# Order-disorder phenomena from X-ray diffraction in FeCo alloys annealed and ground at high energy

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Order-disorder transformations were studied in annealed and high-energy ball-milled near-equiatomic FeCo alloys. X-ray diffraction with Co  $K\alpha$  radiation enables to follow disordering with milling time because of anomalous dispersion. © 2011 International Centre for Diffraction Data. [DOI: 10.1154/1.3624320]

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## I. INTRODUCTION

Order-disorder transformations in intermetallic compounds had a major role in the development of modern physical metallurgy. Potential applications of ordered alloys are based on combinations of magnetic, transport, and structural properties which are strongly dependent on chemical order. This includes, for instance, magnetic memory and hard disk read-heads or applications in actuators and sensors. For both applied and fundamental aspects, it is thus necessary to characterize and master the state of order of alloys, in particular for the more demanding class of nanostructured materials for which there are prospects of improved properties with respect to those of their classical counterparts and expectations of extended fields of applications.

Equiatomic FeCo is a good example of the above mentioned intermetallic alloys. FeCo undergoes an order-disorder transformation from the ordered, primitive cubic CsCl structure, or B2 phase, stabilized by magnetism (Neumeyer and Fähnle, 2001), to a disordered A2 bcc structure, at hightemperature annealing (Sourmail, 2005). The  $A2 \rightarrow B2$ phase transformation causes various changes. For instance, changes of magnetic moments of the lattice parameter (Sourmail, 2005) and of the phonon density of states (Lucas et al., 2010) are noted. Another notable ordered alloy is Fe<sub>65</sub>Co<sub>35</sub>, which has the largest room-temperature magnetization but it is a soft magnet (Sellmyer, 2002). A way to enhance the properties of permanent magnets is through "exchange coupling" between a hard material and a soft material with a large magnetization. Models show that the soft phase must have grains of about 10-nm diameter and that the hard phase too must have nanosized grains (Sellmyer, 2002). An ideal exchange-coupled magnet, consisting of aligned grains of Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub> and of Fe<sub>65</sub>Co<sub>35</sub>, could reach an energy product of about 125 MGOe (Skomski and Coey, 1993). However, this objective is still very challenging.

Plastic deformation is a means of changing the state of order of alloys which was and is still used to prepare and investigate structures far from equilibrium. A technique to plastically deform particles and to disorder alloys is ballmilling (Gaffet et al., 1999; Suryanarayana, 2001), which is the main method besides isothermal annealing and quench, which are considered in the present paper. Furthermore, alloys prepared in that way are nanostructured, a characteristic which may be essential as discussed above. Mechanical alloying (MA) of mixtures of powders of pure elements or of powders of already partially combined elements must, however, be distinguished from the grinding (MG) of materials whose chemical composition remains the same during milling but whose structure is expected to evolve. Earlier works on grinding of ordered alloys deal, for instance, with the influence of grinding on the long-range order parameter as measured by X-ray diffraction on the magnetic properties and on the hyperfine magnetic properties measured by NMR and by <sup>57</sup>Fe or <sup>119</sup>Sn Mössbauer spectroscopy. Magnetic properties depend very sensitively on chemical disorder and give thus an indirect and precious information on it. Studied alloys were, for instance, B2 FeAl (Pochet et al., 1995), B2 Fe<sub>60</sub>Al<sub>40</sub> (Gialanella et al., 1998), B2 NiAl, B2 AlRu, L1<sub>2</sub> Ni<sub>3</sub>Al (Cahn, 1999), and L2<sub>1</sub> Heusler alloys  $X_2$ MnY ( $X = Ni_1$ , Cu, Pd, Y = Al, In, Sn, Sb) (Shinohara *et al.*, 1981; Schaf *et* al., 1983; Le Caër et al., 1997). More recent studies of ballmilling of ordered alloys are aimed at characterizing ground alloys with nanometer-sized grains and their reordering kinetics.

