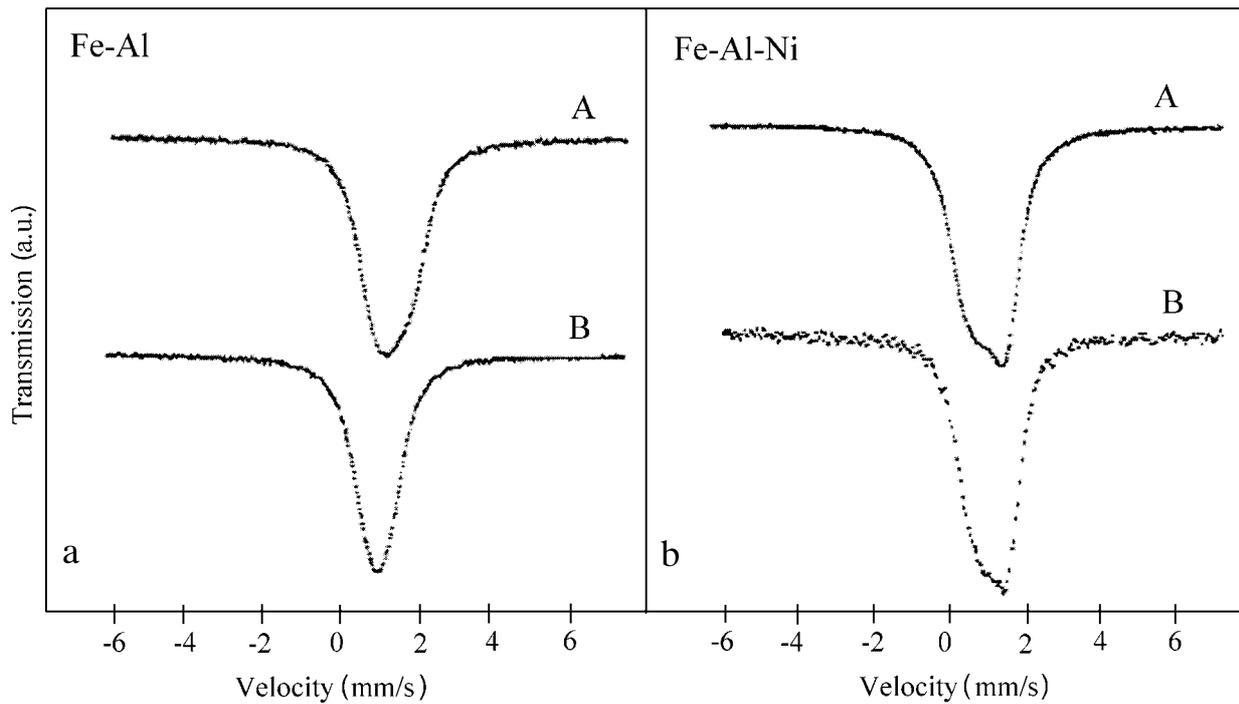


Fig. 1. The ^{57}Fe Mössbauer transmission spectra for the Fe-Al (a) and Fe-Al-Ni (b) metallic powders. A – produced by the self-decomposition method; B – intensive grinding in an electro-magneto-mechanical mill with Fe and Ni additions.

Results and discussion

The measured Mössbauer spectra were fitted based on the model proposed in papers [3, 9] taking into account the presence of point defects (vacancies and antisite atoms (Fe-AS) in the Mössbauer probe environments. According to this model, each crystal phase was characterized by four components describing the local environment of a ^{57}Fe nuclide. The first component (i) – is a single line and represents the ordered B2 structure. The second component (ii) – is a single line and represents the case when the Mössbauer Fe nuclide located in the corners of a cubic centred unit cell has in its environment a Fe atom found in the Fe-AS-corner. The third component (iii) – is a quadrupole doublet and represents an unresolved sextet and is connected to the case when a Fe atom is located in the Fe-AS position. The fourth component (iv) – being also a quadrupole doublet, defines the case where there is a vacancy in the Fe environment. Single phase samples of the FeAl type were analyzed with the usage of four

components, while samples of the Fe-Al-Ni series were investigated as two-phase systems with the dominant phase of the FeAl(Ni) type and the second AlNi(Fe) phase with low Fe concentration. The analysis of the two phase samples was carried out by fitting the two sub-spectra, each sub-spectrum was characterized by four components. The relative intensity ratio of the two sub-spectra was constrained by the ratio of the percentage content of the phases in the sample. The area ratio equal to $8 \cdot (1 - (1 - 2 \cdot c_{\text{Fe}}))$ between Fe corner atoms and Fe-AS atoms was used, where c_{Fe} is the concentration of Fe atoms [6].

