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Point defects in the B2-phase region of the Fe-Al system studied by Mössbauer spectroscopy and X-ray diffraction

Aneta Hanc-Kuczkowska

Abstract. In this work Mössbauer spectroscopy and X-ray powder diffraction was used to study of point defects formation in intermetallic phases of the B2 structure of the Fe-Al system as a function of Al concentration. The results are compared with the concentrations of point defects determined from positron annihilation data. The values of the ^{57}Fe isomer shift and quadrupole splitting for the components describing the point defects in the local environment of a Mössbauer nuclide are presented. The concentration of the Fe vacancies and Fe atoms substituting Al (i.e. anti--site atom, Fe-AS) are determined. The results show that an increase in Al content causes an increase in vacancy and Fe-AS concentrations.

Key words: iron aluminides • Mössbauer spectroscopy • point defects • X-ray diffraction

Introduction

Iron aluminides represent an intriguing class of new materials: they offer a good combination of mechanical properties, specific weight/strength ratio, corrosion and oxidation resistance and low raw material cost [1, 3–9], which makes them potential candidates for the substitution of stainless steel in applications at moderate to high temperatures. The extensive technological application of iron aluminides, however, is impaired by their low room temperature tensile ductility. This is attributed to extrinsic (environmental embitterment) or intrinsic (low grain boundary cohesion) mechanisms, with the dominant mechanism depending on the aluminum content of the alloy. 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 [1, 3–13], suggest that point defects in iron aluminides present complex, especially triple defect structure. 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 [1, 3–9]. It is expected that the concentration of point defects can be strongly affected by the variation of Al content and the composition modification of the aluminides by transition metal ternary additives [3].

In this paper, Mössbauer spectroscopy and X-ray powder diffraction (XRD) are used in a study of point

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Table 1. Chemical compositions of the investigated material

Contents (at.%)	I	II
Fe	61.64	54.64
Al	38	45
Additions	Mo-0.20; Zr-0.05; C-0.1; B-0.01	

defects formation in intermetallic phases of the B2 structure of the Fe-Al system. The investigated samples are Fe-Al (Al content below 50 at.%) with small additives. Mössbauer spectra are analyzed with a model [2], according to which the vacancies and Fe atoms substituting Al (Fe-AS) in atomic shells close to the probe atom, influence the isomer shift and quadrupole splitting of particular spectrum components. The concentrations of point defects are determined from the intensities of these components and are correlated with the changes of Al content.

Experimental details

The chemical compositions of the investigated samples are presented in Table 1. The samples were obtained from Armco iron, aluminum of 99.99% purity, and a small amount of other additives added in order to improve the thermal and mechanical properties of al-loys. The samples were prepared by melting in spinel $Al_2O_3 \times MgO$ crucibles in an induction furnace in a vacuum of 10^{-2} Torr. The ingots were re-melted three times to insure homogeneity and annealed in a vacuum furnace for 48 h and then cooled down slowly with the furnace.

Phase analysis was carried out by applying X-ray diffraction using an X-ray Philips diffractometer equipped with a graphite monochromator. The $CuK\alpha$ radiation was used. The samples were rotated during X-ray data collection. The ^{57}Fe Mössbauer spectra were measured in transmission geometry at room temperature by means of a constant-acceleration spectrometer of the standard design. The 14.4 keV gamma rays were provided by a 50 mCi source. 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 which enables determination of real intensities of the fitted components.

