The magnetic and structural properties of nanostructured $(Fe_{75}Al_{25})_{100-x}B_x$ alloy prepared by mechanical alloying

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Abstract

New magnetic nanocrystalline powders $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9, 12) were elaborated by mechanical alloying. The boron content and milling time affect the magnetic and structural properties of these alloys. Morphological, microstructural, structural, thermal and magnetic characterizations of the powders milled for different time were investigated by scanning electron microscopy, X-ray diffraction, vibrating sample magnetometer and SQUID techniques. Increasing the boron content, the formation of two crystalline phases was observed, the major one corresponding to FeAl bcc phase and another in lower proportion corresponding to a Fe-B phase which needed more milling time. Increasing the milling time, the crystallite size decreases to the nanoscale, the micro-strain increases as do the lattice parameter of the disordered solid solution. A decrease in coercivity (Hc) with increasing the boron content was also observed. These variations are explained on the basis of crystallite size and strain variation in the samples during milling.

Keywords: FeAl alloy; High energy ball milling; X-ray diffraction; nanostructure; Magnetic properties; thermal stability.

1. Introduction

Nowadays, nanomaterials are the subject of many studies due to their potential application in the transfer and storage of magnetic information, catalysis, biotechnology and medical diagnostics, such as magnetic resonance imaging and energy [1]. In addition, the increasing complexity of these applications has resulted in the design of new nanomaterials and thus, the design/invention of new generation of nanoparticles. For this reason, an intensive research is still in development, particularly on the relationship between microstructure and magnetic response of these nanostructured alloys [2,3,4].

Iron-based alloy powders are one of the main components of the soft magnetic composites. These composites are being developed to provide materials with competitive magnetic properties (good relative permeability and magnetic saturation) but with high electrical resistivity. In particular, Fe–Al binary compounds have been widely studied due to their interesting magnetic properties from both fundamental and applied point of view [5]. The low fabrication cost, their low corrosion at high temperature as well as the low density and good wear resistance are attractive factors for numerous technological applications.

Mechanical alloying (MA) is an applied powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases. The advantage of this process technology is that the powder can be produced in large quantities and the processing parameters can be easily controlled. Hence, it is a suitable method for commercial applications [6,7,8]. This latter, being a widely used solid state processing route, can easily fabricate nanocrystalline materials with good homogeneity from elemental powders, thus MA can be an ideal way to prepare Fe-Al alloys doped with different contents of Boron. In addition, a recent study showed that the addition of a small amounts of boron changes the fracture mode during milling and also produces a certain growth of the grain boundaries resulting from the formation of Fe_2B [9]. However, knowledge of the effect of boron addition on microstructural and magnetic properties of Fe-Al alloys remains limited [9,10].

More recently, a renewed interest/curiosity about this ternary system has grown especially in relation to the modification of the magnetic properties generated by compositional and microstructural changes [9,10,11]. The purpose of the present study is to investigate the combination of the microstructure evolution and the magnetic behavior in $Fe_{75}Al_{25}$ alloy doped with boron during mechanical alloying.

2.   Experimental details

Mixtures of powders of Fe, Al and B with purity of 99.99% of the nominal composition $Fe_{75}Al_{25}$ powder doped with small amounts of boron (x=0, 5, 9 and 12 at%) were mechanically alloyed using a laboratory planetary ball mill (Type PM 400) under Ar atmosphere. Ball milling experiments were carried out in a hardened steel container. The ball-to-powder weight ratio used was maintained at 12:1 and the milling speed was adjusted to 400 rpm [11]. The milling sequence was selected such as 10 minutes of milling are followed by 5 minutes of idle period, to prevent sticking of the powder to container walls and the balls, and powder agglomeration during milling. X-ray diffraction (XRD) measurements were done using a D-500 Siemens equipment with CuKα radiation. The size of the coherently diffracted crystallites, the lattice strains and lattice parameter were calculated based on the Rietveld method using the X'Pert High Score Plus program. In all refined XRD patterns, refinement parameter R_{exp} is lower than 13.5% and GOF parameter lower than 1.8.   The morphology and the composition of mechanically alloyed powders were examined by scanning electron microscopy in a DSM960A ZEISS microscope in secondary electron mode operating at a voltage of 15 kV. The SEM was equipped with a Vega_ Tescan energy dispersive X-ray spectrometry (EDS) analyzer.   The magnetic characterization was carried out by Superconducting Quantum Interference Device from Quantum Design SQUID MPMS-XL at 300 K (about 150 mg of powder in each experiment).

