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Local Structure of Fe–Cr Powders Prepared by Mechanical Alloying

Yu. V. Baldokhin[a] **and V. V. Cherdyntsev**[b, *]

[a]*Semenov Institute of Chemical Physics, Russian Academy of Sciences, ul. Kosygina 4, Moscow, 119991 Russia*
[b]*Moscow Institute of Steel and Alloys (National University of Science and Technology), Leninskii pr. 4, Moscow, 119991 Russia*
**e-mail: vvch@misis.ru*

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Abstract—We have studied the structure of Fe–Cr alloys prepared by mechanical alloying. According to X-ray diffraction data, all of the alloys are single-phase and consist of a BCC solid solution. The local structure of the alloys has been studied by Mössbauer spectroscopy. Analysis of the local environment of the Fe atoms leads us to assume clustering processes in the alloy containing 20 at % Cr and suggests that the alloys containing 35 and 50 at % Cr experience phase separation into regions of a homogeneous disordered chromium-enriched solid solution and an iron-enriched imperfect structure containing a considerable amount of dissolved gases.

Keywords: alloys, structure, Mössbauer spectroscopy, solid solutions, ordering, phase separation

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INTRODUCTION

The iron–chromium system is basic to a considerable number of materials of practical importance, including reactor steels, electronic alloys, and protective coatings. An important feature of this system is that there is low-temperature phase separation into two solid solutions, even though both iron and chromium have body-centered cubic lattices, with similar lattice parameters [1]. The effect is of practical interest, because the precipitation of second-phase clusters has a significant effect on the properties of alloys and their stability to external influences. Because of this, the study of phase separation processes and clustering in the Fe–Cr system has recently been the subject of considerable attention [2, 3].

The use of mechanical alloying (MA) for the preparation of metallic materials is of interest primarily because this method offers the possibility of producing highly imperfect structures and metastable phases. The formation of solid solutions in the Fe–Cr system as a result of MA was first detected by Pavlyukhin et al. [4] by analyzing changes in Mössbauer spectra of milled powders. They claimed that MA in this system was accompanied by the formation of a γ-phase with a face-centered cubic lattice [4].

To date, MA in the Fe–Cr system has been the subject of rather extensive research. Most studies dealt with individual compositions, in particular, with those containing 10 [5, 6], 20 [7], 30 [8], 40 [9–12], 50 [13], 70 [14–19], and 80 at % chromium [20]. Several studies [21–24] were concerned with MA Fe–Cr materials in a wide composition range, but they contained no detailed analysis of Mössbauer spectroscopy data.

The purpose of this work is to analyze in detail the structure of MA materials in the Fe–Cr system in a wide composition range using Mössbauer spectroscopy data.

EXPERIMENTAL

The starting materials used in this study were powders of hydrogen-annealed carbonyl iron of 99.95% purity in the form of powder with a particle size from 5 to 10 μm and chromium of 99.9% purity in the form of powder with a particle size from 10 to 20 μm. We studied $Fe_{100-x}Cr_x$ samples with $x = 5$, 10, 20, 35, 50, and 90 at %. The powders were processed in an AGO-2U planetary ball mill in hermetically sealed vials in air using 8-mm-diameter ShKh15 steel balls. The total ball weight was 200 g and the powder weight was 20 g. The rotation rate of the supporting disk of the mill was 1200 rpm. As shown previously using calculations and experimental data [25], processing under such conditions corresponds to a specific power of ~30 W/g, and the temperature in the vial of the mill is then ~450°C. All of the samples were milled for 1 h. As shown earlier, under these processing conditions the time needed for MA in similar systems containing solid solutions in a wide composition range does not exceed 30 min. During further milling, the structure and phase composition of the alloys remain unchanged, as evidenced by both X-ray diffraction and Mössbauer spectroscopy data [26–28].

