

## A Review On Mechanical Alloying

K V Nagesha<sup>1</sup>, Rajanish M<sup>2</sup>, D.Shivappa<sup>3</sup>

<sup>1</sup>Assistant professor, Department of Mechanical Engineering, AIET, Moodbidire

<sup>2</sup>Assistant professor, Department of Mechanical Engineering, DSATM, Bangalore

<sup>3</sup>Professor. Department of Mechanical Engineering, BAE, Bangalore

### Abstract

Ball milling is one of the most extensively used technique to produce ultrafine materials. The ball milling process is widely used in ceramic and metal processing industries. Ball milling consists of repeated fracture, mixing, and cold welding of a fine blend of metal, oxide, and alloy particles resulting in size reduction and sometimes in chemical reactions. In recent years the ball milling process is employed to prepare nanostructured materials which are intensively studied, particularly because the physical and chemical properties of these materials are quite different from those of the bulks material. In nano-materials research, this technique is well used to fine-tune the grain sizes of the materials in nano-scales. However scientists and engineers are optimizing the processing parameters and machine construction to obtain powders with desired size and performance characteristics. The purpose of this work is to review the findings of some of the researchers, relevant to the ball milling process for soft magnetic materials such iron, cobalt and nickel and to emphasize the importance of key process parameters on grain size, particle size and performance characteristics.

### 1. Introduction

Mechanical milling involve the use of mechanical forces such as compressive force, shear or impact to effect particle size reduction of bulk materials. This is sometimes referred to as mechanical alloying or ball milling. Ball milling of powder particles as a method for material synthesis has been developed as an industrial process to successfully produce new alloys and phase mixtures, since 1970 [1]. This powder metallurgical process allows the preparation of alloys and composites, which cannot be synthesized via conventional routes. In nano-materials research, this technique is well used to fine-tune the grain sizes of the materials in nano-scales. Nanostructure is obtained by repeated mechanical deformation and alloying as the powder is vigorously shaken in a vial or jar containing a number of milling balls [2 & 3]. The energy transfer to the powder particles in these mills takes place by a shearing action or impact of the high velocity balls with the powder. The size of the powder particle depends on several factors such as

Milling speed, Type of milling equipment, Size of balls used and Ball to powder weight ratio

### 2. Review of previous work

H.F.Li and Ramanujan, School of Materials Engineering, Nanyang Technological University, Singapore, have studied the microstructure evolution and formation of nanocrystalline FeCo based alloys by mechanical alloying. They have reported that planetary ball milling of 22 mesh iron powder with 100mesh cobalt powders with a ball to powder ratio 10:1 and speed of 300rpm would result in powder particle size of 3.3  $\mu\text{m}$  after 70hrs. Table 1 shows the reduction in particle size as a function of milling time and composition. They have characterized the powders using SEM, TEM and X-ray analysis. Finally they have concluded that the milled powders are not magnetically soft but a variation in coercivity with milling time has been observed that is related to the microstructural evolution. After 5hours of milling, the saturation magnetization for the unalloyed  $\text{Fe}_{50}\text{Co}_{50}$  and  $\text{Fe}_{44.5}\text{Co}_{44.5}\text{P}_7\text{B}_4$  were 175.7emu/g and 167.2 emu/g [4].

Table: 1 Powder particle size (in  $\mu\text{m}$ ) of as milled powder after different period (in hours) of milling

Milling time/ composition	5h	10 h	15 h	20	50	70
$\text{Fe}_{50}\text{Co}_{50}$	19.6	12.3	5.0	4.6	4.3	3.3
$\text{Fe}_{44.5}\text{Co}_{44.5}\text{P}_7\text{B}_4$	3.2	2.1	1.7	1.5	1.4	1.1
$\text{Fe}_{41}\text{Co}_{41}\text{P}_{14}\text{B}_{14}$	2.6	3.1	1.6	-	-	-
$\text{Fe}_{42.5}\text{Co}_{42.5}\text{P}_7\text{B}_8$	9.4	1.8	1.9	-	-	-