3.   Results and discussion

3.1 Morphology

Fracturing and cold welding lead to significant changes in the morphology of the powder particles during the milling process. Fig.1 shows the microstructure of different samples. It can be seen that increasing milling time from 10 hours to 40 hours, particle size decreases. After 10 hours of milling, a shape distribution of the particles is shown in Fig.1(a.1, c.1, e.1 and g.1), for all compositions and powders, they form larger particles with irregular shape and size due to the event of cold welding. The compressive forces into the particles generated by ball-powder-ball collisions flatten the spheres. Depending on the dominant forces, the particle size may either becomes smaller through fracturing or suffering agglomeration by welding as the milling process progresses. At higher magnification as can be seen in Fig.1(a.2, c.2, e.2 and g.2), the powder particles appear to be largely distorted due to the high rate of deformation introduced by mechanical milling. This is the effect of plastic deformation induced by collisions between the balls and the particles against the walls jar. The powder is characterized by flattened facies. Subsequent to milling for 40 hours, the powder particles get work-hardened, consequently the hardness and brittleness increases, and the agglomeration of particles start to break down Fig.1(b.1, d.1, f.1 and h.1). Also, prolonging the milling time resulted in a narrower distribution of particle sizes as a balance is reached between the fractures and the welding processes. Finally, the obtained morphology of the particles is finer and more homogeneous. One can clearly deduce that boron helps in refining the morphology resulting in smaller particle size and narrower size distribution of particles Fig.1(b.2, d.2, f.2 and h.2). This effect could be due to the preferential diffusion of boron atoms to the interstitial sites of the bcc phase through grain and particle boundaries enhancing the precipitation of Fe-B compounds there. Intergranular segregation of boron has a beneficial effect on the fracture mode of FeAl alloys. It changes from intra-granular to inter-granular fracture mode. These observations suggest that the presence of the fracture surfaces can be associated with the presence of borides in grain boundaries. On the other hand, the morphology of the powder doped with 9 at% of boron milled

for 40 h showed a certain agglomeration and consequently the powder size was comprised between 100 μm and 400 μm.

The samples milled 40 h were observed by TEM and analyzed by EDS (in Fig. 2 the EDS spectra corresponding to samples doped with x= 0 and 9 are depicted) and we found that the composition is somewhat different from that of the nominal one in all the cases. This difference in composition may be due to the adhesion of this of powder particles to the jars and the balls during a rather severe and very long milling (with a speed of 400 rpm ad at a milling time of 40 hours). No contamination from jars and/or vials is observed and no oxygen impurities are detected.

3.2 Structural properties

Fig.3. shows the XRD patterns obtained of the milled samples with different B content as function of milling time. All the intensity peaks observed in each of the diffraction patterns can be indexed as the Bragg reflections corresponding to bcc- Fe peaks and fcc- Al peaks accompanied with the appearance of new picks can be attributed to two types of iron boride FeB and Fe_2B phases for the doped samples [12,13]. The formation of bcc Fe(Al) solid solution was accomplished after 20 h milling in the undoped sample (Fig.3.a), this can be followed by the progressive disappearance of the Al (111) peak as well as the broadening and a tiny shift to the right of the Fe (110), (200) and (211) peaks. A similar phase evolution was found for $Fe_{70}Al_{30}$ by Kezrane et al. [14]. In their report, the bcc Fe(Al) solid solution was completely formed after 27 h of milling time. With increasing MA time up to 80 h, they found that the angular position of the peak (1 1 0) slightly shifted to a higher 2θ value and they attributed this shift to the ordering in the alloyed powders. In addition, Rajath Hegde et al. [15] reported that the Fe(Al) solid solution was transformed into an ordered FeAl phase after 28 h of milling in the case of $Fe_{50}Al_{50}$. On the other hand, we can see in Fig.3.b that the peak of Al disappeared after 26 h milling. So the solid solution, bcc Fe(Al) was completely formed at this time in $(Fe_{75}Al_{25})_{95}B_5$ sample. Contrariwise, based on the Fig.3.c and Fig.3.d, the bcc solid solution