Due to a combination of good mechanical and magnetic properties, Fe-Co alloys have triggered a wealth of publications which are most often devoted to studies of bulk alloys, sometimes monocrystalline, of thin films and of powders. Materials with nanosized characteristic lengths and nanograined alloys were thoroughly investigated in recent years. Most studies of nanostructured FeCo are devoted to the mechanical alloying of elemental powder mixtures of Fe and Co (Brüning *et al.*, 1992; Collins and Meeves, 1993; Elkalkouli *et al.*, 1995; Moumeni *et al.*, 2005; Sarkar and Bansal, 2001; Sorescu and Grabias, 2002; Li and Ramanujan, 2004; Sourmail, 2005; Chakka *et al.*, 2006; Zelenakova *et al.*,

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2007; Nie et al., 2007; Garroni et al., 2007). Given milling conditions and a powder mixture of two elements M and T, whose mean composition is  $M_{1-x}T_x$ , at least two characteristic times can be defined. The longest, t<sub>f</sub>, is the minimum time needed to enter a stationary state in which all kinds of powder characteristics remain essentially unchanged when milling for times longer than t<sub>f</sub> (hardness, magnetic properties, average grain sizes, crystallographic structures, etc.). The shortest characteristic milling time t<sub>cm</sub> was named "chemical mixing time" by Le Caër et al. (2000). It is the minimum milling time needed for all powder particles to have essentially the same average chemical composition, namely  $M_{1-x}T_x$ , where, obviously,  $t_f \ge t_{cm}$ . These powder particles may indeed be made up of various phases whose structures and compositions still need to evolve up to a milling time  $t_f > t_{cm}$ . For binary Fe–T (T = V, Cr, Mn) alloys, the chemical mixing times, some hours, were measured to be a significant fraction of t<sub>f</sub> (from 30% to 100%, Le Caër et al., 2000). As an ordered FeCo alloy cannot be synthesized by mechanical alloying at room-temperature from starting mixtures of Fe and Co, no information can be obtained from such experiments about the kinetics of disordering by ballmilling. The mixing of elements and the microstructural evolution can be followed in that case, most often through their effects on various physical properties.

By contrast, "mechanical grinding," in which one starts from an already ordered *B2* FeCo alloy, makes it possible to follow the disordering kinetics by milling and, as above, the microstructural evolution. The disorder induced by milling in *B2* FeCo with 4-at.% Mo was, for instance, followed by Mössbauer spectroscopy (Sarkar and Bansal, 2001). The disordering of *B2* FeCo by ball-milling was generally less considered than the mechanical alloying of nanocrystalline bcc FeCo.

In this study, we examine chemical disorder produced in *B*2 FeCo by ball-milling using mainly X-ray diffraction with Co  $K\alpha$  source. Fultz (1991) used Co  $K\alpha$  X-ray diffraction to study the kinetics of short-range and of long-range *B*2 ordering of *A*2 FeCo samples rapidly quenched by the piston-anvil technique. Combining Monte-Carlo simulations and X-ray diffraction measurements, he was able to follow the evolution of the long-range order parameter during thermal annealing.

Long-range order (LRO) is usually studied from the intensities of superlattice diffraction lines. For a B2 structure with atom 1 at the corner of the cell and atom 2 in its center, the structure factor is recalled to be

$$F_{hkl} = f_1 + f_2(-1)^{h+k+l}, \tag{1}$$

where  $f_k$  is the atomic scattering factor of the *k* atom (Guinier, 1963). The intensities of superlattice lines (h + k + l = 2n + 1) are then proportional to  $|f_1 - f_2|^2$ , while the intensities of fundamental diffraction lines (h + k + l = 2n) are proportional to  $|f_1 + f_2|^2$ . The proportionality constants depend, however, on the multiplicity of the considered diffracting planes and on corrections terms, such as an absorption term and Lorentz and Debye-Waller factors, which depend on the diffraction angle. Providing evidence for the existence of long-range order in FeCo alloys by X-ray diffraction is all the more difficult that the difference between the atomic form factors of Fe and Co is very small. For instance, the LRO parameter cannot be determined from XRD with Cu  $K\alpha$  radiation, as the atomic scattering factors of Fe and Co are practically equal. By contrast, it can be determined from neutron diffraction because the neutron coherent scattering lengths of neighbor atoms may be very different,  $9.45 \times 10^{-15}$  and  $2.49 \times 10^{-15}$  m for Fe and Co, respectively. Neutron diffraction has, however, a lack of immediate availability and needs a larger sample mass for analysis than X-ray diffraction.

