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Mechanical Synthesis of Iron and Nickel Solid Solution by High Energy Ball Milling for Packaging

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ABSTRACT

Mechanical alloying (MA) is a solid-state powder processing technique involving repeated cold welding and fracturing of powder particles in a high-energy ball mill. It has been used to obtain nanocrystalline binary system FeNi. Fe and Ni elemental powders have been ball milled in a planetary mill (Pulverisette5, Fritsch) for various times up to several hours. The morphology of the powders was examined using scanning electron microscopy (SEM). X-ray diffraction (XRD) has been employed to follow the structural evolution during the ball milling process. X-ray pattern of argon-milled powders show NiFe Solid solution after 30 h milling accompanying. Furthermore, SEM micrographs show that increasing milling time to 70 hrs results in cold welding phenomena to be main phenomena. It also became clear that at the time of 50 hours, between fracturing and cold welding the balance phenomenon was created.

KEY WORDS: Ball milling, Solid Solution, SEM, XRD

INTRODUCTION

In recent years, nanoparticles construction has been considered very much, and this is because of their different features such as: magnetic, electric, optic and chemical characteristics [1]. From all nanoparticles, magnetic particles have a special importance because of expanded applicability in industry. FeNi nanoparticles are a group of magnetic materials which have distinct characteristics such as high saturation magnetism, low coercivity and high permeability. This product has many applications in electronic and magnetic industry such as use in inductors, data storage tools, sensors and magno-optic devices [2, 3]. Properties of these particles have been improved by lowering their size. So, different methods have been studied for making particles with smaller sizes. Many construction techniques such as heat evaporation [4], electric settlement [5], heat spray [6], laser analysis [7] and mechanical alloying [8-10] used for making FeNi nanoparticles, that, among them, mechanical alloying method (MA) has been more successful for producing pure, homogenous and little samples [11]. What is happening in mechanical alloying is a complex process including fracturing , deformation, cold welding and permission in little distant which happens among powder layers [12]. Milling time length is one of the important parameters in milling operation. Usually the time length chosen for milling operation is the one in which a balance state is created between fracturing and cold welding in particles powder. The time required is different based on following parameters, from which, type and speed of milling, ration of ball to powder and the temperature are mentioned. It should be considered that by increasing the operation time, there could be a possibility of contamination increase and creation of unwanted phases. So, optimized conditions should be determined for MA process.

In current research it is tried to create an optimum understanding from mechanical alloying in Fe-Ni system with the use of microstructures by scanning electron microscopy (SEM) and X-ray diffraction device. The influence of milling operation time parameters and weight ration of ball to the powder, studied for the related system, too.

EXPERIMENTAL METHOD

In this research, after purchasing Fe powder with the purity of 99.5% and Ni powder with purity of 99.98% (product of Sigma Aldrich) and calculating stoichiometry ratio of Ni and Fe (with the ration $Fe_{50}Ni_{50}$) and then weighing primary material, powders and balls were put into milling vial. Milling operation was done in milling satellite ball Fritsch-P5 and hardened steel vial and steel balls with 10 mm diameter in constant speed of 300 rpm. Some Toluene used as reaction controller factor (PCA), for prevention of powder particles aggregation, considering weight ration of ball to the powder in each test. Some papers studied the impact of PCA in material chemical reactions in alloying process, and it became obvious that use of reaction controller factor in little amounts has no impact on the product chemical composition [13]. It should be mentioned that all phases of the test such as

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weighting, powder charge into the vial and the powder extraction after milling operation for preventing oxidation and unwanted contamination entrance into glove box vial are done under Ar controlled atmosphere.

Increase in balls weight and vessel weight and total powder weight reduction in each test shows the creation of a covering from powders on balls surface and interior walls and vessel floor. For separating this created covering from balls and vial, after each test, for 10-15 minutes, balls were milled without Argon gas with the speed of 300 rpm and prepared for the next test.

After each test, the products discharged from vial, weighted and sent for analysis. For determining grain size and phase changes, qualitatively and semi-quantitatively, X-ray diffraction device (XRD), D8 advanced model, product of 2002 in Bruker company used with a CuKα lamp (λ=1.54Å) and Ni filter. As usual, most crystalline size was measured by measuring Bragg peak band in half of intensity maximum which is acquired by Schrerrer formula:

$$D = \frac{0.9\,\lambda}{\beta \cos\theta}$$

Where D is crystalline size, λ is wavelength of used ray, β is peak wide in half of the most intensity (radiation) and θ is Bragg angle.

Range of X-ray diffraction angle is from 20 to 70. For the study of particles size differences in milling operation, images of field emission scanning electronic microscopy (FESEM) were used. For providing these images, very little amount of milled powder poured on carbon glue which is connected to sample vessel so that powder particles would not piled on each other. For preventing electron aggregation inside the sample, gold covering was used by DC spattering device. Then sample-holder is placed in device and after maintaining proper vacuum, imaging is performed. FESEM device used has Hitachi label, S4160 model, product of Japan in 1996.