Table 1. X-ray diffraction results for the MA Fe–Cr materials

at % Cr	*a*, nm	*D*, nm	ε, %
5	0.2871	19.6 ± 3.0	0.56 ± 0.06
10	0.2872	17.2 ± 4.0	0.57 ± 0.06
20	0.2878	16.8 ± 3.0	0.56 ± 0.06
35	0.2877	16.7± 2.5	0.56 ± 0.06
50	0.2876	16.3 ± 2.5	0.54 ± 0.06
90	0.2885	17.2 ± 4.0	0.66 ± 0.07

The phase composition of the samples was determined by X-ray diffraction on a DRON-3 diffractometer (CoK_α and CuK_α radiations). For the $Fe_{50}Cr_{50}$ sample, we evaluated the time needed for a single-phase state to be reached: from X-ray diffraction data, it was determined to be 7.5 min. Thus, the processing time chosen is quite sufficient for the MA process in the system under investigation to reach completion.

The samples were characterized by Mössbauer spectroscopy in transmission geometry at a Mössbauer gamma-ray energy of 14.4 keV. We used a ^{57}Co(Rh) gamma ray source and a Persei constant acceleration Mössbauer spectrometer with laser calibration and source velocity stabilization. The measurements were performed at room temperature. The velocity scale was calibrated against Armco iron. The Mössbauer spectra were decomposed into their components by fitting with Voigt line profiles [29].

RESULTS AND DISCUSSION

The X-ray diffraction data are summarized in Table 1. According to X-ray diffraction data, all of the samples under investigation are single-phase and have a BCC (*A*2) structure. Comparison of their lattice parameters with reference data [30] indicates that, throughout the composition range studied, the lattice parameters of the MA materials exceed those of cast alloys. The present results agree well with data reported by Jithender and Gopikrishna [24], which demonstrate that the lattice parameter of MA Fe–Cr materials considerably exceeds that of cast alloys over the entire composition range. An increase in lattice parameter was observed for both the mechanical activation of pure metals [31–33] and the MA in binary systems [24, 34, 35]. Data reported by Lemoine et al. [22] suggest that an increase in lattice parameter can be caused by oxygen dissolution in the lattice, without formation of oxides.

The crystallite size (the size of coherent X-ray scattering domains) *D* of the samples under investigation is independent of their composition and ranges from 15 to 20 nm. The lattice strain ε in the samples ranges from 0.55 to 0.7%. These results are well consistent with data reported by Delcroix et al. [17] for an MA material containing 70 at % Cr. The *D* values obtained in this study slightly exceed those typical of MA materials in the system under investigation: 5–10 nm [6–8, 12, 22]. One possible reason for the observed distinctions is that the mechanical activation processing intensity in the water-cooled planetary mill used in this study is higher than that in typically used air-cooled mills, respectively, and the temperature in the vials is also higher [36]. The higher milling temperature in our case may lead to recrystallization processes, which result in a slight increase in *D*. Any case, it is reasonable to conclude that, in a wide composition range, MA in the Fe–Cr system leads to the formation of an X-ray uniform BCC structure with a high density of lattice defects.

Figure 1 shows Mössbauer spectra of the samples under investigation. The unfolding parameters of the spectra are presented in Table 2. The spectra of the samples containing 10 at % chromium or less consist of several sextets, corresponding to different numbers of chromium atoms in the nearest neighbor environment of iron atoms [37]. The spectra are similar in shape to those reported by Pavlyukhin et al. [4] and Pandey et al. [6] for MA materials with similar compositions. The spectrum of the sample containing 20 at % chromium shows, in addition to sextets, a small central component (singlet). The general shape of the spectrum of this sample is similar to that reported previously [7, 21–23] for samples with this composition. In those studies, the spectra were not completely unfolded, so it is impossible to ascertain whether they contained a central component, given that it is usually very weak. The sample containing 35 at % chromium has a noticeable central component, which can be unfolded into a singlet and doublet. The Mössbauer spectrum reported by Bentayeb et al. [8] for an MA material containing 31 at % Cr also has a prominent central component.

