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Mössbauer Spectroscopy Studies of Mechanosynthesized Fe_2CrSi and Co_2FeAl Heusler Alloys

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Two Heusler alloys, i.e., Fe_2CrSi and Co_2FeAl, were successfully prepared using the mechanical alloying method. X-ray diffraction and Mössbauer spectroscopy were applied to monitor the process of alloy formation. In both cases, the disordered body-centered cubic solid solution was obtained after 20 h of milling, with an average crystallite size of 11 and 23 nm for Fe_2CrSi and Co_2FeAl, respectively. Thermal treatment caused an increase in grain size and the formation of Heusler phases. Mössbauer spectroscopy revealed atomic disorder in the occupation of the crystallographic positions in the $L2_1$ structure. Macroscopic magnetic measurements showed soft magnetic properties of the milled and annealed powders. The saturation magnetization values of 68 and 140 emu/g and magnetic moments of 2.33 and 5.14 μ_B/f.u. determined for, respectively, Fe_2CrSi and Co_2FeAl alloys correspond well with the average values of the hyperfine magnetic fields ~ 20 and ~ 31 T revealed by Mössbauer spectroscopy.

topics: Heusler alloys, mechanical alloying, Mössbauer spectroscopy, magnetization

1. Introduction

In 1903, German engineer Friedrich Heusler discovered ferromagnetic order in Cu_2MnAl alloy which consisted of non-magnetic elements [1]. Since then, over one thousand Heusler alloys have been synthesized and studied because of their excellent properties and extraordinary functionalities. Heusler alloys can exhibit ferro-, ferri-, or antiferromagnetism, superconductivity, giant magnetoresistivity, half-metallic ferromagnetism, and magneto-caloric effect [2–5]. These alloys can be shape-memory materials, topological insulators, semiconductors, piezo-electric and catalytic materials, etc. [6–8]. For the reasons mentioned above, the Heusler alloys find wide applications in spintronics and electronics, energy technologies, superconductivity, and magnetocaloric devices [9–12]. Among the properties, special attention is paid to Heusler alloys exhibiting half-metallic ferro-magnetism discovered by de Groot et al. [13] in 1980. The concept of half-metallic ferromagnet concerns the majority spin band, which shows the typical metallic nature, and the minority spins, which exhibit semiconducting behavior with a gap at the Fermi level. Because half-metallic ferromagnets have an energy gap leading to 100% spin polarization at the Fermi level, a fully spin-polarized current should be feasible in these types of materials.

In the present paper, the attention is focused on two Heusler alloys, namely (i) Fe_2CrSi, which is theoretically predicted to be a half-metallic ferromagnet, and (ii) Co_2FeAl, which exhibits half-metallicity and large (86%) spin polarization, which makes this compound a good material for spintronic THz emitters [14]. However, the main goal of our studies concerns the method of synthesis of these Heusler alloys. We want to show that it is possible to synthesize the Fe_2CrSi and Co_2FeAl alloys using the mechanical alloying (MA) method with subsequent thermal treatment. Among methods of preparation of the Heusler alloys, we can distinguish classical arc-melting, melt-spinning, or planar flow casting (for ribbons), electrodeposition (for nanowires), magnetron sputtering (for thin films)ånd co-precipitation (for nanoparticles) [15–18]. In dependence on the synthesis method and heat treatment, the Heusler alloy can be obtained as an ordered phase, a non-ordered phase, or a mixture of ordered and non-ordered phases.