There is a way to bypass the intrinsic difficulties of performing LRO analyses in FeCo by XRD measurements through anomalous dispersion that occurs when the selected wavelength is close to an absorption edge of one of the constituent elements of the alloy. In this case, the superlattice line intensities are not as high as they are in the neutron diffraction patterns, but they are still sufficiently high to evidence the existence of long-range order LRO.

Defining a simplified indicator of the relation between superlattice and fundamental peak intensities as

$$R = |f_{\rm Fe} - f_{\rm Co}|^2 / |f_{\rm Fe} + f_{\rm Co}|^2 \qquad (2)$$

for a perfectly ordered *B*2 FeCo, we obtain 0.013 < R(Co  $K\alpha$ ) < 0.04 and 0.00046 < R(Cu  $K\alpha$ ) < 0.005 for  $30^{\circ} < 2\theta < 130^{\circ}$  (Prince, 2006). This ratio *R* indicates that it is possible to observe superlattice peaks with Co  $K\alpha$ radiation, as Co is one of the elements of the studied alloy. Furthermore, the use of Co  $K\alpha$  radiation has an additional advantage as it cannot excite absorption by Fe in contrast to Cu  $K\alpha$ . Effects due to absorption and fluorescence background, which would be detrimental to experimental observation of superlattice lines, are thus avoided.

## **II. EXPERIMENTAL**

Appropriate amounts of elemental iron (99.9% in purity) and cobalt (99.8% in purity) were melted together in an induction furnace in argon atmosphere, in order to obtain a near equiatomic FeCo alloy. The melting process was repeated three times in order to ensure homogenization. For further homogenization, the ingots were annealed at 900 °C for 24 h. Then, they were cryo-fractured into a microcrystal-line powder and reduced to particles with a size inferior to 110  $\mu$ m.

Annealing in a vacuum at 550 °C over 52 h and slow cooling in vacuum was done to promote ordering. To promote disorder, ball-milling was performed in argon atmosphere in a planetary Fritsch P6 mill at 500 rpm for different milling times, up to 150 min. In the latter case, after being ball-milled, the samples were sieved below 90  $\mu$ m. The vials and the seven balls used to mill were made of hardened steel. The powder to ball weight ratio was 1:20.

Chemical analyses of ingots and ball-milled samples were done using SEM/EDX (SEM: Jeol, EDS: Oxford) in a standard analysis with 20-keV electron beam. The composition of the as-cast alloy was  $Fe_{51.2}Co_{48.8}$  at.%. This composition remains constant until 60 min of milling. The final

composition of the alloy milled during 150 min was  $Fe_{51.6}Co_{48.4}$  at.% because of a slight contamination by the milling materials.

X-ray diffraction patterns were recorded using a Seifert diffractometer with Co  $K\alpha$  radiation ( $\lambda = 0.17903$  nm). The mean crystallite size and microstrain were obtained from XRD patterns with the Williamson–Hall method (Williamson and Hall, 1953). Software package FULLPROF (Rietveld refinement) (Rodriguez-Carvajal, 1998) was used to determine the unit-cell parameters of the bcc and *B*2 phases.

Mössbauer spectroscopy (MS) was also used to characterize the state of order of alloys through hyperfine magnetic field distributions (HMFDs). <sup>57</sup>Fe spectra were recorded at RT in a transmission geometry using a standard constant acceleration spectrometer. A <sup>57</sup>Co source in Rh matrix with a strength of  $\approx 10$  mCi was used. The experimental spectra were analysed by a constrained Hesse-Rübartsch method (Le Caër and Dubois, 1979), which yields a HMFD, P(B). Lorentzian line-shapes were employed in this procedure. As usual, the isomer shifts are given with respect to  $\alpha$ -Fe at RT.