was accomplished after 30h of milling in $(Fe_{75}Al_{25})_{91}B_9$ and after 60 h for $(Fe_{75}Al_{25})_{82}B_{12}$. One can deduce that the addition of B delays the formation of the bcc-solid solution Fe(Al). In addition, it can be concluded that for higher content of boron (x>5 at%), the Fe(Al) solid solution has been accomplished after 40 h of milling and for this reason milling time was prolonged to 80h. The peaks of pure Fe (Fig.3.a) have high intensities and their width is narrow, but with the introduction of Al and B and subsequent milling up to 40 h and 80h, the intensities of diffraction patterns have diminished and the peaks have become broadened (Fig.3). This is due to the contribution of the effective crystallite size and an increase of the atomic level strain because of heavy plastic deformation [16]. The formation of the FeB and Fe_2B types boride with lattice parameters (a = 0.4053 nm, b = 0.5495 nm, c = 0.2946 nm) and (a = 0.5137 nm and c = 0.4245 nm) respectively [12,13], during milling, can be related to the crystallite size refinement and the structural defects. In fact, since nanocrystalline materials contain a very large fraction of atoms at the grain boundaries, the many interfaces provide a high density of short diffusion paths and they are expected to exhibit an enhanced diffusivity. Consequently, the diffusion through the grain boundaries controls some precipitation reactions in these solids.

Figure 4 exhibits the evolution of the phase's volume fractions as a function of milling time. One can observe a monotonic increase of Fe(Al) solid solution proportions at the expense of those of α-Fe and Al which disappear completely after 20, 26, 30 and 60 hours for $(Fe_{75}Al_{25})_{95}B_5$, $(Fe_{75}Al_{25})_{91}B_9$ and $(Fe_{75}Al_{25})_{82}B_{12}$ samples. However, the Fe_2B phase proportion varies from 3 and 7% on further milling time. Such variation can be attributed to the complexity of the alloying through the movement of atoms.

3.2.1 Lattice parameter, crystallite size and micro strain calculation

X-ray peak position analysis allows the determination of the average lattice parameter for all powder samples. Figs. 3a-3d showed that after 2 h milling for $Fe_{75}Al_{25}$ and $(Fe_{75}Al_{25})_{95}B_5$ and after 4h milling for $(Fe_{75}Al_{25})_{91}B_9$ and $(Fe_{75}Al_{25})_{82}B_{12}$ samples, the peak (110) of α-Fe shifted towards the higher diffraction angles. The degree of the shift seems to depend on the

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composition of the milled sample. This shift can be related to the lattice compression and/or the diffusion of B into the Fe lattice and cosequently, to the formation of heavy deformed α-Fe and/or bcc substitutional Fe(B) metastable solid solution. In addition, for extended milling time this shift becomes towards the lower diffraction angles for the doped samples. This variation in peak positions is probably caused by the lattice expansion due to the increase in the density of dislocations with their characteristic strain fields on the nanograin boundary.

The lattice parameter of the solid solution bcc $(Fe_{75}Al_{25})_{100-x}B_x$ in these alloys is higher than that of α-Fe (2.8644(1) Å). The increase in the lattice parameter may be associated with the effect of solid solution of substitution and insertion. Since Al atomic radius (1.43Å) is larger than Fe atomic radius (1.26Å) its dissolution into Fe matrix increased the lattice parameter of bcc $(Fe_{75}Al_{25})_{100-x}B_x$ solid solution as was observed in Fig. 5. Furthermore, the increase of the density of the defects during milling can give rise to an increase of the lattice parameter. Unlike the doped alloys, for the undoped alloy, the lattice parameter decreases slightly depending on the milling time. This can be explained by the effect of severe plastic deformation, which can cause compression of the crystal lattice. In fact, during the MA process, severe plastic deformation leads to an increase of the dislocation density. The successive accumulation and interaction of dislocation cause a reduction in the crystallite size and the lattice parameter decreased with milling time. Such variation in the lattice parameter of FeAl powders was observed by Hamlati et al. [2], Fan et al. [17] and Baris et al. [18] and was attributed to the ordered structure as reported by Chan [19]. So, it can be concluded that elemental Fe and Al are alloyed to form slightly ordered bcc-Fe(Al) after 20 h milling, consistent with ordered phase. No obvious change is observed from the XRD patterns of powders milled for longer time and the lattice reached a steady value of 0.28649(1) Å.