Recently, it was reported that Fe_2CrSi and Co_2FeAl Heusler alloys might be successfully obtained via the MA synthesis, and the effect of milling time and annealing temperature on the structure and some physical properties of these alloys has been investigated [19–21]. However, Mössbauer spectroscopy has not been used in the published works on mechanosynthesized Heusler alloys. Thus, we propose Mössbauer spectroscopy to monitor the formation of the Heusler phase at every stage of the milling process. It is important to apply Mössbauer spectroscopy together with X-ray diffraction because these two methods complement each other and allow us to recognize the phases and determine their magnetic state and local magnetic structure of the alloys. In our recent paper, it was shown that the Fe_2CrSi Heusler alloy can be prepared by 20 h of MA and subsequent thermal processes, i.e., heating from room temperature up to 700°C and isothermal annealing at 700°C [22]. The aim of the present work was to show the influence of the temperature and time of the thermal process on the phase composition and hyperfine interactions of Fe_2CrSi and Co_2FeAl Heusler alloys prepared by the mechanosynthesis method. Moreover, the macroscopic magnetic properties of the alloys were investigated.

2. Experimental details

In general, the mechanical alloying process consists in grinding the initial powders of components in a special ball mill. During collisions with the balls, the powder particles are flattened and fractured. Between the pieces with a layered structure, diffusion takes place; it is so-called cold welding. Particles are repeatedly flattened, fractured, and welded. After a certain milling time, the components are mixed on an atomic scale. Milled powders of a given composition reach a stationary state, or dynamical equilibrium, through the combined action of plastic deformation and diffusion.

In order to obtain the Heusler Fe_2CrSi and Co_2FeAl alloys, stoichiometric amounts of the Fe, Cr, Si, Co, and Al powders (with particle sizes less than 100 μm and a purity over 99.5%) were weighted and mechanically synthesized in a planetary ball mill FRITSCH PULVERISETTE P5. Stainless steel vial and balls (10 mm in diameter) were used with a ball-to-powder weight ratio of 10:1 and a total mass of 10 g. The MA process was conducted in an argon atmosphere at room temperature with an operating velocity of 200 rpm for Fe_2CrSi and 250 rpm for Co_2FeAl alloys. As we recently reported, in the case of Fe_2CrSi alloy, after 20 h of MA, the Heusler phase was not obtained [22]. Similarly, the milling process of Co, Fe, and Al powders did not lead to the formation of Co_2FeAl Heusler alloy.

The key issue is the heat treatment of the MA product. For both alloys, differential scanning calorimetry (DSC) was applied to study the thermal stability of the powder mixture after 20 h of MA. Based on DSC curves and the literature data, the following annealing temperatures have been chosen: 300, 500, and 700 °C. However, the annealing at 300 and 500 °C was not enough to form the Heusler phase. Isothermal annealing was performed for 1 h in an argon atmosphere in a tube furnace. Moreover, the powders milled for 20 h were subjected to continuous heating from room temperature up to 700 °C with a rate of 20°C/min.

To study the structural features and phase composition, room-temperature X-ray diffraction (XRD) on a Rigaku MiniFlex II diffractometer with Cu K_α radiation was used. Analysis was performed using the PDF4 + database. The lattice parameter was determined based on the Nelson-Riley approach [23].

^{57}Fe Mössbauer spectroscopy (MS) was applied to monitor the Heusler phase formation and to determine the hyperfine interaction parameters of the MA product at every stage of milling and after thermal treatment. The spectra were registered at room temperature using a conventional constant-acceleration-type POLON spectrometer calibrated with α-Fe foil. The ^{57}Co in a Rh matrix was a radioactive source of gamma radiation. The macroscopic magnetic properties of the materials were studied using a vibrating sample magnetometer (VSM) under the magnetic field between -2.0 to 2.0 T at room temperature.

3. Results and discussion

Detailed XRD patterns of the powders mixtures milled for 1, 2, 5, 10, and 20 h showed the disappearance and overlapping of diffraction peaks originating from the appropriate elements. In both Fe_2CrSi and Co_2FeAl cases, a body-centered cubic (BCC) solid solution was formed after 20 h of MA (Figs. 1 and 2).