#### **III. RESULTS AND DISCUSSION**

Figure 1 shows that the ratio between superlattice and fundamental peak intensities [see Eq. (2)] is much higher for Co  $K\alpha$  than it is for Cu  $K\alpha$ ,  $R(Co K\alpha) \gg R(Cu K\alpha)$ . The angular dependence of  $R(Co K\alpha)$  is about 25 times larger than that of  $R(Cu K\alpha)$  below 40°, then it decreases gradually becoming about 10 times more around 130°. The observation of superlattice lines at high angles  $2\theta$ , for instance the (210) superlattice line, is thus favored. Anomalous dispersion suffices then to evidence the presence of superlattice peaks with Co  $K\alpha$  radiation.

Figure 2 shows the XRD patterns taken for an ordered sample and for samples ball-milled for 20 and 150 min. Apparently, the patterns consist only of the fundamental lines of a bcc lattice, but superlattice reflections are clearly evidenced when zooming on the angular ranges where they are expected to appear (Figure 3). The (100) and (210) superlattice lines are both clearly observed. These reflections are observed not only for the ordered sample but also for the bulk as-cast sample and for samples milled up to 20



Figure 1. (Color online) Ratio *R* as a function of  $2\theta$  Bragg angle for Co  $K\alpha$  and Cu  $K\alpha$  radiations.

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Figure 2. XRD patterns of a FeCo alloy ordered and milled for 20 and 150 min.

min. The superlattice lines are no longer seen after ball-milling for periods longer than 20 min. This method can thus be used to characterize the disordering kinetics with milling time through the associated decrease of normalized intensities of a few superlattice lines. Figure 2 shows that the diffraction peak linewidths increase, as expected, with milling time. This increase results from the decrease of crystallite sizes and from the increase of microstrains with milling times (Table I). The values shown in Table I were obtained from the analysis of fundamental reflections by the Williamson–Hall method. Values ( $\langle d \rangle$  and  $\xi$ ) obtained using superlattice reflections using the same method are 35(1) nm; 0.12(1)%, 45(1) nm; 0.74(1)%, 20(1) nm; 0.86(1)% for ordered FeCo and for FeCo milled for 5 and 10 min, respectively. The decrease of the LRO parameter with milling time (Figure 4) is confirmed by the decrease of the unit-cell



Figure 3. Enlargement of XRD patterns in the angular range of appearance of superlattice reflections, S(100), S(111), and S(210), for as-cast and ordered samples, as well as for samples ordered and then ball-milled for the indicated periods.

TABLE I. Mean crystallite sizes,  $\langle d \rangle$ , and microstrains,  $\xi$ , obtained by the Williamson-Hall method from fundamental reflexions of XRD patterns of the indicated samples.

Sample	$\langle d \rangle$ (nm)	$\xi$ (%)
Ordered	47(1)	0.2(1)
Milled 5 min	24(1)	0.3(1)
Milled 10 min	21(1)	0.4(1)
Milled 20 min	20(1)	0.4(1)
Milled 30 min	19(1)	0.5(1)
Milled 45 min	19(1)	0.4(1)
Milled 60 min	17(1)	0.5(1)
Milled 90 min	17(1)	0.5(1)
Milled 150 min	15(1)	0.5(1)

parameter of the bcc phase obtained from Rietveld refinement of the XRD patterns. The overall decrease in the unitcell parameter of 0.0002 nm is in full agreement with the change reported to occur between the ordered and disordered FeCo, namely 0.0002 nm from 0.28570 to 0.28550 nm (Sourmail, 2005). The ordering reaction in equiatomic FeCo is so rapid that the cooling rates required to fully disorder it cannot be achieved in industrial processes at least about 4000 °Cs<sup>-1</sup> (Sourmail, 2005). Disordered FeCo can, however, be produced by ultrafast quenching, for instance by the piston-anvil technique (Fultz, 1991). This alloy can be efficiently disordered too by mechanical means, here high-energy ball-milling.

The settings of the XRD acquisitions were chosen to maximize the intensities of B2 superlattice lines. In that way, it is possible to establish unambiguously the existence of long-range order. However, these changes saturated the detector and introduced distortions of the top of the most intense peaks. Because of this, it was not possible to quantify the LRO parameter.