Fig.6 shows the effect of milling time on crystallite size variation in $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9 and 12) alloys and it is clear that the crystallite size decreased with an increase in the milling time for all alloys. However crystallite size of $Fe_{75}Al_{25}$ alloy drastically decreased

during the first ten hours approximately to 10±2 nm. Increasing the milling time to 40 h, the size remains stable to 10±2 nm. Similar values were reported by Mhadhbi et al. [16] for the composition Fe-40at%. For the doped alloy with 5 % at of boron, this value is attained after 20 hours of milling. But the crystallite size of doped alloys with 9 % at and 12 % at of boron decreases slowly during milling. The lower crystallite size of 10±2 nm is only reached after milling time of 80 h. At the same time, the micro-strain increases with milling time. Fig.7 shows the changes of internal micro-strain of all powders. As expected, internal strain increases with increasing of milling time more rapidly at the beginning and with slower trend at longer milling times. For $Fe_{75}Al_{25}$ and $(Fe_{75}Al_{25})_{95}B_5$ powders, the micro-strain increases linearly to reach, respectively, values around 0.32 and 0.25%, during the first ten hours. Increasing milling time to 40 h, micro-strain increases slowly until stabilization, respectively, at 0.52 and 0.41%. On the other hand, the micro-strain of $(Fe_{75}Al_{25})_{91}B_9$ and $(Fe_{75}Al_{25})_{86}B_{12}$ alloys, increases progressively during the first 40 hours, respectively, up to 0.2 and 0.15%. Prolonging the mechanical milling to 80 hours, the microstrain of these last two powders increases to 0.38%. The value of the micro-strain for Fe_3Al alloy is relatively high compared to that of other powders. This can be associated with the segregation of boron atoms in grain boundaries [20], which avoids the crystallite growth of the bcc phase. In addition, the instability in the segregation process can be explained by the increasing of the crystallites size and the decreasing of internal stresses during milling. The increasing of residual strains inside the material could be due to stress field associated with the multiplication of the dislocations [21]. Micro-stress in crystallites is generated by vacancies, and other defects such as thermal lacuna, shear planes and contractions. Clearly, the reduction in crystallite size is accompanied by an increase in the lattice strain level as the MA time increases. This is a common behavior for all metallic systems prepared by MA [22]. The state of stabilization during the last hours of milling can be associated with the segregation of boron in the grain boundaries too [20] which prevent the growth of crystallites of the bcc phase. In fact, in the case of non-equilibrium segregation during their

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migrating to the grain boundaries, the boron atoms can interact with vacancies to form complexes vacancy-boron. The elimination of vacancies can be explained by the migration of complex to wells and this latter will be enriched by boron [20].When the equilibrium is attained, the process of segregation is no longer observed. Several studies have shown that this type of segregation affects the structural and magnetic properties of Fe-Al system doped with boron during milling [23,24].

3.2.2 Dislocation Density

During milling, the samples subjected to severe plastic deformation, dislocations are the main defects, and the dislocation density, ρ, can be calculated using the values of the lattice parameter a, crystallite size (D), and microstrains (ε) determined from the XRD pattern refinements, from the following relation [25,26]:

$$\rho_D = 2\sqrt{3}\frac{\langle\varepsilon^2\rangle^{1/2}}{D.b} \tag{1}$$

where b is the burgers vector of dislocations and equals (a√3)/2 for the bcc structure.

When the content of boron increases, micro-strain decreases and the fraction of Fe-B phase increases during milling. Diffusion of the new phase of Fe-B to the grain boundaries delays the refinement of crystallite size and results in a partial annihilation of dislocations during milling. This may explain the evolution of the dislocation density depending on the content boron shown in Fig.8. In fact, the dislocation density gradually decreases while increasing the boron content. One can conclude that for higher content of boron (x≥9 at %), the dislocation density stabilized around $1.6(2)\times10^{15}$ /m^2 after 40h of milling. Prolonging milling time to 80h, this dislocation density (of x≥9 at %) increases to $6.0(3)\times10^{15}$ /m^2. Flowing dislocations from the grain boundary give rise to relaxation of defects. These results confirm the assumption of an increase of the cohesive energy of the grain boundaries during the boron diffusion.

