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Friction mode and shock mode effect on magnetic properties of mechanically alloyed Fe-based nanocrystalline materials

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The so called "vario mill" (P4 Fritsch) planetary ball mill has been used to prepare nanocrystalline Fe-10 wt% Ni and Fe-20 wt% Ni alloys from powder mixtures. For both studied alloys, a disordered body cubic centered (BCC) solid solution has been formed after 36 h of milling. The higher the shock power, the larger the lattice parameter of the investigated systems. It has been found that in friction mode process (FMP), the lower the crystallite size, the lower the lattice strain of the prepared alloy. In shock mode process (SMP), the lower the crystallite size, the higher the lattice strain. FMP has been found to induce a soft magnetic behavior for Fe-10% Ni and F-20% Ni alloys. The highest values of coercivity have been found in materials prepared by SMP.

1. Introduction

Mechanical alloying is an intensive energy process of mechanical grinding for the preparation of alloyed powders or composites in powder form. It is known that high-energy ball milling in a planetary mill leads to mechanical alloying of the constituent powders through a process involving repeated deformation, fragmentation and rewelding [1]. In the past, several attempts have been made to simulate the dynamics of this milling process in terms of ball velocity, frequency of impact and kinetic energy transferred to the powder charge during milling [2–5]. Maurice and Courtney [2] and Courtney [3] have simulated the mechanism of milling on the basis of Hertzian criterion of perturbed impact to predict the volume of material affected per impact, impact duration, strain-rate, temperature rise and cooling rate. Burgio *et al.* [4] have derived a set of kinetic equations to compute the velocity and acceleration of a ball in a planetary mill, and thereby, estimate the energy transferred to the powder particles. The ball distribution inside the vial is considered to be independent of the kinematics of the ball motion. However, the analysis does not provide a governing principle to predict an optimum milling condition. Abdellaoui and Gaffet [5] have suggested through more rigorous analyses that the powder or ball impact rather than the kinetic energy or frequency may determine the end products and efficiency of the milling process. They have introduced the concept of the shock frequency, the kinetic shock energy and the shock power.

The purpose of this paper is to report the effect of the friction mode and the shock mode of a planetary ball mill (P4 vario mill, Fritsch, Germany) on structure and magnetic properties for Fe-10% Ni and Fe-20% Ni obtained by mechanical alloying.

2. Experimental procedure

Elemental Fe powder, with an average particle size of 7 μm and Ni powder of purity 99.5% with maximal particle size of 250 μm were used. The milling was carried out using a planetary high-energy ball mill P4 vario mill that is the commercialized version of the G5 prototype. The planetary ball-milling equipment (G5) was designed by Gaffet [6], which allows the independent choice of the values of Ω and ω ($\Omega \leq 1000$ rpm, $|\omega| \leq 1200$ rpm), where Ω is the rotation speed of the disc on which the vial holders are fixed. The vial holders turn at a rotation speed of ω. The duration of the milling process is 36 h. Table I shows the shock power, and friction energy (expressed in percentage of total injected power). These values are based on the kinematic studies of the G5 ball mill by Abdellaoui and Gaffet [5]. According to (Ω/ω) the rotation speed choice, we can have the shock mode process (SMP) when $\Omega \gg \omega$, and the friction mode processes (FMP) when $\Omega \ll \omega$. Experimental details for characterization procedure of materials have been described elsewhere [7].

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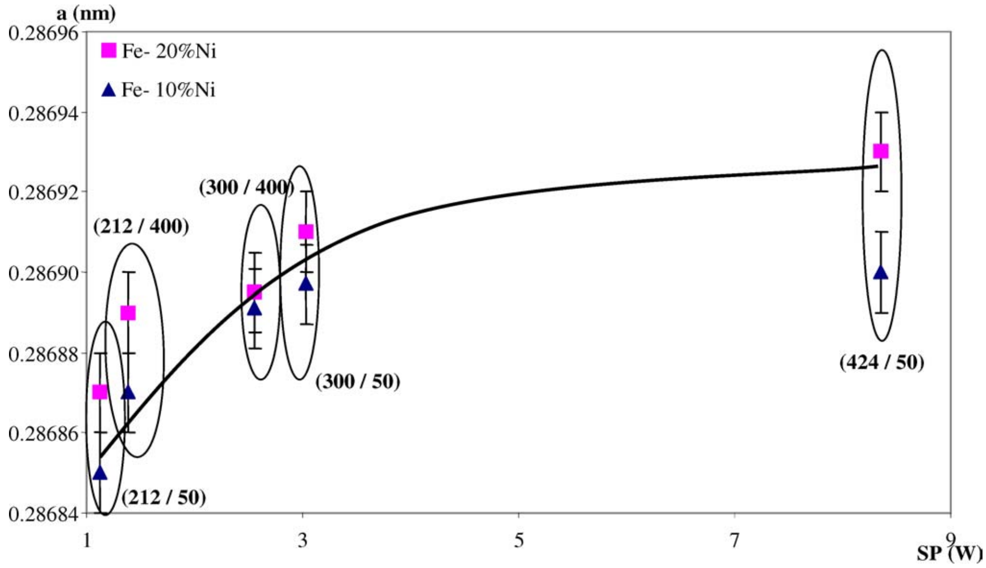

Figure 1 BCC lattice parameter for nanocrystalline Fe-10% Ni and Fe-20% Ni after 36 h of milling versus shock power (SP expressed in W).