RESULTS AND DISCUSSION

Results of field emission scanning electron microscopy are: In figure 1(a) as it is shown by the arrow, because of cold welding phenomena, powder layers piled on each other and made an increase in particles size. By continuing milling process up to 50 hours, because of crystalline defects enhancement and mechanical hardening operation phenomenon, particles hardening increased and the condition for their breakdown was provided, so particle size decreased and their distribution became more monotonic. Increasing milling period up to 70 hours, not only didn't reduce particles sizes, but also provided the condition for prevailing over cold welding phenomenon and the increase of particles sizes. This is because of total increase in temperature at the time of milling. By more precision in figure (c) it can be seen that because of ferromagnetic characteristics of Fe and Ni elements, powder fine particles, aggregation happened.

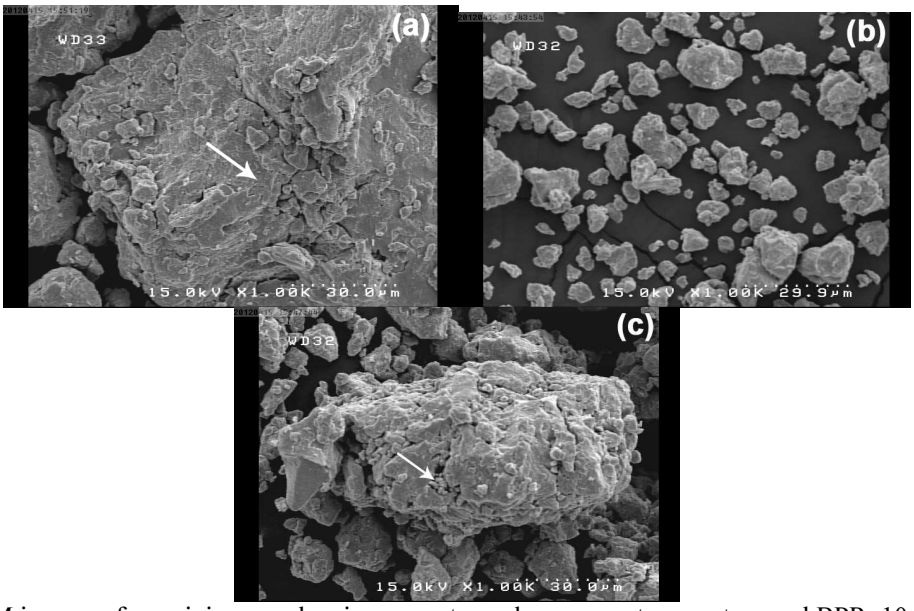

Figure 1- SEM images of remaining powders in argon atmosphere, room temperature and BPR: 10:1, in different milling times, (a) 30h, (b) 50h, (c) 70h.

For the study of weight ration parameter of ball to powder, as it can be seen in figure 2, at the time of 50 hours, with BPR enhancement, no considerable changes observed in particles size and their homogeneity was kept. This can be related to the balance between the two phenomenon of cold welding and fracturing at the time of 50 hours. It should be mentioned that in BPR less weight ratios, because the two elements of iron and nickel are soft, powder particles are piled on each other layer by layer in primary times of milling, and this can be found by comparing figure (a) and (b).

