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Mössbauer and structure studies on metallic powders from Fe-Al-X (X = Ni, Cu, Cr)

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ABSTRACT

Purpose: The Fe-Al-X and Fe-Al-Ni-X metallic powders produced by the self-decomposition process and then intensive grinding in an electro-magneto-mechanical mill with X = Fe, Ni, Cu, Cr additions was determined by applying the X-ray powder diffraction (XRD) and Mössbauer spectroscopy.

Design/methodology/approach: The X-ray diffraction and Mössbauer spectroscopy were applied to identify the phase composition of the studied materials. Ordering process was analyzed by X-ray diffraction methods and Mössbauer spectroscopy.

Findings: The effects of solute addition (X = Fe, Ni, Cu, Cr) in the investigated Fe-Al and Fe-Al-Ni metallic powders are presented and discussed. Good correlation between the results of X-ray diffraction and Mössbauer spectroscopy studies was obtained.

Research limitations/implications: The Mössbauer spectroscopy appeared to be usefull in the identification of phase composition in studied materials. Application of Rietveld refinement method enabled the verification of this composition.

Practical implications: The information on the phase transformation during the self-decomposition process and then intensive grinding in an electro-magneto-mechanical mill with additions are of prime importance for technological processing.

Originality/value: All the phases that exist in the milled material contain high concentrations of aluminium (over 50 at. %) and have the ordered B2 structure. Alloying additions (Ni, Fe, Cu, Cr) and intensive high-energy grinding in the EMM mill modified the chemical and phase composition of the Fe-Al-X and Fe-Al-Ni-X samples.

Keywords: Metallic alloys; Iron aluminides; Mössbauer spectroscopy; XRD

MATERIALS

1. 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 [1]. Above properties make 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 [2]. However, the technical application of these alloys is presently restricted by poor ductility at low temperatures and low fracture toughness [3]. 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 defects in these materials. Experimental as well as theoretical studies suggest that iron aluminides present complex point defects, especially triple defects [1-4]. 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 [4]. Alloying additions such as Ni, Cu, Mn increase the equilibrium hardness and only slightly affect the concentration of thermal vacancies [2-4, 7-14].

In this paper Fe-Al and Fe-Al-Ni metallic powders produced by the self-decomposition method [5] and modified by intensive grinding in an electro-magneto-mechanical mill [6] with Fe, Ni, Cu, Cr additions were studied by X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and Mössbauer spectroscopy. The results of solute Fe, Ni, Cu, Cr additions and intensive grinding on the structure in the investigated metallic powders are presented and discussed.

2. Experiment details

The investigated Fe-Al and Fe-Al-Ni metallic powders with the average grain size about 50 μm were made by the self-decomposition process of the Fe-Al based alloys. Technical details of this method are described in [5]. The chemical composition presented in Table 1 indicated that samples are characterized by the aluminum concentration above 50 at.%. Then, the Fe-Al-X and Fe-Al-Ni-X (X=Fe, Ni, Cu, Cr) powders were intensively ground in an electro-magneto-mechanical (EMM) mill [6]. 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 [6]. In the present work the 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 X-ray patterns were obtained using X-Pert diffractometer of Philips equipped with Soller slits both on incident and diffracted beams and with graphite monochromator on the diffracted beam. Copper radiation was applied.

3. Results and discussion

From the analysis of the X-ray diffraction patterns (Fig. 1 and Fig. 2) the following conclusion can be drawn: Fe-Al-X (X = Fe, Cu, Cr) powder contains FeAl(X) phase of B2 type structure and $Al(OH)_3$ phase and Cu. Metallic powders Fe-Al-Ni-X (X = Fe, Cu, Cr) contain two phases, both of B2 type structure: Fe(Ni)AlX and Ni(Fe)AlX enriched in Fe and Ni respectively.

Moreover $Al(OH)_3$ and Cu are also present. Shifting and broadening of diffraction lines indicate that each of the mentioned phases contain also addition atoms introduced during the grinding process.