3.3 Magnetic measurements

The coercivity is frequently considered as an important parameter if low losses are to be reached and affected by most types of defects. This includes grain boundaries, dislocations, vacancy, precipitates and non-magnetic particle distribution. This magnetic property is an important factor to identify the soft magnetic behavior. The high increase of the coercivity is due to internal microstrain, pores, impurities and defects which are introduced during milling. To reduce the coercivity, the values of all these factors should decrease. Fig.9 shows typical hysteresis loops of $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9 and 12) powders as function of milling time at 300 K. These sigmoidal shapes are habitually observed in nanostructured materials with small magnetic domains. The ferromagnetic metallic alloys are isotropic magnetostrictive materials. The magnetostriction saturation depends on the chemical composition of these alloys. For the undoped alloy, the zoom of the rings at the central portion (Fig.9.a) shows that the coercitif field for different milling time is less than 800 Oe. This reflects the soft-hard ferromagnetic behavior of the $Fe_{75}Al_{25}$ alloy. On the other hand, it is noteworthy that all doped powders had similar hysteresis loops and that they are narrow. The zoom of the rings for these last ones, at the central portion (Figs.9b, 9c, 9d) shows that the coercive fields for different milling time is less than 100 Oe. This entails that these samples have a soft ferromagnetic behavior. We can see the magnetization saturation of all powders has varied with the milling time. To understand the effect of boron substitution on the magnetic properties of $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9 and 12) powders, magnetic hysteresis (M-H) loops were superposed and depicted in Fig.10. The important difference observed is found in the undoped sample which has the highest saturation. In addition, the coercive field decreases with boron addition. This can be explained by the precipitation of FeB and Fe_2B phases in grain boundaries.

Fig.11 shows the dependence of the coercivity (Hc) for $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9 and 12 at %) powders as a function of milling time at 300K. The coercivity of crystalline alloys depends strongly on grain size because of the interaction of the magnetic domain walls with the grain boundaries. Increasing the milling time will increase Hc but this is not the case. At first,

the coercivity in the samples increases which can be related to the introduction of residual stresses, impurities, micropores and defects produced during milling. The large density of dislocations produced by the severe plastic deformation during milling reacts with domain walls and increases the coercivity by blocking the movement of the domain wall [27]. The following formula translates the relationship between Hc and the related structural parameters:

$$H_c \infty \frac{\lambda_s \varepsilon \delta}{M_s} \tag{2}$$

where λ_s is the magnetostriction constant, ε is the internal strain and δ represents the width of the wall domain. For the doped powders, in addition of the internal stress, the Fe_2B phase is the second reason behind the increasing of the Hc. The magnetic domain walls are blocked by precipitation of the inclusions Fe_2B that are paramagnetic phase in the grain boundaries. This can be expressed by the following equation [28]:

$$H_c = 2.4 \frac{\delta_W K_1}{\mu_0 M_S} \beta^{2/3} \tag{3}$$

where β the number of inclusions or pores per unit volume, d is the diameter of the inclusion or pores and k_1 is the magnetocrystalline anisotropy constant.

Between 20 and 40 hours, a slight decrease is observed for the undoped powder. This can be explained by the decrease in the magnetocrystalline anisotropy due to the stabilization of the crystallite size and the micro-strains. Moreover, a significant decrease was observed in the case of doped powders. This is in agreement with the decrease of crystallite size. Beyond 40h of milling, the variation of Hc of doped powders of 9% at and 12% at of boron can be explained by the surface anisotropy. The dependence of the coercivity (Hc) for $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9 and 12 at %) samples as a function of crystallite size shown in Fig.12 highlights the concept of the magnetic exchange length Lex. For the undoped and doped powders of 5% of boron, the L_{ex} is of the order of 10 nm but for the doped powders of 9 at % and 12 at % of boron is of the order of 32 nm. When the grain size is larger than the magnetic exchange length, Lex, the

coercivity depends on the crystallite size. The saturation magnetization is given by the following relation [29]:

$$H_c = 3\sqrt{\frac{k\,T_C K_1}{a\,M_S}}\frac{1}{D}$$ (4)

where D is the crystallite size, K_1 is the magnetocrystalline anisotropy, M_S is the saturation magnetization, T_C is the Curie temperature, k is the Boltzman constant and a lattice constant. Lex can be calculated as [30]:

$$L_{ex} = \sqrt{\frac{A}{K_1}}$$ (5)

where A is the exchange stiffness constant, the grain boundaries result in the pinning of the domain walls when the grain sizes are above Lex. The increasing of the volume fraction of grain boundaries through grain refinement impedes the domain walls movement which leads to the increase of the coercivity. However, when the grain size is smaller than the magnetic exchange length, domain wall effect diminishes and each grain behaves as a single domain. Based on the random anisotropy model, the coercivity can be expressed as [31,32]:

$$H_C = \frac{P_C K_1{}^4 D^6}{\mu_0 M_S A^3}$$ (6)

where A is the exchange stiffness constant, Pc is the constant of the order of unity and μ_0 the permeability of free space. According to equation (6), as grain size decreases less than Lex, the Hc will decrease and this is in accordance with Fig.11. The value of the magnetic exchange length increases when the added content of boron increases. This can be explained by the increases of the volume fraction of Fe_2B phases in the grain boundaries.