Both thermal processes led to the formation of the Heusler Fe_2CrSi phase; however, the obtained material consists of two variants of the Heusler alloy, namely the Fm-3m phase (ICDD card no. 04-015-2526; open blue circles in Fig. 1) and Pm-3m phase (ICDD card no. 04-005-1780; blue squares in Fig. 1) [22]. The relative contribution of the phases was estimated as $\sim 86\%$ of the Fm-3m phase and $\sim 14\%$ of the Pm-3m phase. However, it can be noted that during isothermal annealing at 700°C, small amounts of the secondary phases, i.e., FeCr and Cr_3Si, were formed. The lattice parameter of the main Fm-3m phase listed in Table I is in good agreement with the database value $a = 0.5679$ nm (ICDD card no. 04-015-2526). Both thermal processes caused grain expansion, i.e., the average

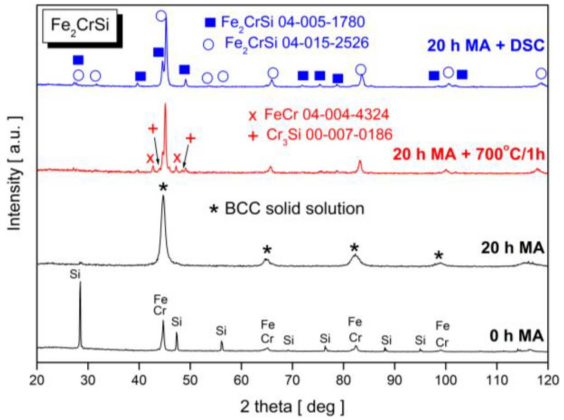

Fig. 1. XRD patterns of the initial powder mixture and Fe_2CrSi alloy after 20 h of MA and subsequent isothermal annealing at 700°C and heating up to 700°C (DSC).

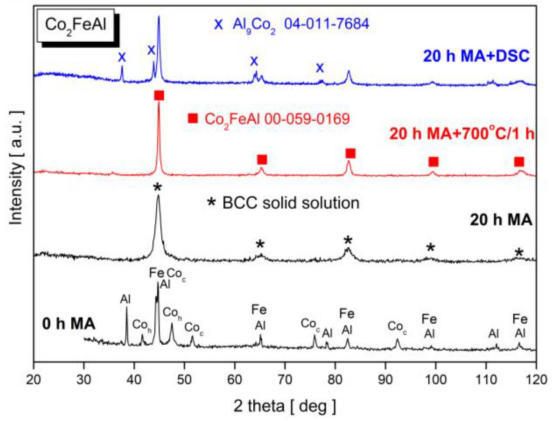

Fig. 2. XRD patterns of the initial powder mixture and Co_2FeAl alloy after 20 h of MA and subsequent isothermal annealing at 700°C and heating up to 700°C (DSC).

crystallite sizes (D) increased from 11 nm after 20 h of MA to about $\sim$ 30 nm after subsequent annealing/heating. It is worth underlining that secondary phases are not visible in the XRD pattern for the sample heated continuously from room temperature up to 700°C (blue pattern in Fig. 1). This suggests the influence of the time of the thermal process on the structure of the mechanosynthesized material.

Similarly, in the case of Co_2FeAl, both heat treatments caused the formation of the Heusler phase Pm-3m (ICDD card no. 00-059-0169; red squares in Fig. 2). However, during heating from room temperature to 700°C, a relatively high amount ($20 \pm 10\%$) of the secondary phase was formed. It was recognized as Al_9Co_2 (according to ICDD card no. 04-011-7684; blue x's in Fig. 2).

Mössbauer spectra measured for the samples milled for various periods (between 1 and 20 h of MA) showed mainly sextets characteristic of iron. After 20 h of milling, a contribution of additional

TABLE I

Structural parameters of the Fe_2CrSi and Co_2FeAl Heusler alloys; a — lattice parameter for the main phase, D — average crystallite sizes; BCC solid solution.