The Mössbauer spectrum of the sample containing 50 at % chromium was unfolded like that of the above sample. The overall fraction of the central component in the total area was found to exceed 20%. Analysis of data in the literature indicates that the shape of the Mössbauer spectrum of MA materials containing 40–60 at % chromium depends significantly on the milling atmosphere. After milling in an atmosphere containing O_2 and/or N_2 (air [11], insufficiently pure argon [9, 10, 12, 23], or nitrogen [21]), the Mössbauer spectrum of the MA materials consists entirely or almost entirely of a singlet line. If the processing atmosphere during MA is sufficiently protected from O_2 and N_2 (vacuum [10, 11] or preevacuation of the vial before introduction of argon [23]), the Mössbauer spectrum has the form of a set of sextets. According to Findiki et al. [9] and Fnidiki et al. [23], an MA material prepared in the presence of O_2 and N_2 has a nearly

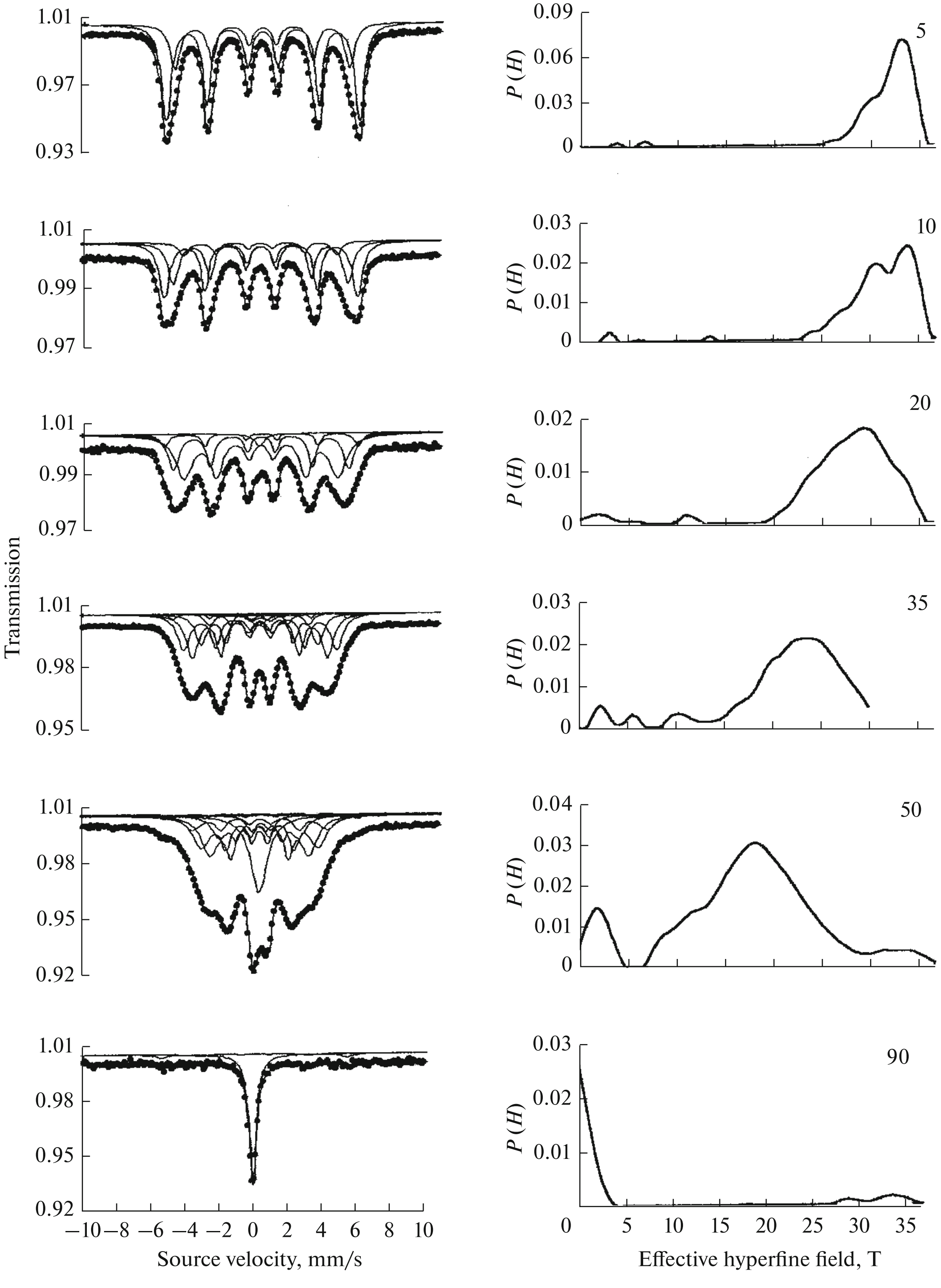

Fig. 1. Mössbauer spectra and the corresponding hyperfine field distributions $P(H)$ of the samples. The Cr content of the samples (5–90 at %) is indicated at the curves.

amorphous defect structure, which is paramagnetic, unlike the structure of an MA material prepared in an inert atmosphere.

The Mössbauer spectrum of the sample containing 50 at % chromium is similar to those obtained in previous studies [10, 11, 21, 23], where milling was per-

Table 2. Parameters of the Mössbauer spectra of the $Fe_{100-x}Cr_x$ alloys

x	Component	δ, mm/s	Δ, mm/s	H_{eff}, kOe	Γ, mm/s	$S(k)$, % (meas)
5	Sextet 1	0.01	0.01	336	0.52	67.6
	Sextet 2	–0.01	0.00	304	0.54	32.4
10	Sextet 1	0.01	0.00	336	0.51	50.2
	Sextet 2	–0.01	0.00	304	0.52	38.5
	Sextet 3	–0.03	0.00	266	0.46	11.3
20	Sextet 1	0.01	0.00	336	0.30	8.2
	Sextet 2	–0.01	0.00	304	0.43	30.6
	Sextet 3	–0.03	0.00	266	0.67	58.8
	Singlet	–0.12	–	–	0.64	2.4
35	Sextet 1	0.01	0.00	336	0.30	0.1
	Sextet 2	–0.01	0.00	304	0.37	2.4