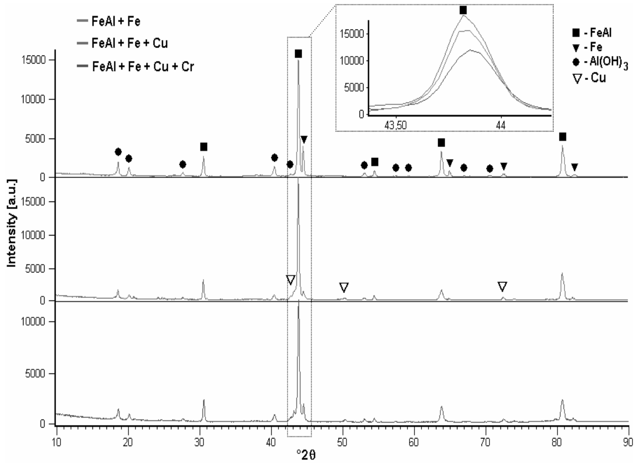

Fig. 1. X-ray patterns of sample the Fe-Al-X (X = Fe, Ni, Cu, Cr) metallic powders produced by the self-decomposition method and then intensive grinding in an electro-magneto-mechanical mill

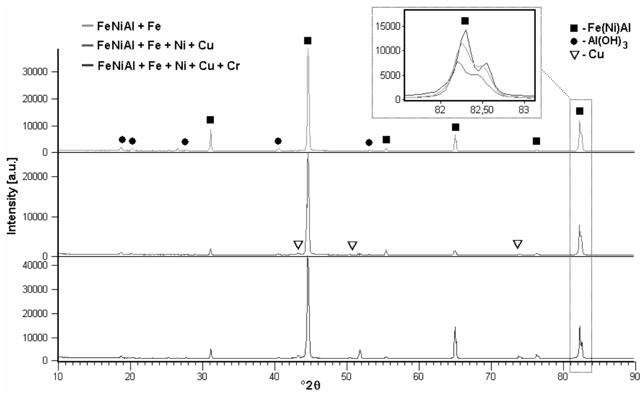

Fig. 2. X-ray patterns of sample the Fe-Al-Ni-X (X = Fe, Ni, Cu, Cr) metallic powders produced by the self-decomposition method and then intensive grinding in an electro-magneto-mechanical mill

Table 1.
The chemical composition of the investigated metallic powders

SAMPLES	PS-X X=(Fe, Ni, Cu, Cr) PS= $Fe_{46}Al_{54}$	PS*-X X=(Fe, Ni, Cu, Cr) PS*= $Fe_{37}Al_{52}Ni_{11}$
I	72g PS + 8g Fe	72g PS* + 8g Fe
II	57.5g PS + 8g Fe + 14.5g Cu	48g PS* + 16g Fe + 8g Cu+ 8g Ni
III	48g PS + 16g Fe + 8g Cu+ 8g Cr	50g PS* + 8g Fe + 7.5g Ni+8g Cu+ 6.5g Cr

PS- Fe-Al metallic powders produced by the self-decomposition method
PS*- Fe-Al-Ni metallic powders produced by the self-decomposition method