Sample	a [nm]	D [nm]	Main phase
	Fe_2CrSi		
20 h MA	0.2873(5)	11(2)	BCC sol. sol.
20 h MA+700°C/1 h	0.5668(5)	30(5)	Fm-3m
20 h MA+DSC	0.5669(5)	27(4)	Fm-3m
	Co_2FeAl		
20 h MA	0.2868(1)	23(2)	BCC sol. sol.
20 h MA+700°C/1 h	0.2859(1)	25(2)	Pm-3m
20 h MA+DSC	0.2860(1)	22(2)	Pm-3m

components is visible in each spectrum (Figs. 3 and 4). In the case of Fe_2CrSi, the spectrum for the 20 h MA sample was numerically fitted by one sextet due to α-Fe (with a contribution of $\sim$ 52%), one doublet ascribed to the FeSi phase ($\sim$ 19%), and six sextets with hyperfine magnetic fields between 9 and 27 T coming from BCC solid solution of Cr and Si atoms in the iron lattice [22]. For the Co_2FeAl, in the spectrum of the sample milled for 20 h, apart from the iron component (60%), another sextet (40%) with a hyperfine magnetic field $B_{hf} = 29$ T was fitted. This sextet may indicate the beginning of a new phase, namely the Co_2FeAl Heusler phase.

Thermal processes caused the creation of Heusler phases with or without secondary phases. Mössbauer spectrum of the Fe_2CrSi sample isothermally annealed at 700°C was elaborated using one singlet ($\sim$ 14%), one doublet ($\sim$ 10%), and the quasi-distribution of eight sextets (red spectrum in Fig. 3). The single line component with an isomer shift $IS = -0.24(1)$ mm/s can be attributed to the FeCr compound (a similar value was reported in [24] for FeCr nanoparticles). The doublet with $IS = 0.11(1)$ mm/s and quadrupole splitting $QS = 0.32(2)$ may originate from the equiatomic FeSi alloy (similar values were reported in [25] for $Fe_{50}Si_{50}$ nanopowders). The hyperfine magnetic field distribution observed in the Mössbauer spectrum indicates atomic disorder in the Heusler Fe_2CrSi alloy. As proved by XRD, two variants of the Heusler phase were obtained after annealing. Besides an ordered $L2_1$-type phase (Fm-3m), the Pm-3m phase also exists. In the $L2_1$ phase, the iron atoms are in 8c positions and have 4 Cr atoms and 4 Si atoms in the nearest neighborhood (Fig. 5). However, besides 8c positions, the Fe atoms may occur at 4a or 4b sites, especially since the atomic radii of Fe, Cr, and Si are very similar to each other. Moreover, in the Pm-3m phase, the iron atoms can occupy 6c or 2a positions.

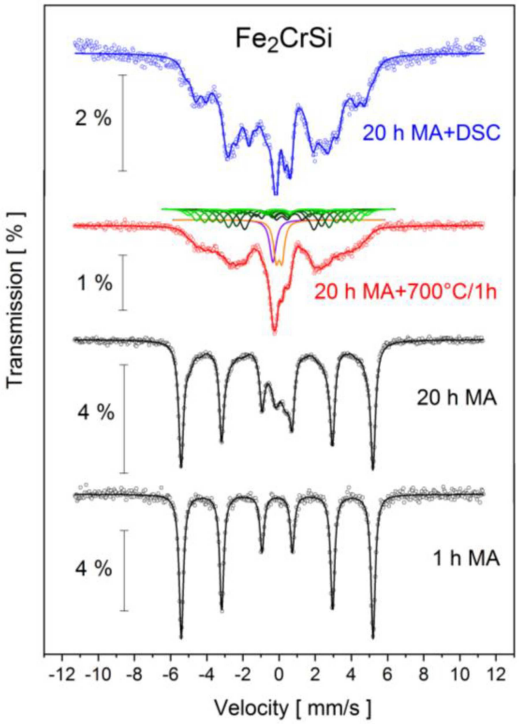

Fig. 3. Room-temperature Mössbauer spectra of the Fe, Cr, and Si powder mixture milled for 1 h, after 20 h of MA and after subsequent isothermal annealing at 700°C and heating up to 700°C (DSC).