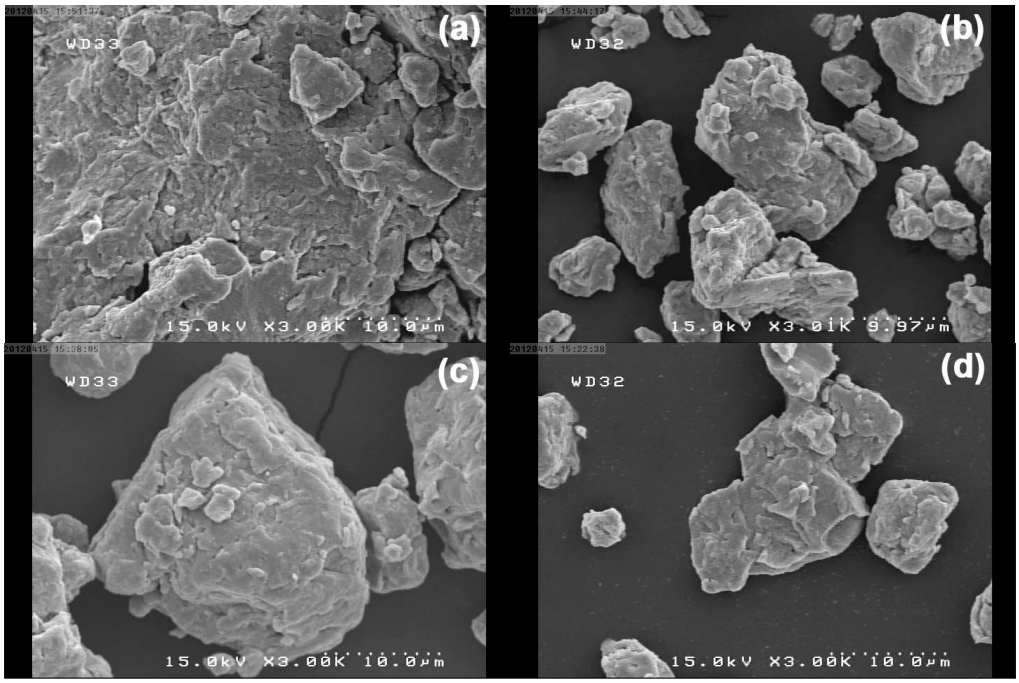

Figure 2- SEM images of remaining powders in argon atmosphere, room temperature, for various BPR and milling times: (a) BPR: 10:1, 30h, (b) BPR: 10:1, 50h, (c) BPR: 20:1, 50h, (d) BPR: 30:1, 50h.

X-ray diffraction analysis results

considering figure 3, X-ray diffraction analysis results, it can be seen that immediately after milling operation, at the time of 30 hours, peaks related to pure elements of Fe and Ni disappeared and a new peak was created in angles less than 45 degrees.

Results of XRD graphs analysis show that the acquired peak is related to the NiFe solid solution. Also with the use of Schrerrer relation, size of powder grains calculated in different times and the results provided in figure 4. It can be seen that size of grains has been decreased with the increase in milling time which can be considered a parameter for decreasing perm eation distance and provides the condition for Fe and Ni permeation into each other crystalline structure and then, NiFe solid solution is made. As can be seen in Fig. 4, during the early stage of milling the crystallite size decreases rapidly in the beginning of milling and slows down afterwards and becomes gradually smaller with increasing milling time with a final value of about 15.3 A^0. This value is smaller than the 45 nm [14] and 16.5 nm [15] for the iron–nickel ultra-fine particles obtained by a gas- condensation method, with the same composition.

The lattice parameters (d) for (Fe, Ni) are plotted in Fig. 5 as a function of milling time. The value of lattice parameters increased from d = 2.05 A^0 for 30 h to 2.08 A^0 for 70 h of milling. Such a minute evolution in the lattice parameter is conceivable since the atomic radii of Fe and Ni are close [16]. The increase in lattice parameter with milling time is due to the disordering of the alloy as observed by Chen et al. [1] in the case Fe–Ni. The lattice constant (a) obtained as a consequence of the alloying process increase with milling time and reach the final value of a=3.5840 A^0 as seen in Fig. 6.