The value of the saturation magnetization, Ms, has been evaluated from the high magnetic field region of the M(H) curves using a fit to an approach-to-saturation law [33]:

$$M = M_S\left(1 - \frac{a}{H} - \frac{b}{H^2}\right) + \chi H$$ (7)

where a is the coefficient related to defects and/or micro-stresses, b is the coefficient related to crystal anisotropy and χ is ascribed to an independent filed susceptibility. In our case, the a/H term is very small and can be discarded [31]. Saturation magnetization is the property which refers to the atomic origin of magnetism. It strongly depends on the chemical composition of the local environment of atoms and their electronic structure. It involves quantum phenomena, such as exchange, crystal-field interaction, inter-atomic hopping and spin-orbit coupling [34].

Variation of Ms with milling time has been illustrated in Fig.13. For the undoped powder, the increase of Ms from 153 emu/g to 177 emu/g during the first ten hours of milling, can be attributed to the formation of the Fe(Al) solid solution. Prolonging milling time to 20 hours, Ms decreases strongly to 66 emu/g. The decrease of Ms can be linked of a significant change in the magnetic moment due to the existence of Al atoms in the neighboring of the Fe atoms which reduces the direct interaction between the ferromagnetic Fe-Fe sites. Finally, the status of 10h is regenerated after 40 hours of milling. This rise of Ms can be related to the decrease of magnetocrystalline anisotropy which is due to the refinement of grains. Differently, the Ms of doped powders decreases during the rest 20 hours of milling. It can be due to the substitution of Fe atoms by Al and B atoms. Between 20h and 40h, the increase in Ms can be explained by the formation of Fe_2B phase in the grain boundaries. The tetragonal ferromagnetic phase Fe_2B exhibits high uniaxial anisotropy (K_1=-4.27×10^5 Jm^{-3}) at room temperature [35]. The saturation magnetization of Fe_2B phase is about 1.2×10^{6}A/m [36]; which is lower than the saturation magnetization of iron. This finding can explain the overall decrease in magnetic performance of nanocomposites Fe (Al, B). A local perturbation called demagnetizing field in magnetism is created by the presence of two different phases and residual stress in mechanics generates decay of the Ms. [21,37].This is due to piezomagnetic character of Fe_2B and their magnetostrictive behavior cited in references [38,39]. In addition, the presence of inclusions Fe_2B phase with the dislocations blocks the magnetic domain walls movements. After 40 hours of milling, Ms of doped powders with 9% at and 12 % at of boron decrease to 142 emu/g. This

decrease can be related to the decrease in magnetocrystalline anisotropy and the increase of the energy of the surface anisotropy [40].

4.   Conclusions

Nanostructured $(Fe_{75}Al_{25})_{100-x}B_x$ (x=0, 5, 9, 12) alloy with good magnetic properties were prepared by mechanical alloying. The most interesting results of this research are as follows:

1.   The completion of bcc Fe(Al) solid solution formation is related to the boron content of the sample. The results of X-ray diffraction indicated that the formation of bcc Fe(Al) solid solution was accomplished after 12 h milling in the undoped sample, after 6 h milling in $(Fe_{75}Al_{25})_{95}B_5$ contrariwise between 30h and 60 h of milling in $(Fe_{75}Al_{25})_{91}B_9$ and in $(Fe_{75}Al_{25})_{82}B_{12}$.

2.   During milling, the crystallite size decreased and reached a steady-state at the nanoscale, in addition the amount of strain associated to the processing increased by increasing the milling time.

3.   An increase of the lattice parameter was observed during milling when adding Boron. This can be explained by the precipitation of tetragonal phase Fe_2B. Also dislocation density decreased when the amount of boron added increased.

4.   When the boron is added the hard ferromagnetic behavior becomes soft magnetic. The inhomogeneous evolution of saturation magnetization can be explained by the refinement of the crystallite size below or above of the magnetic exchange length and the averaging out of the magnetocrystalline anisotropy.

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