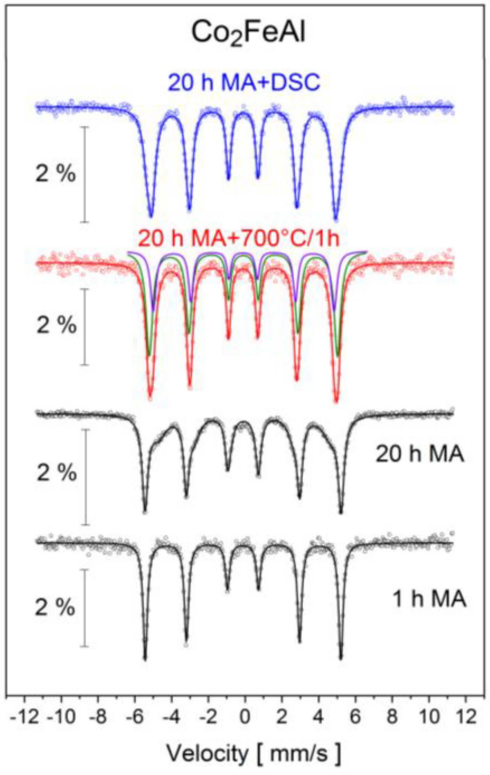

Fig. 4. Room-temperature Mössbauer spectra of the Co, Fe, and Al powder mixture milled for 20 h, after 20 h MA and after subsequent isothermal annealing at 700°C and heating up to 700°C (DSC).

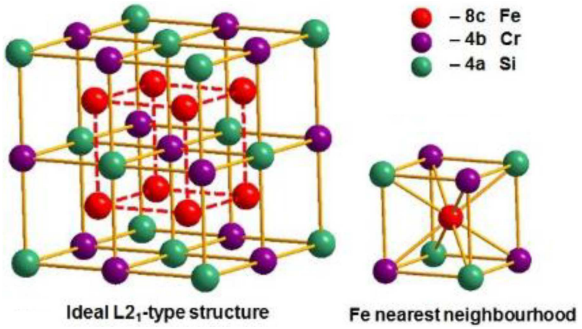

Fig. 5. The unit cell of the ideal $L2_1$-type structure of the Fe_2CrSi Heusler phase and the nearest neighborhood of the iron atom.

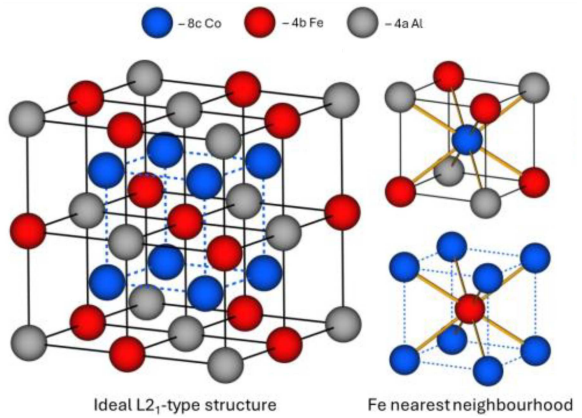

Fig. 6. The unit cell of the ideal $L2_1$-type structure of the Co_2FeAl Heusler phase and the nearest neighborhood of the iron atom.

As suggested by XRD results, there were no secondary phases in the 20 h MA Fe_2CrSi sample after continuous heating from room temperature up to 700°C (DSC). However, the Mössbauer spectrum revealed similar components, as in the case of the annealed sample, i.e., singlet, doublet, and the distribution of sextets. The only difference is that after this type of thermal treatment, the share of singlet and doublet components decreased, namely the contribution of singlet is 3.2% and of the doublet 7.4%.