	Sextet 3	–0.03	0.00	266	0.66	27.8
	Sextet 4	–0.05	0.00	234	0.64	32.8
	Sextet 5	–0.06	0.00	202	0.57	20.6
	Sextet 6	–0.08	0.00	172	0.44	6.0
	Sextet 7	–0.10	0.00	137	0.78	2.3
	Doublet	–0.09	0.83	–	0.77	5.0
	Singlet	–0.12	–	–	1.13	3.0
50	Sextet 1	0.01	0.00	336	0.31	1.6
	Sextet 2	–0.01	0.00	304	0.30	0.5
	Sextet 3	–0.03	0.00	266	0.30	0.7
	Sextet 4	–0.05	0.00	234	0.58	10.9
	Sextet 5	–0.06	0.00	202	0.55	23.0
	Sextet 6	–0.08	0.00	172	0.51	26.5
	Sextet 7	–0.10	0.00	137	0.47	13.7
	Doublet	–0.09	0.91	–	0.71	7.4
	Singlet	–0.12	–	–	1.13	15.7
90	Sextet 1	0.01	0.00	336	0.30	6.6
	Singlet	–0.12	–	–	0.52	93.4

x is the atomic percent of Cr, δ is the isomer shift relative to α-Fe, Δ is the quadrupole splitting, Γ is the full width at half maximum of the line, H_{eff} is the effective hyperfine field, and $S(k)$ is the percent of the area of the component relative to the total area of the spectrum.

formed in an inert atmosphere. At the same time, in this study MA was carried out in air. Based on previous data [9–12, 21, 23], it might be expected that, in our case, the main component of the Mössbauer spectrum will be a singlet, whereas actually the sextets account for most of its area. To obviate this seeming contradiction, it should be taken into account that, in the studies in question, MA was performed using air-cooled planetary mills, in which the rotation rate of the supporting disk did not exceed 650 rpm. In this study, we used AGO-2U, a water-cooled planetary mill in which the rotation rate of the supporting disk is 1200 rpm. As shown previously using theoretical and experimental methods [25, 38], at the rotation rate of the supporting disk and ball diameter used in this study the temperature in the vial during MA is ~420–520°C. For comparison, according to the same studies the temperature in the vial does not exceed 230°C if the rotation rate of the supporting disk is 685 rpm. On the other hand, as shown by Findiki et al. [9] and Costa et al.