Results and discussion

The X-ray diffraction patterns of $Fe_{62}Al_{38}$ and $Fe_{55}Al_{45}$ samples are presented in Fig. 1. The features of B2-type structure prevail in the diffraction spectra. Lattice con-
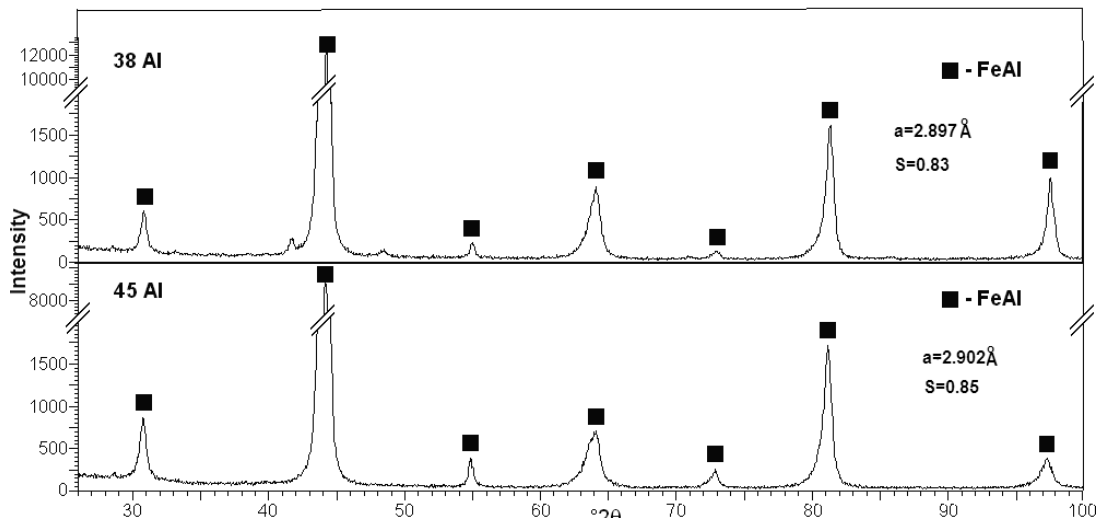
Fig. 1. The X-ray diffraction patterns of $Fe_{62}Al_{38}$ and $Fe_{55}Al_{45}$ samples annealed at 1000°C for 48 h.
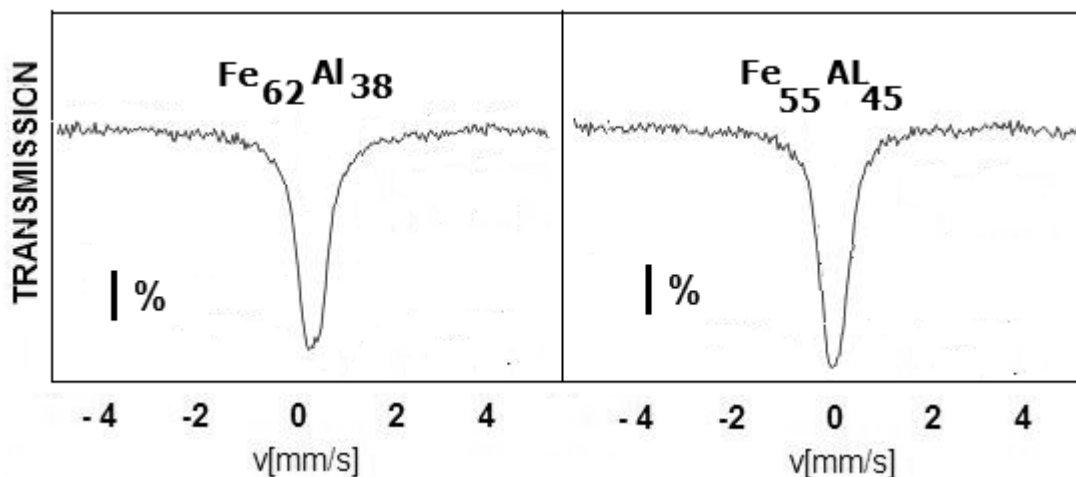
Fig. 2. The Mössbauer spectra for $Fe_{62}Al_{38}$ and $Fe_{55}Al_{45}$ alloys annealed at 1000°C for 48 h.

Table 2. Values of hyperfine parameters (IS, QS); Γ – line width and A – subspectra area

Samples	Component	IS*,a (mm/s)	QS* (mm/s)	Γ (mm/s)	A (%)
$Fe_{62}Al_{38}$	L-I	0.19		0.27	83
	L-II	0.06		0.34	14
	Q-III	0.04	0.25	0.36	1.9
	Q-IV	0.23	0.13	0.29	1.1
$Fe_{55}Al_{45}$	L-I	0.24		0.25	85
	L-II	0.07		0.32	11.7
	Q-III	0.05	0.22	0.34	2.1
	Q-IV	0.21	0.17	0.26	1.2

* Uncertainty estimated from the fitting procedure is equal to ±0.005.
a Relative to the α-Fe foil at room temperature.

Table 3. Values of vacancy and anti-site atom Fe-AS concentrations in the samples of Fe-Al determined with Mössbauer spectroscopy investigations

Estimated phase composition	Vacancy concentration V_{Fe} (%)*	Concentration of Fe-As (%)**
$Fe_{62}Al_{38}$	0.04	1.9
$Fe_{55}Al_{45}$	0.05	2.1

* Uncertainty estimated is equal to ±0.006.
** Uncertainty estimated is equal to ±0.05.

stant parameters – *a* and long-range order parameters – *S* determined by the Rietveld refinement method show the tendency to increase with increasing in aluminum content in the samples.

The Mössbauer spectra are presented in Fig. 2. All Mössbauer spectra were fitted with the model proposed by Bogner *et al.* [2]. According to this model, the spectrum contains four components describing different local environments of the ^{57}Fe nuclide. The first component (I) – a single line – represents an ordered B2 structure. The second component (II) – a single line (which approximates an unresolved quadrupole doublet) – relates to the case when the Mössbauer Fe nuclide is located in a corner of the cubic centered unit, and an Fe-AS atom is situated in the centre of this unit. The third component (III) – a doublet of lines (which approximates an unresolved Zeeman sextet) – corresponds to a Fe atom located in the Fe-AS position. The fourth component (IV) – also a quadrupole doublet of lines – represents the case where there is a vacancy in the near Fe surrounding.