In the case of Co_2FeAl alloy, Mössbauer spectra of both powders after 20 h of MA and subsequent annealing and heating consist of two sextets (Fig. 4). These two components come from two nonequivalent positions of ^{57}Fe atoms in the crystalline lattice of Co_2FeAl alloy. As revealed by XRD, the Pm-$3m$ Heusler phase was formed after heat treatment. In this phase, two Wyckoff positions exist, i.e., $1a$ occupied by Co and $1b$ occupied by Fe and Al atoms. The position $1a$ in the Pm-$3m$ phase corresponds to the $8c$ site in the $L2_1$ ordered phase, while the $1b$ position corresponds to the $4a$ (or $4b$) site in the $L2_1$-type structure (Fig. 6).

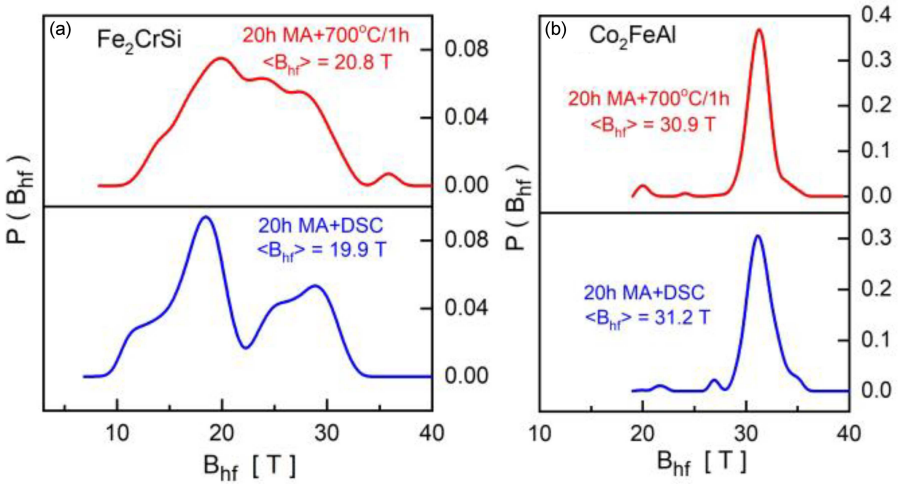

Fig. 7. Hyperfine magnetic field distributions for the Fe_2CrSi (a) and Co_2FeAl (b) alloys isothermally annealed at 700°C for 1 h and heated up to 700°C (DSC).

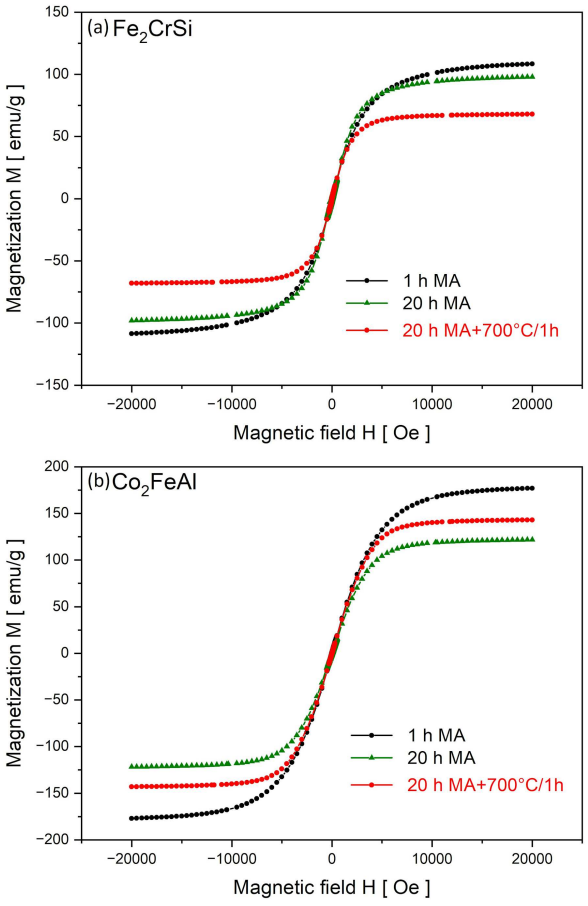

Fig. 8. Magnetization curves for the powder mixture (a, b) milled for 1 and 20 h, after 20 h of MA and subsequent isothermal annealing at 700°C.