[12], during annealing of MA materials prepared in the presence of oxygen and nitrogen the singlet component of their Mössbauer spectrum gradually disappears and, accordingly, sextet components emerge. The reason for this is that, during annealing, dissolved nitrogen and oxygen precipitate from the solid solution to form nitrides and oxides [9]. Accordingly, the Mössbauer spectrum takes the form characteristic of alloys prepared in an inert atmosphere. According to Findiki et al. [9], these transformations have the highest rate at temperatures from 430 to 560°C. The sample annealed at 560°C has no singlet component. Thus, it is reasonable to assume that, in our case, the temperature in the vials is sufficient for the dissolved gases to convert into oxides and nitrides, which accounts for the observed shape of the Mössbauer spectrum of the sample containing 50 at % chromium.

In the Mössbauer spectrum of the sample containing 90 at % chromium, the major component is a singlet. This correlates well with previous reports [14–17, 20–22, 39] that the Mössbauer spectrum of MA materials containing 70 at % and more chromium has the form of a singlet line, independent of the milling atmosphere. At the same time, the measured spectrum contains, in addition to the singlet line, a weak sextet line with an effective hyperfine field characteristic of pure Fe. It seems unlikely that, after prolonged milling, undissolved Fe persisted in the sample. Most likely, the sextet is due to the contamination of the sample with the ball and vial material, which is an unavoidable problem in the MA process [40–42].

Parameters of the Mössbauer spectra of the alloys under investigation are presented in Table 2. The H_{eff} values obtained for the sextets corresponding to the presence of Cr atoms in the nearest neighbor environment of Fe in the BCC lattice are similar to those presented in Litvinov et al. [43]. It is of interest to compare the experimentally determined percentages of Fe atoms having different nearest neighbor environments in the BCC phase to theoretical values calculated under the assumption that the solid solution is chemically homogeneous. Under this assumption, the fraction of the sextet corresponding to a particular number of Cr atoms in the nearest neighbor environment, k, in the overall area of the sextets, $S(k)$, corresponds to a probability $W(k)$; that is, we assume Cr atoms to have an additive effect on the hyperfine structure parameters of the Mössbauer spectra. In accordance with previously reported results [34, 44], the number of atoms in the nearest neighbor environment j was taken to be eight; that is, we took into account only the nearest neighbors. The probability to find Fe atoms that have k Cr atoms in their nearest neighbor environment is given by the binomial distribution

$$W(k) = \frac{j!}{k!(j-k)!} x_{\text{Fe}}^{j-k} x_{\text{Cr}}^{k}, \tag{1}$$

where x_{Fe} and x_{Cr} are the corresponding mole fractions. Figure 2 shows calculated $W(k)$ for the alloys under investigation and experimentally determined $S(k)$. For the spectra of the alloys containing 20 at % or more Cr, which have a central component, $S(k)$ was calculated with allowance for only the sextet components of the spectrum.

For the alloys containing 5 and 10 at % Cr, there is very good agreement between $W(k)$ calculation results and $S(k)$ experimental data, which can be interpreted as additional evidence for the formation of homogeneous, disordered solid solutions in these samples. The present findings correlate well with results of previous studies [5, 6], where experimental evidence was presented for the formation of a chemically homogeneous solid solution as a result of the MA of a material containing 10 at % chromium.