Yang Yuanzheeng, et al, at the Academia Sinica, Heifa have studied nano-structure of iron formed by mechanical milling. They have conducted the experiments using Pulverisette-5 planetary ball mill with steel vials and steel balls. The starting material used was iron powders with an average particles size of 76  $\mu\text{m}$  powders. The ball to powder weight ratio (BPR) was 20:1. Milling speed was set to 230 rpm and powder particles were milled for 80h in one vial. Powder sample was picked up from the

vials after selected interval of milling time to see the change in shape and size reduction of powder sample. The powder milling resulted in particles with grain size of up to 6nm and the conclusion of this work is that mechanical grinding is an effective method to produce nanometer powders of pure elements. [5]. Fig. 1 shows the reduction in crystallite size and increase in strain as a function of milling time.

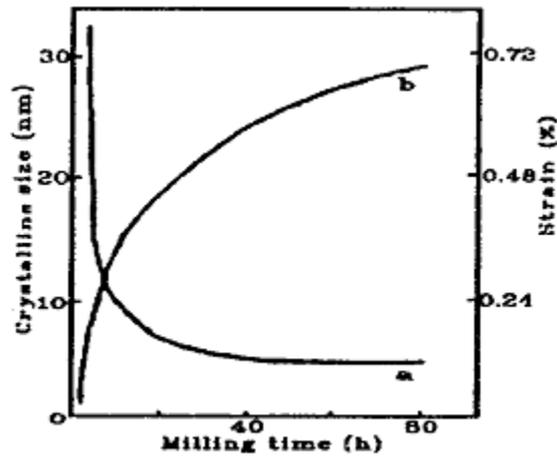


Fig 1 Reduction in crystallite size and increase in strain as a function of milling time.

R.Hamzaoui of University de Technologie de Belfort-Montbeliard, France, has studied the structure and magnetic properties of nanocrystalline mechanically alloyed Fe-10wt % Ni and Fe-20 wt. %Ni alloys of mixtures. The average particle size of Fe was 7 $\mu$ m and Ni was 250 $\mu$ m; the ball to powder ratio 10:1, and rotational speed was kept constant at 400rpm. They have reported that the crystallite size decreases from 54 $\pm$ 1.5 to 10.4 $\pm$ 1.5nm and the internal strain  $\epsilon$  increases from 0.10  $\pm$  0.05 to 0.69  $\pm$  0.05. The crystalline size and lattice strain with respect to Fe-20 Ni is D= 51.2 $\pm$ 1.5 to 9.8 $\pm$  1.5 nm for 96hrs of milling and lattice strain  $\epsilon$  = 0.11 $\pm$  0.05% to 0.72  $\pm$ 0.05 %. The conclusion of this work is Fe and Ni powders form solid solution accompanied by grain refinement and grains reduced resulted in an increase of the magnetization and a decrease of the coercivity values as shown in the graph. (Fig.2) [6].

