Functional Materials 12, No.1 (2005)

X-ray diffraction study of atomic long-range order of Fe–Al alloy

S.P.Repetsky, R.O.Yaremko

Physics Department, T.Shevchenko Kyiv National University,
6 Acad. Glushkov Ave, 03022 Kyiv, Ukraine

Temperature dependence of nonequilibrium values of Fe–Al alloy long-range order parameter has been studied using X-ray diffraction by comparing the integrated intensity of superstructure reflection and fundamental one at small Bragg angles. An additional atomic occupancy of corresponding sublattice sites has been shown to occur as the annealing temperature increases. A partial disordering of the alloy was provided by mechanical polishing with diamond paste; it was manifested itself as a decreasing of superstructure reflection intensities. Such behavior of the long-range order parameter of Fe-50 at. % Al alloy shows that the second order phase transformations are typical of that alloy.

Рентгенографически исследована температурная зависимость неравновесных значений параметра дальнего атомного порядка сплава Fe-50 ат.% Al по отношению интегральных интенсивностей надструктурного к структурному отражений на малых брегговских углах. Обнаружено, что с повышением температуры отжига происходит дополнительное заполнение атомами узлов "своих подрешеток". Частично разупорядоченное состояние было достигнуто механическим полированием сплава, что наблюдалось по уменьшению интенсивностей надструктурных рефлексов. Такое поведение параметра дальнего порядка свидетельствует о том, что для сплава Fe-50 ат.% Al характерны фазовые превращения второго рода.

Aluminides of the 3*d* transition metals (Fe, Co, Ni) exist within a wide concentration range. High corrosion and temperature stability, as well as interesting magnetic properties make it possible to use those alloys as high-temperature structural materials and soft magnetic ones. The crystal structure of B2 (CsCl) type is one of the simplest and most investigated ordered structures. The equiatomic alloy FeAl (β-phase) is crystallized in this structure. The phase diagram of Fe–Al system is shown in Fig. 1. The point of the order-disorder transition lies above the melting point and is situated near 1723°C. This alloy belongs to systems where second order phase transformations are characteristic due to monotonous change of the long-range order parameter to the Kurnakov point.

A number of works deal with the crystal structure, atomic ordering, and other physical properties of equiatomic FeAl alloy. The character of the above described properties for Fe-50 % Al alloy was theoretically studied in [1]. Various trends of atomic ordering in paramagnetic and ferromagnetic states of stoichiometric Fe–Al alloys were studied using the generalized perturbation theory of density functional (linear "muffin-tin" orbitals), that is described in [2]. In [3], specific resistance, thermal capacity, magnetic properties of β-phase of Fe–Al system were studied for alloys containing 30 to 52 at. % of Al. It is possible to conclude from results of these experiments that the atomic ordering influences the physical properties of that alloy and vice versa. It was found that at short-range ordering of the Fe–Al alloy, there is a characteristic dip in the curve of energy dependence of electronic states density which results in an energy gap occurrence as a long-range ordering is established [4–5]. But the energy band splitting and changes in electronic properties of Fe–Al alloy caused by that split at long-range ordering are not studied

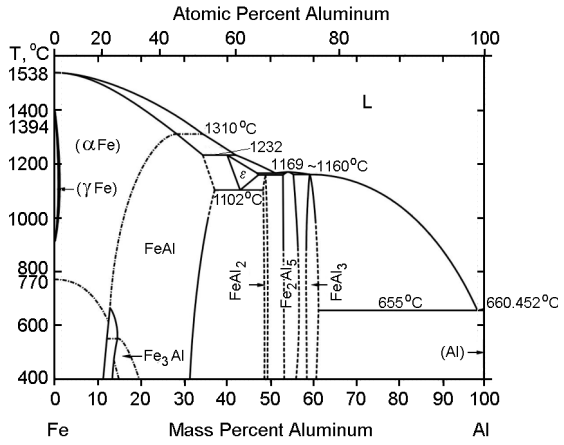

Fig. 1. Phase diagram of Fe–Al system.

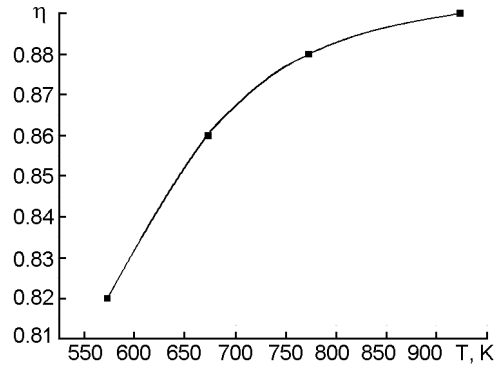

Fig. 2. Dependence of long-range order parameter on the annealing temperature of Fe–Al alloy.

sufficiently in experiment. In this connection, this work is devoted to the variation of the long-range order parameter (obtained by heat treatment and mechanical polishing) for Fe-50 % Al alloy.