Unlike in the case of the less chromium-rich alloys, there is a significant discrepancy between the $W(k)$ calculation results and $S(k)$ experimental data for the alloy containing 20 at % chromium. The observed discrepancy suggests that the structure produced by the MA of this material is not a homogeneous disordered solid solution. Indeed, it has been pointed out in a number of reports that, at 20 at % Cr, the structure of the alloy produced by MA is far from being homogeneous. Pandey et al. [7] obtained an experimental $S(k)$ distribution in the form of an essentially horizontal line; that is, the sample contained regions both deficient and enriched in Cr atoms relative to the described $W(k)$ distribution of the Cr atoms in a homogeneous disordered solid solution. Clearly, this means that, in this case, phase separation of the solid solution, characteristic of the system under investigation [1], was observed. As a criterion for atomic-scale intermixing during MA in the Fe–Cr system, Lemoine et al. [22] used the ratio of the experimentally observed decrease in the H_{eff} averaged over the Mössbauer spectrum relative to that for pure Fe to the decrease in H_{eff} for a completely disordered homogeneous solid solution. It was found that, whereas at 30 and 40 wt % Cr this parameter approached unity in the course of prolonged MA, for the sample containing 20 at % Cr it did not exceed 0.8 even after prolonged milling; that is, at this composition, MA does not lead to the formation of a disordered homogeneous solid solution [22].

Comparison of the $S(k)$ distribution obtained in this study with $W(k)$ (Fig. 2) indicates that, in the sample under investigation, the number of iron atoms that have two chromium atoms in their nearest neighbor environment is considerably increased, whereas the number of iron atoms that have zero, three, or four chromium atoms as nearest neighbors is reduced relative to the described $W(k)$ distribution of the Cr atoms in a homogeneous disordered solid solution. The observed $S(k)$ distribution suggests that the chemical inhomogeneities present in the alloy are unrelated to