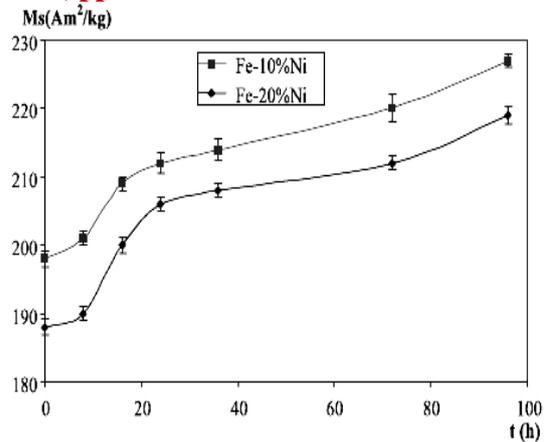


Fig 2 (a). Evolution of saturation magnetization versus milling time

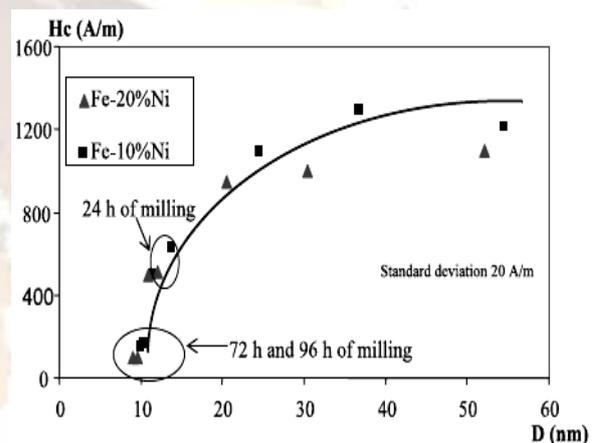


Fig 2 (b). The coercivity as a function of crystallite size

W.Pilarczyk of Institute of Engineering Materials and Biomaterials, Poland, have studied the structure and properties of Fe-Co-Ni-B-Si-Nb alloy prepared by mechanical alloying method. In this paper the test material was the mixture of iron, cobalt, nickel, boron, silicon, niobium material with different proportions. The mechanical alloying process was done using high energy SPEX 8000 mill of the shaker type under inert argon atmosphere. The ball to powder weight ratio is 9:1 and the powder were grounds for 10, 45, 100hrs. The crystallite size is measured by using Scherrer's method. The conclusion of this paper was that powder structure becomes more homogeneous after 100hrs of mechanical alloying with the particle size in the range of 0.4 $\mu$ m [7].

D.A. Sanchez of Universidad Autonoma de Coahuila, Mexico, have reported that the material used for experiment was high purity cobalt nitrate hexahydrate, iron nitrate, sodium hydroxide, citric acid monohydrate and ethylene glycol. In ball milling experiment milling was performed in a planetary ball mill, the milling time is 4hours at a speed of 300rpm with ball to powder mass ratio

10:1. After 4hr of ball milling the materials have improved crystallinity of magnetic phase with increasing temperature the growth of crystalline from 5nm to 26nm has also been reported [8].

A.M. Glushenkov, Department of Electronic Materials Engineering, Research School of Physical Science and Engineering, Australia have performed reactive ball milling experiments to produce nanocrystalline ZnO. The experiment were carried out in oxygen atmosphere leading to nano crystalline ZnO. Initially the powder particle size was 150 microns after 100hrs of milling the powder particles had crystallite size of 10 to 100nm. The conclusion drawn from this paper is that mechanochemical procedure is simple and applicable to producing material in large quantities and the oxidation of zinc is a gradual process in contrast to combustion oxidations of other metals [9].

S.R.Mishra, et al, University of Memphis, USA, have studied the magnetic properties of iron nitride – alumina nano-composite materials prepared by high-energy ball milling. In this study milling was carried out in Pulverisette-5 planetary ball mill with WC vials and WC balls. Starting material used for milling was 99.9% pure 50 $\mu$ m iron nitride powders. The ball to powder weight ratio (BPR) was 20:1. Milling was conducted at 400 rpm. Powder particles were milled for 64h. The reduction in the particle size of Fe<sub>x</sub>N leads to a decrease in the saturation magnetization (62.5 emu/g) and a corresponding increase in the coercivity. The observed enhancement in coercivity results from a combination of particle size effect, surface defects, and mechanical stress. The presence of a super paramagnetic component in the Mossbauer spectra and the decrease in saturation magnetization are related to the grain size. The observed increase in the isomer shift and rapid decrease in hyperfine field in the highly dispersed magnetic particles of (Fe<sub>x</sub>N)<sub>0.2</sub>(Al<sub>2</sub>O<sub>3</sub>)<sub>0.8</sub> are compatible with surface defects [10].