However, the occupation of $1a$ site ($8c$ in $L2_1$) by iron atoms is also not excluded. Two sextets with hyperfine magnetic fields $B_{hf} = 31.84(2)$ T and $B_{hf} = 30.59(2)$ T and the relative contribution of 67 and 33%, respectively, were assigned to $1b$ and $1a$ positions for the alloy synthesized for 20 h and isothermally annealed at 700°C. Mössbauer spectrum for the sample heated continuously from room temperature up to 700°C (DSC) revealed two sextets with $B_{hf} = 32.72(2)$ T and $B_{hf} = 31.07(2)$ T and the relative contribution of 28 and 72%, respectively. Thus, this kind of thermal treatment caused preference in the occupation of $1a$ sites by iron atoms. Regardless of the type of heat treatment, the values of the hyperfine magnetic field in Co_2FeAl Heusler alloy are relatively high, which is caused by the Co atoms in the nearest neighborhood of iron.

The occurrence of atomic disorder in the studied alloys prompted us to use another method of fitting, namely hyperfine magnetic field distribution using the Hesse–Rübartsch procedure [26]. In the procedure, the linear correlations between B_{hf} and IS as well as between B_{hf} and QS were applied. The results of numerical elaboration of the Mössbauer spectra for the thermally treated samples are presented in Fig. 7. In the case of Fe_2CrSi alloys, the hyperfine magnetic fields occur in the range from 9 to 35 T. The distribution observed for the annealed Fe_2CrSi sample has one broad mode, from which the average value of the hyperfine magnetic field was calculated to be $\langle B_{hf} \rangle = 20.8$ T. However, in the distribution for the heated Fe_2CrSi alloy, two modes can be observed around B_{hf}~18 and ~28 T. The second Co_2FeAl Heusler alloy is characterized by a narrow hyperfine magnetic field distribution. Depending on the type of heat treatment, the highest values of the hyperfine fields occur with different probabilities. The average value of the hyperfine magnetic field $\langle B_{hf} \rangle = 30.9$ T for the alloy isothermally annealed, and $\langle B_{hf} \rangle = 31.2$ T for the heated Co_2FeAl sample agree well with the values obtained from the discrete model of the fitting. The observed distributions, especially in the

TABLE II

Macroscopic magnetic properties of the Fe_2CrSi and Co_2FeAl Heusler alloys; M_S — saturation magnetization, H_C — coercive field, μ — magnetic moment per formula unit, μ_{th} — theoretical value of the magnetic moment.

Sample	M_S $\left[\frac{\text{emu}}{\text{g}}\right]$	H_C [Oe]	μ $\left[\frac{\mu_B}{\text{f.u.}}\right]$	μ_{th} $\left[\frac{\mu_B}{\text{f.u.}}\right]$
	Fe_2CrSi			
1 h MA	108(1)	22(1)	3.72	
20 h MA	98(1)	59(1)	3.36	2.00
20 h MA+700°C/1 h	68(1)	10(1)	2.33	
	Co_2FeAl			
1h MA	177(1)	23(1)	6.36	
20 h MA	122(1)	36(1)	4.38	5.00
20 h MA+700°C/1 h	143(1)	10(1)	5.14	

case of Fe_2CrSi alloys, are relatively broad, which can be the result of both chemical and structural disorders.

Macroscopic magnetic properties of the powder mixtures milled between 1 and 20 h and after isothermal annealing were checked. As seen in Fig. 8, the magnetization curves $M(H)$ for both alloys after 1 and 20 h of MA and after annealing at 700°C are characteristic of soft ferromagnets.