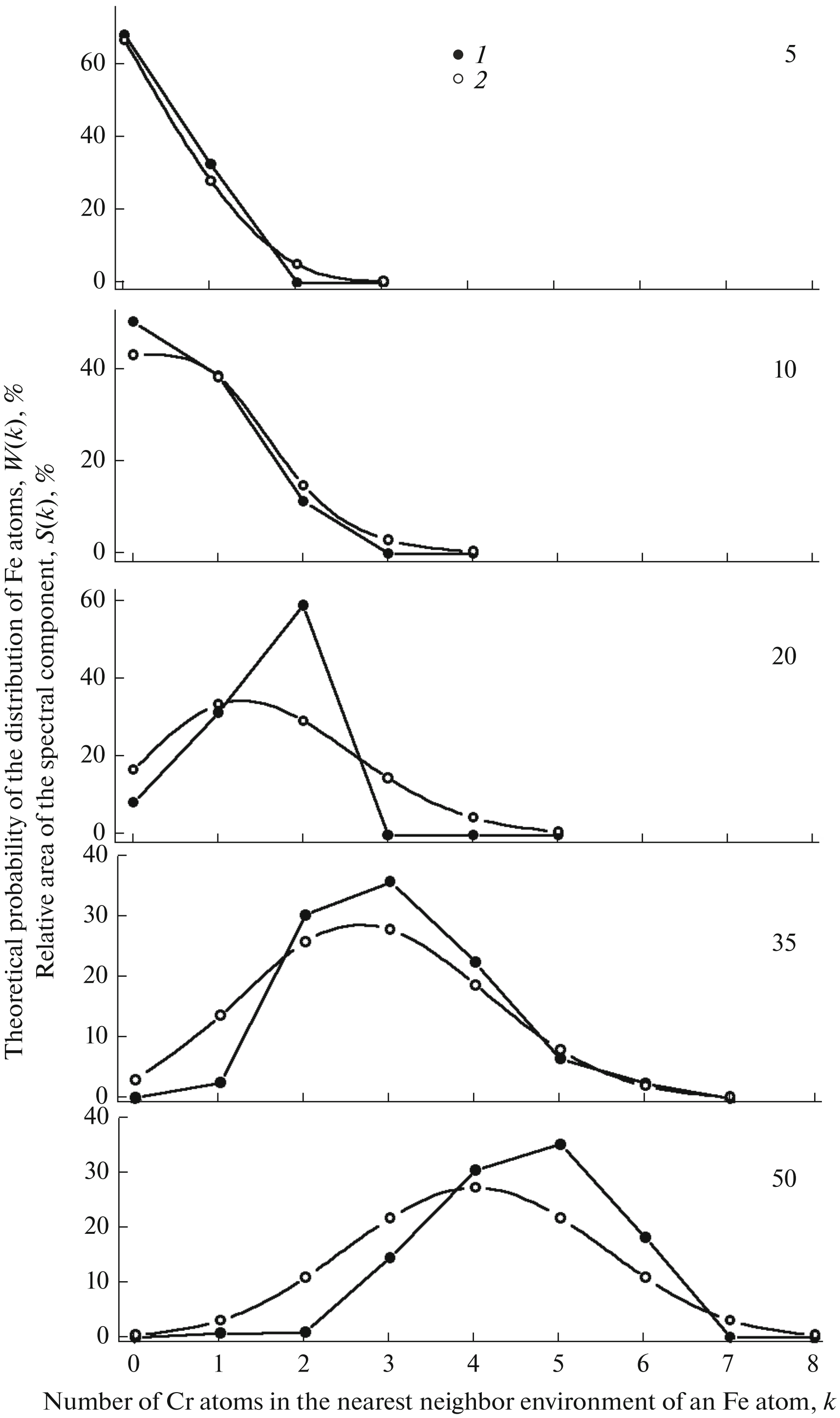

Fig. 2. (*1*) $S(k)$ and (*2*) $W(k)$ distributions for the alloys under investigation. The Cr content of the samples (5–50 at %) is indicated at the curves.

the phase separation of the solid solution. It is known that heat treatment or irradiation of alloys containing 20 at % chromium leads to the formation of chromium nanoclusters [2, 3]. It is reasonable to assume that the $S(k)$ distribution observed in this study is related to the initial stage of clustering. As a result of the MA-related thermal activation, the chromium atoms, first evenly distributed over the crystal lattice, form groups of a few atoms. Such associates may be responsible for the fact that the nearest neighbor environment of the iron atoms contains predominantly two chromium atoms.

The $S(k)$ distribution for the alloys containing 35 and 50 at % chromium is similar in shape to the $W(k)$ distribution describing the distribution of Cr atoms in a homogeneous disordered solid solution. At the same time, the experimentally determined $S(k)$ values are displaced to higher Cr contents relative to calculated $W(k)$, especially in the case of the sample containing 50 at % Cr. It can be seen from Table 2 that, in the alloys under investigation, a rather considerable part of the iron atoms (8.0 and 23.1, respectively) corresponds to the central components of the Mössbauer spectrum. As shown earlier [9–12, 23], after MA of materials with similar compositions in an atmosphere containing O_2 and/or N_2, the Mössbauer spectrum of the alloys consists entirely or almost entirely of a singlet line. As assumed above, in our case the temperature in the range ~420–520°C in the vials of the mill during MA facilitates the precipitation of dissolved nitrogen and oxygen from the solid solution. At the same time, according to Findiki et al. [9] after annealing of MA materials their Mössbauer spectrum has a form characteristic of alloys produced in an inert atmosphere, but after annealing at 560°C. It is reasonable to assume that the temperatures of the MA process in this study are insufficient for complete release of gas impurities from the solid solution. As a result, the X-ray single-phase alloys containing 35 and 50 at % chromium actually consist of a combination of two structures: a rather homogeneous disordered chromium-enriched solid solution and an iron-enriched imperfect structure containing considerable amounts of dissolved oxygen and nitrogen.