The evolution of the values of the isomer shift (IS) and quadrupole splitting (QS) of the spectra components depending on the aluminum concentration are present in Table 2. Similar values of the IS and QS for the components describing the ordered B2 structure and the point defect were observed in theoretical calculations [11, 12] and experimental research [2, 11].

The values of vacancy and anti-site atoms concentrations found using the described model are shown in Table 3. To estimate the value for the concentration of vacancies on the Fe sublattice, the intensity of a subspectrum was divided by 26 [2]. The obtained values of concentrations of vacancies and anti-site atoms (Fe-AS) show an increase with Al content, which confirms the results of theoretical calculations [11, 12] and some experimental data [2–13]. According to the literature [1, 3–9], vacancies in the Fe sublattice V_{Fe} are the dominant type of defects in Fe-Al system (maybe organized in triple defects, i.e. two vacancies and an anti-site atom [1, 3–13]).

The values of vacancies concentrations in the samples containing micro-additions, estimated in this work, are slightly lower then in vacancies concentra-

tions in Fe_xAl_{1-x} ($x > 0.5$) alloys, calculated in theoretical papers [11, 12] and than some experimental data [2–7]. We connect this result with a defect and electron structure modification in the examined materials by the micro-additions. Such a character of the defect structure, mainly the lowered concentration of vacancies in the alloys modified with micro-additions, confirms the advisability of their introduction in order to improve plasticity of these materials.

Conclusions

Point defects concentrations for the intermetallic compounds of Fe-Al system were determined applying Mössbauer spectroscopy. It was found that the investigated materials contain high concentrations of point defects, which significantly increases with increasing in aluminum content. The values of vacancies concentrations in the Fe_xAl_{1-x} ($x > 0.5$) samples containing micro-additions, estimated in this work, are slightly lower than vacancies concentrations in Fe_xAl_{1-x} ($x > 0.5$) alloys determined by other authors. Such a character of the defect structure – mainly the lowered concentration of vacancies in the alloys modified with micro-additions confirms the advisability of their introduction in order to improve plasticity of these materials.

References

1. Baker I, Munroe PR (1990) High-temperature aluminides and intermetallics. In: Whang SH, Liu CT, Pope DP, Stiegler

JD (eds) Proc of the Symp on High Temperature Aluminides and Intermetallics, 1–5 October 1989, Indianapolis, USA. The Materials Society, Warrendale, PA, pp 95–114

2. Bogner J, Steiner W, Reissner M *et al.* (1998) Magnetic order and defect structure of FexA$_{1-x}$ alloys around x = 0.5: An experimental and theoretical study. Phys Rev B 58;22:14922–14933

3. Gianella S, Brua RS, Deng W, Marino F, Spataru T, Principi G (2001) Defect structures in FeAl B2 alloys. J Alloys Compd 317/318:485–490

4. Hanc A, Dercz G, Frąckowiak JE, Pająk L, Binczyk F (2004) Structural studies on metallic powders from Fe-Al and Fe-Al-X (X = Cu; Ni; Cr) systems obtained by self--decomposition method. In: Morawiec H, Stróż D (eds) Proc of the XIX Conf on Applied Crystallography, 3–7 September 2003, Kraków, Poland, pp 312–316

5. Hanc A, Frąckowiak JE (2004) Defect structure of Fe-Al and Fe-Al-X (X = Ni; Cu; Cr) metallic powders obtained by the self-decomposition method. Nukleonika 49;Suppl 3:S7–S11

6. Hanc A, Frąckowiak JE (2006) 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. Nukleonika 51;Suppl 3:S24–S28

7. Hillert M, Selleby M (2001) Point defects in B2 compounds. J Alloys Compd 329:208–213

8. Jordan JL, Deevi SC (2003) Vacancy formation and effects in FeAl. Intermetallics 11:507–528

9. Kansy J, Hanc A, Giebel D (2011) Influence of heat treatment on ordering process in Fe72Al28 alloy studied by Mössbauer spectroscopy. Acta Phys Pol A 119:44–47

10. Kogachi M, Haraguchi T, Kim SM (1998) Point defect behavior in high temperature region in the B2-type intermetallic compound FeAl. Intermetallics 6:499–510

11. McKamey CG (1996) Iron aluminides. In: Stoloff NS, Sikka VK (eds) Physical metallurgy and processing of intermetallic compounds. Chapman & Hall, New York, Chapter 9, pp 351–392

12. Michalecki T, Deniszczyk J, Frąckowiak JE (2004) First principles study of the isomer shift in Fe44M6Al50 (M = Ti, V, Cr, Co, Ni, Cu) alloys with B2 structure. Nukleonika 49;Suppl 3:S3–S6

13. Ren X, Otsuka K (2000) A unified model for point-defect formation in B2 intermetallic compounds. Philos Mag A 80:467–491