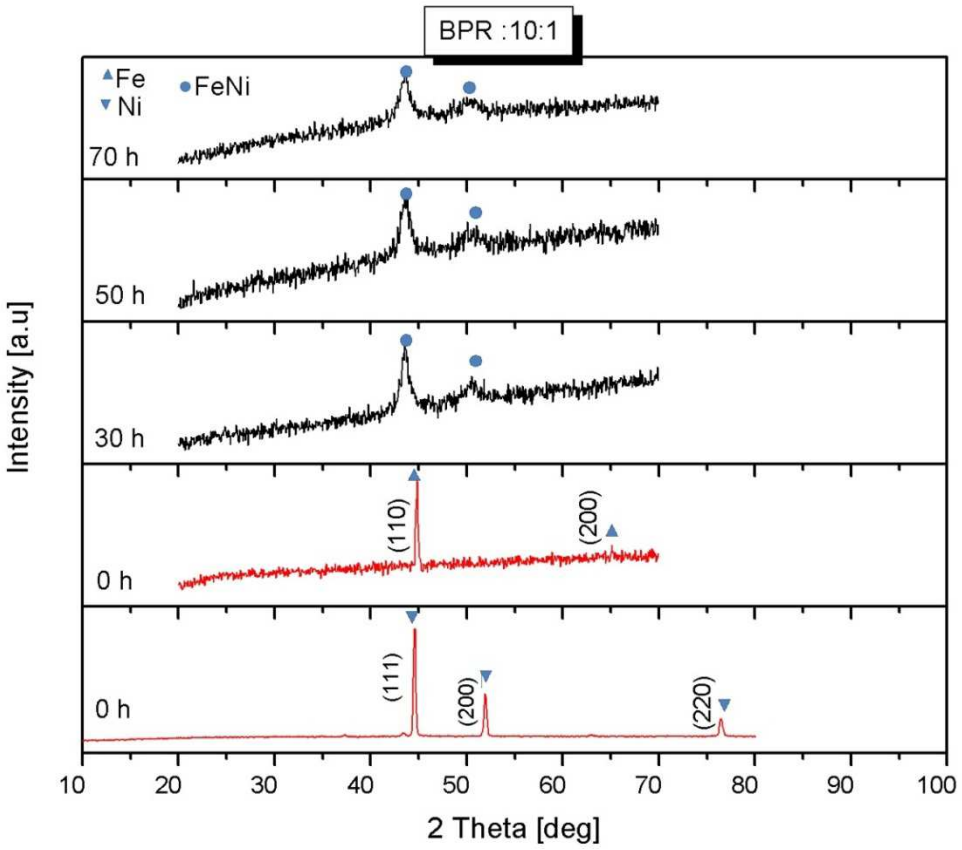

Fig. 3. X-ray diffraction patterns of $Fe_{50}Ni_{50}$ powder alloys synthesized in high-energy mill for various milling times.

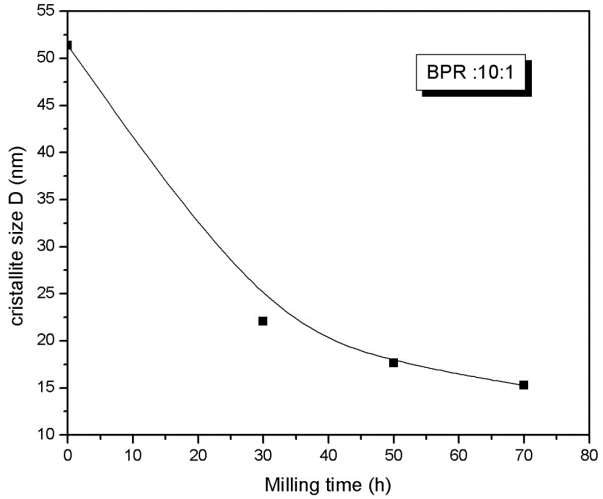

Fig. 4. Crystallite size of the Fe–Ni powders as function of milling time.

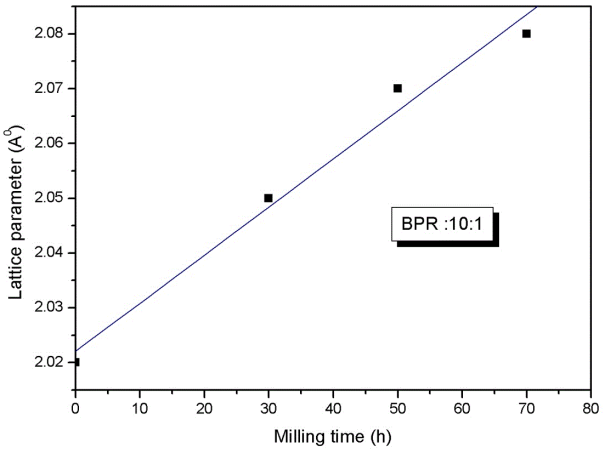

Fig. 5. Lattice parameter of (Fe, Ni) powders synthesized in high-energy mill for various milling times.

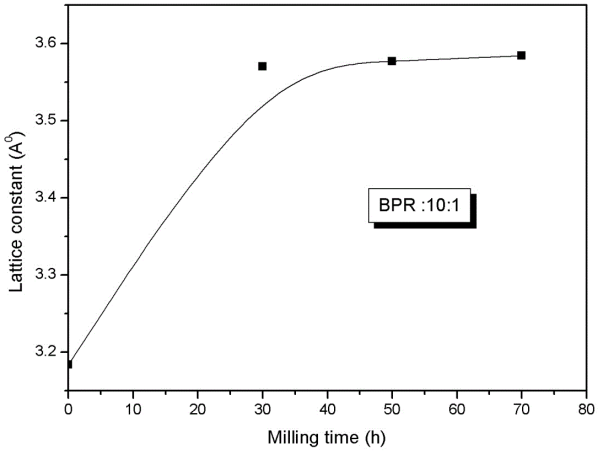

Fig. 6. Lattice constant of (Fe, Ni) powders synthesized in high-energy mill for various milling times.

Conclusion

1. By milling operation, NiFe solid solution produced.
2. Considering SEM images, it can be seen that at the time of 50 hours, with an increase in BPR, no considerable change was seen in particle size and there homogeneity was kept. This can be related to the balance between the two phenomena of cold welding and fracturing at the time of 50 hours.
3- Considering X-ray diffraction analysis results (XRD) it can be seen that the size of grains decreased with an increase in milling time, and the condition for Fe and Ni atoms permeation into crystalline structure was prepared and so NiFe solid solution was created.

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