It is worth noting that the distinctions between the $S(k)$ and $W(k)$ curves observed in Fig. 2 at 20 at % and more chromium can also be due to the fact that relation (1) takes into account only contribution of the nearest neighbor environment to H_{eff}, whereas in the general case in unfolding the spectrum one should take into account the contribution of more distant neighbors to H_{eff} [15, 37]. To discard or confirm the above interpretations of the distinctions between $S(k)$ and $W(k)$, as consequences of chemical inhomogeneities in the samples under investigation, we analyzed the composition dependence of the average hyperfine magnetic field H_{av}. According to Lemoine et al. [22], it can be used as a criterion for the formation of a homogeneous solid solution in the system under investigation. The composition dependence of experimentally determined H_{av} is presented in Fig. 3. In calculating H_{av}, we took into account the magnetic component of the Mössbauer spectrum, which corresponds to the volume of nanocrystalline grains in the MA Fe–Cr material according to Lemoine et al. [22]. Also presented in Fig. 3 are data corresponding to a homogeneous disordered solid solution. According to Dubiel and Zukrowski [45], over the entire composition range, H_{av} in the Fe–Cr system follows the linear relation

$$H_{\text{av}} = H_{\text{eff}}^{\text{Fe}} - 6554\eta C, \tag{2}$$

where C is concentration (atomic fraction) and η is the change in the polarization of the s-electrons in response to the addition of one Cr atom to a unit cell of the BCC structure. According to Dubiel and Zukrowski [45], η lies in the range 0.025–0.035. In Fig. 3, the range of H_{av} values corresponding to a homogeneous disordered solid solution is bounded by dashed lines.

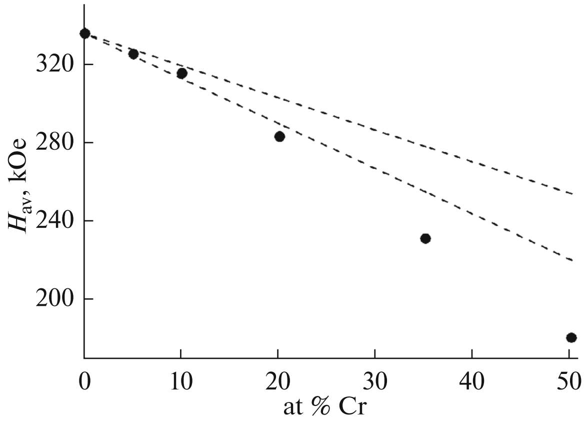

Fig. 3. Composition dependences of H_{av}: experimental data (filled circles) and calculation by formula (2) (dashed lines).

It is seen in Fig. 3 that, the experimentally determined H_{av} in the MA samples containing 5 and 10 at % corresponds to that in a homogeneous disordered solid solution, which supports the above conclusions drawn from analysis of the $S(k)$ and $W(k)$ data. The experimentally determined H_{av} in the alloys containing 20 at % and more Cr is lower than the value following from formula (2), suggesting [22] that our experimental samples suffer chemical inhomogeneity. Thus, analysis of the H_{av} data is consistent with the conclusions drawn from the data in Fig. 2 as to the chemical inhomogeneity of the MA materials containing more than 20 at % Cr.

CONCLUSIONS

The iron–chromium alloys prepared by MA are X-ray single-phase and have a BCC structure over the entire composition range. At the same time, analysis of the present Mössbauer spectroscopy data leads us to assume that some of the alloys are chemically and structurally inhomogeneous.

Structural homogeneity of the alloys has been analyzed by comparing the experimentally determined area of the sextets, $S(k)$, to the area calculated under the assumption that Cr atoms have an additive effect on the hyperfine structure parameters of the Mössbauer spectra of the alloys and that the solid solution is chemically homogeneous with a probability $W(k)$.

The MA powders containing 10 at % or less Cr consist of a homogeneous disordered solid solution. At 20 at % Cr, there are significant structural inhomogeneities. This is tentatively attributed to the initial stage of clustering: as a result of the MA-related thermal activation, the chromium atoms, first evenly distributed over the crystal lattice, form groups of a few atoms. Analysis of the Mössbauer spectroscopy data for the alloys containing 35 and 50 at % chromium suggests the coexistence of an almost homogeneous disordered chromium-enriched solid solution and an iron-enriched highly disordered structure containing a considerable amount of dissolved gases.

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