The vacancy and antisite atoms concentrations determined by the applied procedure are shown in Table 2. The concentration of point defects in the investigated samples is higher as described in the literature [3, 4, 8]. This is related to their preparation by the self-decomposition method and easier generation of vacancies in Al-rich alloys caused by lowering of the energy of formation [3]. Vacancy formation and migration enthalpies have been measured in B2 FeAl and are

Table 2. Values of vacancy concentrations and antisite atom Fe-AS in the samples of Fe-Al and Fe-Al-Ni metallic powders determined with Mössbauer spectroscopy (A – produced by the self-decomposition method; B – intensive grinding in an electro-magneto-mechanical mill with Fe and Ni additions)

Investigated material	Estimated phase composition	Vacancy concentration (%)	Fe-AS concentration (%)
Fe-Al	A $\text{Fe}_{46}\text{Al}_{54}$	0.22	3.60
	B $\text{Fe}_{48}\text{Al}_{52}$	0.16	2.30
Fe-Al-Ni	A $\text{Fe}_{32}\text{Al}_{54}\text{Ni}_{14}$ $\text{Fe}_{12}\text{Al}_{53}\text{Ni}_{35}$	0.42	5.40
		0.62	4.70
	B $\text{Fe}_{36}\text{Al}_{52}\text{Ni}_{12}$ $\text{Fe}_7\text{Al}_{54}\text{Ni}_{39}$	4.60	0.31
		3.80	0.46

found to be in the ranges of 0.3–1.5 eV and are 1.0–1.7 eV, respectively [12].

The results of theoretical calculations [12] and experimental research [3, 4, 6–9, 11] indicate a high increasing of the concentration of point defects with increasing of aluminum concentrations. Those values of point defect concentrations determined for the Fe-Al-Ni samples are higher than those for the Fe-Al powders with similar concentration of Al atoms. This result revealed that Ni addition to FeAl aluminides significantly increases both the concentration of vacancies and Fe antisite atoms. When using the classification according to the preferred type of point defects applied in paper [9], the investigated materials belong to compounds of the TRD type characterized by triple defects, i.e. two vacancies and one antisite atom in the Fe sub-lattice. The influence of Ni additions in milled samples on the increase of the defect concentration is also observed. However, the determined concentrations are lower than for samples produced by the self-decomposition method. As it can be seen in Table 1, the samples after milling contain lower concentration of Al and Fe atoms than samples produced by the self-decomposition method. As it was noted earlier, strong dependence between the concentration of point defects and concentration of Al in aluminides takes place. Taking into account the fact that vacancies are formed mainly on the Fe-sites [6], relatively low concentration of Fe atoms in FeAl(Ni) and NiAl(Fe) phases detected in milled Fe-Al-Ni samples causes also decrease in the point defect concentration. Investigations performed in the paper lead to the conclusion that a significant increase of the point defect concentration in FeAl aluminides caused by Ni addition takes place in compounds with higher than $c_{Al} = 0.5$ concentration of Al atoms and relatively lower concentration of Ni atoms.

Conclusions

In the paper, the point defect concentration for the Fe-Al and Fe-Al-Ni metallic powders produced by the self-decomposition method and then intensive grinding in an electro-magneto-mechanical mill with Fe and Ni additions was determined by applying Mössbauer spectroscopy. It was found that the powders obtained by the self-decomposition method contain high concentrations of point defects. This is a result of lowering of energy formation of vacancies in aluminides together with the increase of aluminum concentration (over 50 at.%) and higher vacancy migration enthalpies. Additionally,

the results presented in the paper show distinctive changes in point defect concentrations caused by the presence of admixture atoms. Applied alloy additions modify the defect structure and the phase composition of the studied materials. Nickel added to FeAl compounds with B2 type structure causes the increase of vacancy and antisite atoms concentrations in comparison with values found for Fe-Al metallic powders. The significant increase of the point defects concentration is observed in compounds with higher than $c_{Al} = 0.5$ concentration of Al atoms and relatively lower concentration of Ni atoms.

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