3. Results and discussion

Fig. 1 presents the change of lattice parameter a, versus shock power for nanocrystalline Fe-10% Ni and Fe-20% Ni after 36 h of milling. Such minor change of the lattice parameter was due to the small atomic size difference of the Ni and Fe atoms [8]. It is observed that the lattice parameter increases when the shock power increases. Using G5 planetary ball mill, Gras *et al.* [9] have found that the lattice parameter of Fe-Si alloys increases when the shock power increases.

TABLE I Shock power and friction energy of G5 planetary ball mill for various ball milling conditions

Ball milling conditions Ω (rpm) / ω (rpm)	Shock power (W)	Friction energy (%)
212/50	1.1	4.5
212/400	1.4	50.1
300/50	3.0	3.6
300/400	2.6	20.8
424/50	8.4	2.2

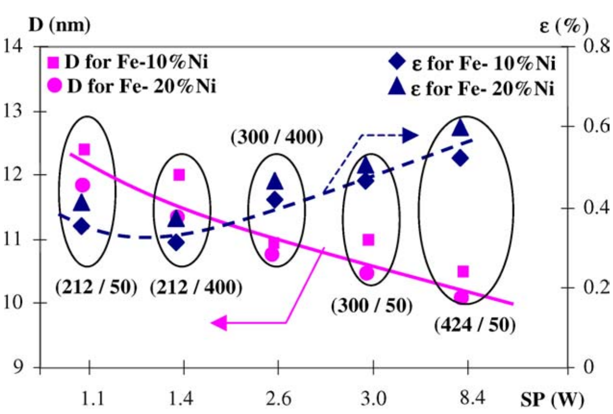

Figure 2 Evolution of the crystallite size (full line) and of the lattice strain (dashed line) for nanocrystalline Fe-10% Ni and Fe-20% Ni after 36 h of milling versus shock power (expressed in W).

Fig. 2 shows the evolution of the crystallite size and lattice strain versus the shock power for nanocrystalline mechanically alloyed Fe-10% Ni and Fe-20% Ni. It is observed for mechanically alloyed Fe-10% Ni and Fe- 20% Ni that generally, the higher the shock power, the lower the crystallite size and the lower the lattice strain. It is noted that the low values of the crystallite size with the high values of the lattice strain is found in materials prepared by SMP, whereas, the low values of the crystallite size with low values of the lattice strain is found in materials prepared by FMP. Using G5 planetary ball milling for mechanically alloyed Fe-Ni, Hays *et al.* [10] have shown that using FMP, the crystallite size decreases gradually versus milling time with particle size varying between 1 to 5 μm, whereas, in SMP, the crystallite size decrease sharply versus milling time with particle size varying between 20 to 100 μm.

Fig. 3 presents the magnetization evolution versus crystallite size for mechanically alloyed Fe-10% Ni and Fe-20% Ni. The increase of magnetization can be found to be linked to the grain size reduction to about 10 nm. Each grain may be treated as a single magnetic domain eliminating the influence from magnetic walls [8]. On the other hand, this increase in magnetization can also be attributed to the increase of the lattice parameter with long milling time. Amils *et al.* [11] have shown that the ball milling process produces a high density of defects, particularly of antisite type, which causes a 0.8% lattice parameter expansion in Fe-Al alloys. The same authors have previously reported on a good correlation between the saturation magnetization and the lattice parameter, which suggests that the magnetic transition observed may, in part, be related to changes in the density of states at the Fermi level. It is clearly shown that the magnetization can be affected by crystallite size and lattice parameter but not by the mode process used.