S.W. Du, R.V.Ramanujan, School of Materials Engineering, Nanyang Technological University, Singapore, have studied the mechanical alloying of Fe-Ni based nanostructured magnetic materials. The various alloys composition Fe<sub>49</sub>Ni<sub>38</sub>B<sub>18</sub>Mo<sub>4</sub>, Fe<sub>49</sub>Ni<sub>46</sub>Mo<sub>5</sub> and Fe<sub>42</sub>Ni<sub>40</sub>B<sub>18</sub> were processed from elemental powders by mechanical alloying of Fritsch pulverisette 5 planetary ball milling under argon atmosphere from pure elemental powder iron, nickel, boron and molybdenum. The ball to powder ratio was kept constant at 8:1. The milling speed was 300rpm for milling times ranging from 1 to 100h. Initially the raw material particle size was iron-35 $\mu$ m, nickel-150 $\mu$ m, boron-46 $\mu$ m, and molybdenum 3 to 5 $\mu$ m.

The grain size of alloy composition of Fe<sub>49</sub>Ni<sub>38</sub>B<sub>18</sub>Mo<sub>4</sub> was 6nm; correspondingly alloys of Fe<sub>49</sub>Ni<sub>46</sub>Mo<sub>5</sub> and Fe<sub>42</sub>Ni<sub>40</sub>B<sub>18</sub> yielded particles with grain size in the range of 8 to 20nm. They have reported that addition of molybdenum and boron have a dramatic effect on the magnetic properties of mechanically alloyed Fe and Ni based material [11].

D.Oleksakova, S.Roth, P.J.Safarik University, Slovakia Germany, have studied the soft magnetic properties of NiFe compacted powder alloys. In this paper they have reported mechanical milling using high energy planetary ball mill in hardened steel vials with steel balls. The milling was performed in the protective argon atmosphere with ball to powder ratio of 6:1 and with a speed of 180 rpm. The conclusions of this paper are that NiFe (81 wt% of Ni) form from a single –phase system and the mechanical milling of NiFe (81 wt% of Ni) results in decrease in coercivity as a function of grain size [12].

H.R. Madhaa Hosseini, have studied preparation of nanocrystalline Fe-Si-Ni soft magnetic powders by mechanical alloying and its on the magnetic properties and microstructure of these alloys. In this research work the powder mix were ball milled under argon atmosphere, iron, silicon and nickel with a ball to powder ratio 10:1 and speed 700rpm. The initial grains size was 400nm, after 100 hours of milling the grain size reduced to 12nm. Detailed characterization of the powders has been done using SEM, and X-Ray analysis. Finally they have concluded that the milled powders had metastable bcc solid solution of Si and Ni in Fe with a fine particle structure. The maximum saturation magnetization (M<sub>s</sub>) and minimum coercivity (H<sub>c</sub>) were obtained at the optimum composition of Fe<sub>85</sub>Si<sub>10</sub>Ni<sub>5</sub> after 70hrs milling [13].

El-Eskandarany et al studied the effect of milling speed in a Fritsch pulverisette-5 mill. They reported that the time required for the formation of an amorphous phase in the Co-Ti system decreased with an increase in the rotation speed. While it took 200h for the formation of the amorphous phase at a speed of 65 rpm, it took only 100h at 125rpm and 24h at 200rpm. They had observed acyclic crystalline amorphous crystalline transformation on continued milling [14].

The effect of milling speed during milling of a Fe-7 wt % C-6 wt % powder blend in a planetary ball mill was investigated at three different speeds of 80, 100, and 120 rpm by Rochman et al. [15]. They noted that the solid solubility of carbon in iron increased with increasing speed of operation of the mill for the same milling time. For example, they reported that on 100h of milling, the solid

solubility of C in  $\alpha$ -Fe was 0.2 at % at 80rpm, 0.5 at% 100rpm, and 1.3 at % 120rpm [16].

### 3 Conclusion

The inferences that can be drawn based on the literature review given above are:

a) Ball milling processing parameters (ball to powder ratio, size of the balls, material of the balls, jar size, milling time, milling speed) have a strong influence on the particles size of brittle materials which undergo particle size reduction, and in the case of ductile materials there is a reduction in crystallite size due to sever plastic deformation as a result of high energy impact.

b) There is a need for a mathematical model, which relates processing parameters to particle size or grain size for a given material. Such a model would accurately predict the rate of grain size or particle size reduction as a function of processing parameters, thus facilitating accurate control of powder characteristics.

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