The samples of Fe-50 at.% Al alloy were melted in a melting-pot furnace under argon atmosphere at 1200°C during 24 h. Since Fe and Al differ in physical properties, the ingots were ground and then remelted once again in the melting-pot furnace during 12 h for homogenization. For X-ray measurements, thin specimens ($10\times10\times1$ mm^3) were machined and then polished. The stoichiometry of Fe–Al alloy was checked by fluorescent analysis which has shown the composition 50.09 at. % Fe and 49.91 at. % Al. An X-ray diffractometer DRON-3 with monochromatic emission of a cobalt tube ($\lambda = 1.79$ Å) was used for experiments. The X-ray patterns were scanned at small angles to study the long-range order under reduced various angle-dependent factors. Then, the integrated intensity of fundamental and superstructure reflections were compared. The determination procedure of the long-range order parameter is described in [6–8]. The necessary reference data were taken from [6, 9]. The main formula used for the long-range order parameter calculation was taken from work [8]:

$$\frac{I_s}{I_f} = \frac{(LP)_s}{(LP)_f}\,\frac{e^{-2M_s}}{e^{-2M_f}}\,\frac{p_s}{p_f}\,\frac{|f_{\mathrm{Fe}} - f_{\mathrm{Al}}|^2\eta^2}{4|c_{\mathrm{Al}}f_{\mathrm{Al}} + c_{\mathrm{Fe}}f_{\mathrm{Fe}}|^2},$$

where (LP) the Lorentz polarization factor; e^{-2M}, the Debye-Waller factor; f_{Fe}, f_{Al}, the atomic scattering factors of Fe and Al; c_{Al}, c_{Fe}, the concentrations of alloy components; η, the long-range order parameter; I_s, I_f, the superstructure and fundamental reflection intensity, respectively, p-the repeatability factor.

It was of interest to investigate the ordering change depending on the system internal state, therefore, the alloy was annealed at 300, 400, 500, and 650°C for three hours each time followed by slow cooling down to room temperature with the purpose to attain a high ordering extent. The recrystallization temperature of the alloy is about 700°C, therefore, to avoid the result distortion (growth of a single crystal), it is just the above heat treatment regime that was chosen. Since the Fe–Al (50 at. % Al) alloy is brittle, it was hard to quench the ingot without fracture of the sample. That is why the oscillation of the long-range order extent was examined depending on mechanic and heat treatment of the sample (nonequilibrium values of the long-range order parameter).

One of the methods providing a partial reducing of atomic ordering is ball milling. In our case, it is mechanical polishing with diamond paste (removing approximately several tens of micrometers from the surface), because this alloy remains a high-ordered up to melting point. For the alloy under study, the layer involved in formation of the X-ray diffraction picture is approximately 20 μm thick, therefore, in our opinion, it is the partially disordered region that is investigated. Temperature dependence of a long-range order parameter is shown in Fig. 2 (for the disordered state, that parameter was found to be $\eta = 0.74$).

It is seen that thermomechanical treatments of the alloy influence insignificantly the numerical value of long-range order parameter. The annealing promotes a gradual

filling of sites in two sublattice by "own atoms "resulting in increasing intensity of superstructure reflection. The reduction of the long-range ordering extent at mechanical polishing is caused by generation of antiphase boundaries in near-surface layers due to mechanical stresses as well as by point defects arising along the superdislocation shear zone. The distortion of correct bonds along antiphase boundaries is extended over several atomic planes. All these considerations can explain the nature of the reduction of the long-range order parameter in Fe-50 at.% Al alloy.

To conclude, the behavior of a long-range order parameter by X-ray diffraction method for Fe-50 at. % Al alloy in different states was experimentally studied. The long-range order extent increases with the annealing temperature (at constant annealing duration) and has the following values: 0.82 (300°C), 0.86 (400°C), 0.88 (500°C), and 0.89 (650°C). For the partially disordered alloy, the value $\eta = 0.74$ has been obtained. The observed changes in the long-range order parameter of the studied equiatomic alloy can be explained by studying its other physical properties (heat capacity, electric resistance, magnetization). Those changes agree well with theoretical electronic structure researches of the Fe-50 at.% Al alloy.

References

1. E.S.Kulkova, V.E.Egorushkin et al., *Phys. Stat. Sol.(B)*, **103**, 653 (1981).
2. S.K.Bose, V.Drchal, J.Kudrnovsky et al., *Phys. Rev. B*, **55**, 184 (1997).
3. A.N.Bashkarov, F.A.Sidorenko, L.P.Zelenin et al., *Fiz.Met.Metalloved.*, **32**, 569 (1971).
4. H.Hasegawa, J.Kanamori, *J. Phys. Soc. Jap.*, **33**, 1607 (1972).
5. V.F.Los, S.P.Repetsky, Methods of Disordered Systems Theory, Naukova Dumka, Kiev (1995) [in Russian].
6. S.S.Gorelik, L.N.Rastorguev et al., X-ray Diffraction and Electron Microscopy Analysis: A Practical Guide, Metallurgia, Moscow (1970) [in Russian].
7. V.I.Iveronova, G.P.Revkevich, Theory of X-ray Scattering, MGU Publ., Academic Press, Moscow (1978) [in Russian].
8. M.Kompatscher, B.Schonfeld, H.Heinrich, *Acta Materialia*, **51**, 165 (2003).
9. L.I.Mirkin, Handbook on X-ray Structure Analysis of Polycrystals, Academic Press, Moscow (1961) [in Russian].

Рентгенографічне дослідження атомного далекого порядку в сплаві Fe–Al

С.П.Репецький, Р.О.Яремко

Рентгенографічно досліджено температурну залежність нерівноважних значень параметра атомного далекого порядку сплаву Fe-50 ат.% Al через відношення інтегральних інтенсивностей надструктурного до структурного відбиттів на малих бреггівських кутах. Виявлено, що із зростанням температур відпалу відбувається додаткове заповнення атомами "своїх вузлів" у гратці сплаву. Часткове розвпорядкування сплаву було досягнуто механічним поліруванням, що спостерігалося через зменшення інтенсивностей надструктурних рефлексів. Така поведінка параметра далекого порядку свідчить про те, що в сплаві Fe-50 ат.% Al відбуваються фазові переходи другого роду.