Fig. 4 presents the evolution of coercivity versus ball milling conditions for mechanically alloyed Fe-10% Ni

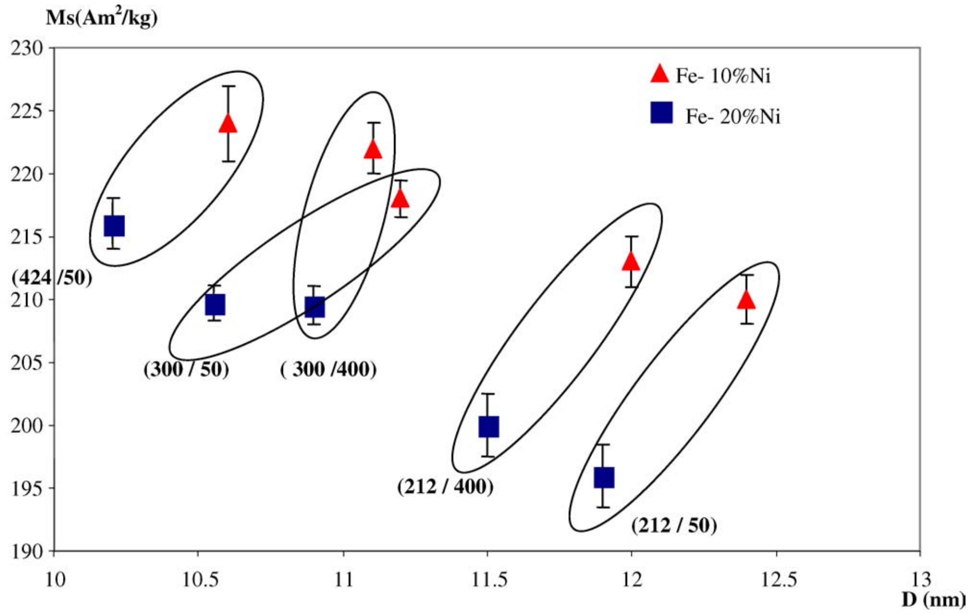

Figure 3 Evolution of saturation magnetization after 36 h of milling versus crystallite size for various ball milling conditions Ω (rpm)/*ω* (rpm).

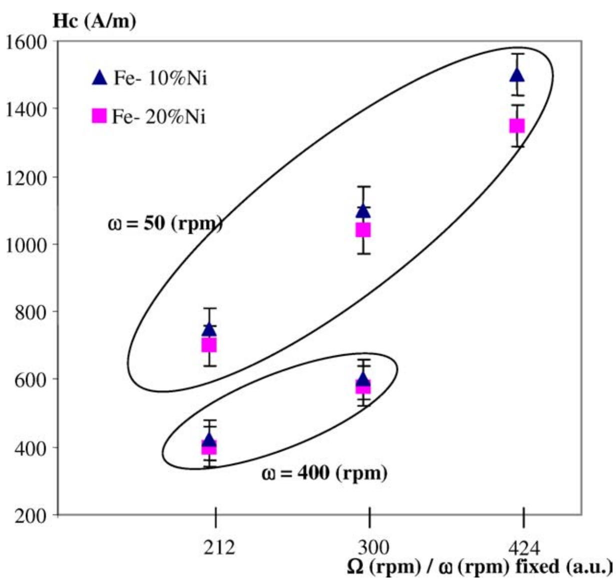

Figure 4 Evolution of the coercivity after 36 h of milling as a function of ball milling conditions Ω (rpm)/*ω* (rpm).

and Fe-20% Ni nanocrystalline powder. It is observed that in the case of SMP, the coercivity increases when the shock power increases, and in the case of FMP, the coercivity decreases when the friction energy increases. The increase of the coercivity in the case of SMP may be understood as due to a considerable introduction of high internal strains into the material, which is inevitably related to the process. Such a hypothesis is more under investigation. Thus, the magnetostriction in combination with the high internal strain is identified as the effect dominating the coercivity via magnetoelastic interaction [12]. The low values of the coercivity in the case of FMP can be due to the low values of the crystallite size and the dependence of coercivity and crystallite size (the lower the coercivity, the lower the crystallite size) that prevails over the other predominant strain influence in this crystallite size range. Nevertheless, the resulting nanocrystalline structure led to excellence soft magnetic properties (low coercivity) as suggested by Herzer [13]. The lowest coercivities, however, can be found again for the smallest structural correlation lengths like in amorphous alloys (grain size of the order of atomic distance) and in nanocrystalline alloys for grain size $D < 20$ nm [14].

4. Conclusion

In order to obtain soft magnetic materials, it is necessary to produce Fe-based alloys, with an average crystallite size largely below 20 nm and most probably even below 10 nm. Mechanical alloying is one of techniques that can synthesize such materials. The results presented in this work clearly show the great potential of this technique. High-energy ball milling of Fe and Ni powders results in the solid solution formation accompanied by a grain refinement. The magnetization is not affected by the mode used (SMP or FMP). The high values of coercivity have been found in materials prepared by SMP. Mechanical alloying (using the FMP mode) has been shown to be suitable for the production of soft magnetic nanocrystalline Fe-10% Ni and Fe-20% Ni.

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