From the magnetic hysteresis loops, the saturation magnetization M_S, coercive field H_C, and the magnetic moment μ per formal molecule were determined and are presented in Table II. It was observed that the magnetization of both powder mixtures decreases as the milling time increases. This effect is caused both by the dilution of non-magnetic elements in the iron lattice and the increase in structural disorder in the surface layer of grains with nanometer sizes. However, after isothermal annealing, the saturation magnetization drops significantly for the Fe_2CrSi alloy, while for the Co_2FeAl alloy, M_S increases. For both alloys, the experimental values of magnetic moments per formula unit are larger than those predicted theoretically based on the Slater–Pauling rule. This result is justified because the theoretical magnetic moment is calculated for an ideal Heusler phase with an ordered $L2_1$ structure. The observed enhancement in the experimental μ values could be related to the atomic disorder revealed by Mössbauer spectroscopy.

Our results should be compared with those reported for the Fe_2CrSi and Co_2FeAl Heusler alloys prepared by the mechanical alloying method. Considering the structure, some differences can be demonstrated, mainly after thermal treatment. Differences in grain sizes, lattice parameters, phase composition, and secondary phase content result mainly from different synthesis conditions (various materials of balls and vial, different ball-to-powder weight ratio, milling time, etc.). To the best of the authors' knowledge, Mössbauer spectroscopy was not used to study mechanically alloyed Fe_2CrSi and Co_2FeAl Heusler alloys, thus our results fill this gap. The results obtained in our experiments can be compared with data published for the Heusler alloys prepared by other methods. A series of non-perfectly ordered Heusler Fe_2MAl compounds with M = V, Cr, Fe, Co, Ni obtained by the Czochralski method from a levitated melt was studied using Mössbauer spectroscopy and characterized by the hyperfine magnetic field distributions [27]. In an ideal ordered $L2_1$-type structure, the Fe atom at site 8c has 4 Al atoms at sites 4a and 4 M atoms at sites 4b as nearest neighbors, and it possesses a low magnetic moment and $B_{hf} \sim 22$ T. In our experiment, disordered Heusler Fe_2CrSi alloy was obtained with low $\mu \sim 2.3$ μ_B and $\langle B_{hf} \rangle \sim 20$ T. In the case of Co_2FeAl alloy, the hyperfine magnetic field values obtained by us are in good agreement with $B_{hf} = 31.0$–31.5 T reported for this alloy prepared via the induction melting method [28]. Comparing the macroscopic magnetic properties, it can be noticed that the magnetic saturation of Fe_2CrSi and Co_2FeAl Heusler alloys prepared by MA occurs at a much lower magnetic field (at about 500 Oe in our study) than in the case of the analogous melted alloys ($\sim$ 1 kOe [27]) or nanoparticles (5–10 kOe [29–31]). This allows us to suppose that mechanosynthesized Heusler alloys possess a magnetic anisotropy smaller than materials prepared by other methods.

4. Conclusions

Nanocrystalline Fe_2CrSi and Co_2FeAl Heusler alloys were successfully prepared by mechanosynthesis of elemental powders using high-energy ball milling and subsequent thermal treatment. It was shown that 20 h of milling resulted in the formation of BCC solid solution. Thanks to Mössbauer spectroscopy, we have proven that mechanical milling itself creates the beginning of a new phase, and the proper Heusler alloy was formed after the thermal process. The kind of thermal treatment and its duration influence the type of structural disorder of a given Heusler phase and the formation of secondary phases. The materials obtained in our experiment exhibited a disordered crystal structure in which iron atoms may occupy various sites, giving rise to the hyperfine magnetic field distribution. Magnetization measurements confirmed the ferromagnetic behavior and soft magnetic properties of the samples. In summary, it can be stated that mechanical synthesis can be an alternative method for obtaining Heusler alloys with iron content, and the Mössbauer spectroscopy is a key technique not only for monitoring the technological process but also for getting information about phase composition and magnetism of the material.

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