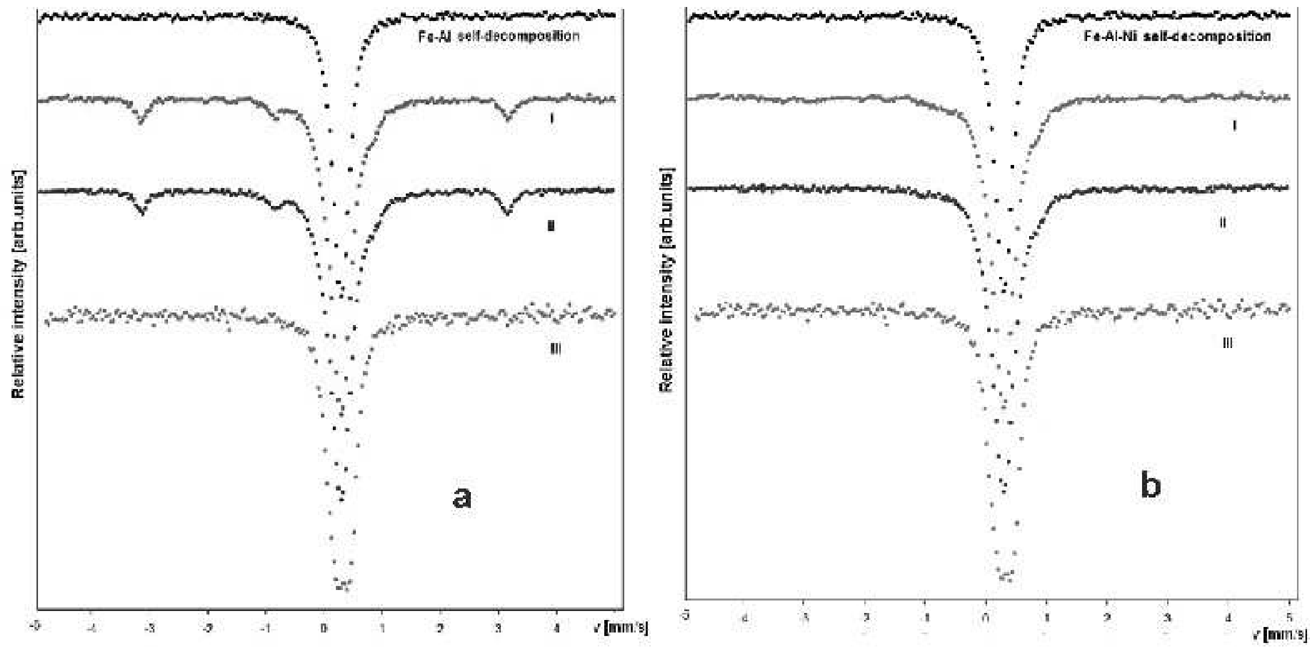

Fig. 3. The Mössbauer spectra of the samples of sample the a) Fe-Al-X and b) Fe-Al-Ni-X (X = Fe, Ni, Cu, Cr) metallic powders produced by the self-decomposition method and then intensive grinding in an electro-magneto-mechanical mill

In the case of both series of the studied metallic powders additional phase of $Al(OH)_3$, whose presence is probably connected with the technology of the self-decomposition process. Additionally, in both series of the studied samples copper atoms are not built into the matrix phase were identified. The process of powder grinding in the EMM mill in the presence of addition atoms makes it possible to fit / build additions into the matrix sublattice. As it was shown in the diffraction studies, as a result of the described process we obtain a multiphase material which, as literature data indicate, is characterized by better physicochemical and mechanical properties (considering application) [14-18].

The measurements of the ^{57}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}Co/Rh. The spectra of the samples were measured at room temperature. Obtained selected spectra are presented in Fig. 3 the Mössbauer spectra shape of the studied metallic powders after the addition and grinding process testifies to their multiphase composition.

Hyperfine parameters of the investigated spectra were related to the α-Fe standard. Experimental spectrum shape was described with a transmission integral calculated according to the numerical Gauss-Legandre's procedure. It made it possible to determine with high precision the value of the isomer shift, the quadruple splitting and the intensities of the fitted components.

Hyperfine parameters calculated on the basis of Mössbauer spectra analysis for e.g. isomer shift and quadruple splitting verify to the existence of phases identified in structural studies. Values of the calculated hyperfine parameters testify to the electron structure modification by the introduced addition atoms. Used as the initial material metallic powders obtained by the self-decomposition method are characterized by high aluminium concentration (54-56 at.%) [19]. Introducing as an addition Fe atoms as a result of the grinding process causes that the modified powders have the chemical composition approximate to the stoichiometric composition, which is testified by the calculated isomer shift. The introduced addition atoms for e.g. nickel, copper and chromium modify the chemical composition and the electron structureof the base metallic powders [20]. The evidence for that are changes of IS and QS values. The values of the isomer shift IS on the aluminum and Ni concentration for the components describing in the local environment of a Mössbauer nuclide.

In general, electron charge density on the iron nucleus decreases upon alloying Ni and Cu into αFe as the average isomer shift increases with increasing Al, Ni and Cu concentration. Nickel and cupper impurities located in distant shells cause a decrease of this charge density. On the other hand, effect due to a Cr impurities located in the Fe environment tends to increase of charge density. The evolution of the values of quadruple splitting testifies to the symmetry disturbance in the environment of the Mössbauer nuclide and thus testifies to the presence of the addition atoms introduced by grinding. A similar influence of copper, nickel and chromium atoms on the hyperfine parameters values was stated by the authors of the work [13].

4. Conclusions

- Powders obtained by the self-decomposition method contain FeAl(X) phase of B2 type structure and $Al(OH)_3$ and Cu phases are also present.
- Fe-Al-Ni-X metallic powders contain two phases, both of B2 type structure: Fe(Ni)AlX and Ni(Fe)AlX enriched in Fe and Ni, respectively. Moreover this powder contain also $Al(OH)_3$ and Cu phases.

- All the phases that exist in the milled material contain high concentrations of aluminum (above 50 at. %) and have the ordered B2 structure. Alloying additions (Ni, Fe, Cu, Cr) and intensive high-energy grinding in the EMM mill modified the chemical and phase composition of the Fe-Al-X and Fe-Al-Ni-X samples.

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