Figure 5 shows the Mössbauer spectra and hyperfine field distributions obtained for the samples annealed and then ball-milled for different periods. Although the form of spectra does not change much, the hyperfine magnetic field distribution has a steady small change with milling time. In Figure 5,  $\langle B \rangle$  and  $\langle IS \rangle$  values are seen to increase with disordering, and the values obtained for ordered and disordered samples are similar to the ones found in literature (Eymery



Figure 4. Unit-cell parameters of the bcc phase for the FeCo sample milled during the indicated periods.

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and Moine, 1978; Hamdeh *et al.*, 1989). The ordered sample has  $\langle B \rangle = 34.3(1)$  T and  $\langle IS \rangle = 0.013(2)$  mm/s, and the disordered sample milled for 150 min has  $\langle B \rangle = 35.2(1)$ T and  $\langle IS \rangle = 0.036(2)$  mm/s. The values found in the literature are  $\langle B \rangle = 34.1$  and 35.1 T,  $\langle IS \rangle = 0.016$  and 0.032 mm/s, for the ordered and disordered samples, respectively (Eymery and Moine, 1978).

The overall changes of the lattice parameter a, the hyperfine magnetic field B and the isomer shift IS of B2 FeCo are



Figure 5. (Color online)  ${}^{57}$ Fe RT Mössbauer spectra of the FeCo samples, first ordered and then milled, and hyperfine magnetic field distributions as a function of milling time: (a) 0, (b) 5, (c) 20, (d) 45, (e) 60, (f) 90, and (g) 150 min.



Figure 6. Decrease with milling time  $t_m$  of the normalized parameters  $(X_M - X(t_m))/(X_M - X(0))$ , where X = a (empty circles), B (empty squares with crosses), IS (full circles), and  $X_M$  is the value of the corresponding parameter for long milling times.

rather limited when the long-range parameter S decreases from 1 to 0. The previous parameters are normalized so as to vary between 0 and 1,  $(X_M - X(t_m))/(X_M - X(0))$  (X = a, B, IS), where  $X_M$  is the value of the corresponding parameter for long milling times. The normalized parameters decrease with milling time as shown in Figure 6. Although they are not necessarily proportional to S when S ranges between 0 and 1, these normalized parameters nevertheless give a fair idea of the speed at which S decreases. Decreases with milling time of the LRO parameter S, measured by X-ray diffraction, are reported for L1<sub>2</sub> Ni<sub>3</sub>Al [Figure 3 of Cahn (1999) and Figure 2 of Jang and Koch (1990)] and B2  $Fe_{60}Al_{40}$  [Figure 2(a) of Gialanella et al. (1998)]. The characteristic disordering times are seen to depend on the dynamical conditions of milling. Altogether, the changes of the lattice parameter, of the superlattice line intensities and of the Mössbauer hyperfine parameters provide a consistent picture of a typical time of  $\sim 0.5$  h for disordering B2 FeCo by ball-milling in our experimental conditions.

### **IV. CONCLUSION**

As recalled in the introduction, high-energy ball-milling is a synthesis method of disordered nanocrystalline FeCo which is now well-established. The ordering kinetics of nanocrystalline disordered FeCo of FeCo-X alloys prepared by mechanical alloying was studied by Sarkar and Bansal (2001). As the small difference in X-ray scattering factors of Fe and Co makes it rather difficult to study the state of order of ball-milled or of mechanically alloyed FeCo by X-ray diffraction, the latter is most often characterized by magnetic and by hyperfine magnetic properties. X-ray diffraction using Co  $K\alpha$  was, however, successfully applied to follow the reordering of rapidly quenched A2 FeCo (Fultz, 1991). The disordering of B2 FeCo by ball-milling was considered briefly, as it is an efficient disordering method. In our dynamical conditions, B2 FeCo is indeed disordered by ballmilling at room-temperature for only 20 to 30 min as shown by the disappearance of superlattice diffraction lines on XRD patterns and by the changes of the lattice parameter and of the hyperfine parameters (Figures 4 and 6).

Qualitative investigations of the LRO restoration on heating at moderate temperatures are planned. X-ray diffraction studies will be complemented by <sup>57</sup>Fe Mössbauer studies.

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