Evolution Study of Crystal Parameter for Iron Powder Subjected to Mechanical Milling

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Abstract

In a planetary mill with balls of high energy, the experiences of milling on different time interval of iron powder were done.

The iron powder with 99.9% purity was introduced in four vials, having every one on average mass of 1.750 kg, varying the number and diameter of balls.

It was noticed, there is a tendency to increase accentuated the crystallographic parameter in the interval of 7.5-9 mm ball radius, it is admitted the same interval to be more efficiently on milling from point of view of evolution of crystal parameter from experimental data.

Keywords

Mechanical milling; Iron powder; Crystal parameter; Dislocation

1. Introduction

The object of process of mechanical milling in mills with balls of high energy is the disintegration of particles with the energy resulting from the moving of milling bodies.

Mechanical milling includes many hardening mechanisms [1]:

- the fine size of particles;
- the high density of dislocation;
- the high hardness by precipitation.

The mechanical milling leads to obtaining of hard materials with high mechanical strength. Obtaining of phases of high hardness on precipitation, process, may introduce problems of corrosion and distortions of slipping planes. During milling the powder reacts with atmosphere of milling.

During milling process, take place the transmitting the impact energy of balls to particles of powders, with implications in their morphology and hardness.

An important variable of process is the development temperature during milling which results from the transformation of kinetic energy in heat and which warms the powder. The temperature may allow critics values for some powders, starting reactions or and form solid solution. Some reactions take place in solid environment if energy of impact is high enough to force the atoms to pass over the starting activation energy.

In the same time the particles of powders are deformed in the same way and subjected continually to process of mixing.

The powder may have a fine microstructure with nanocrystalline grains [2]. The scope of present research is the note the efficiently of milling on iron powder, varying the ball diameter.

2. Description of Mill

The planetary mill has 6 vials, mounted on a plate support; velocity of rotation of plate of planetary mill is n_p = 1200 rpm, velocity of rotation of containers is $n_c = -1.25\ n_p$ and is produced by construction.

Initial parameters are: radius of disk: R (250 mm) and radius of vials, r have a mean weight of 1.750 kg approximately. Each used vial contained a certain mass of balls: m_b with a diameter D (figure 1).

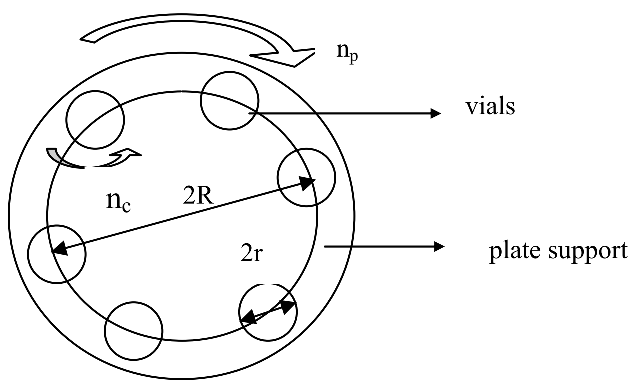

Fig. 1. Mill configuration

3. The Experiments, Results, Discussion of Experimental Results

The experiences took place in the planetary mill with ball of high energy, figure 1, where had been used four vials for these experiments. The vials were produced of OLC 45 steel and the balls of RUL.2 steel. In vial number one are introduced 32 balls with diameter D =18.6 mm.

In vial number two are introduced 13 balls with diameter D = 22 mm, in vial number three are introduced 13 balls with diameter D = 22 mm and in vial number four are introduced 32 balls with diameter D = 12.6 mm.

In every vial is introduced 100 g of iron powder with purity of 99.9 % having a grain size under 200 µm. The milling is done without protective atmosphere and the ratio of weight ball to weight powder was about 8:1.

The experiences were produced for different intervals of milling time; during milling the mill was stopped approximately five minute, necessary for vials to cool dawn.

There were used as milling time interval of 0, 10, 20, 40, 80, 160, 320 minutes.

The energy of impact was transformed mainly in heat, which warm ball, vials and powder principal in warmth having like effect warming ball and plane surface and a quantity of this shall be introduced in powder, producing disorder of structural [3].

In powder is produced a structural disorder also [4].

The term effect gives on intensifying of diffusion. The process of volume diffusion in particle powder is accelerated by the increasing of density dislocations results at plastic deformations.

Figure 2 gives value of crystallographic parameter for iron after 10 minute of milling: 285.58 pm; at 20 minute, with 287.12 pm; at 40 minute with 287.12 pm; at 80 minute with 287.12 pm; at 160 minute: with 287.08 pm; and at 320 minutes with 286.37 pm.

The cut of experimental results line with abscissa gives a_{00} and the line slope is b.

It is noticed oscillations of values till 20 minutes milling after which there is a tendency of decreasing of parameter.

The investigation of figure 2 it shows that a_{00} = 286,832 pm, b = -0.00059 (sloping of straight line).

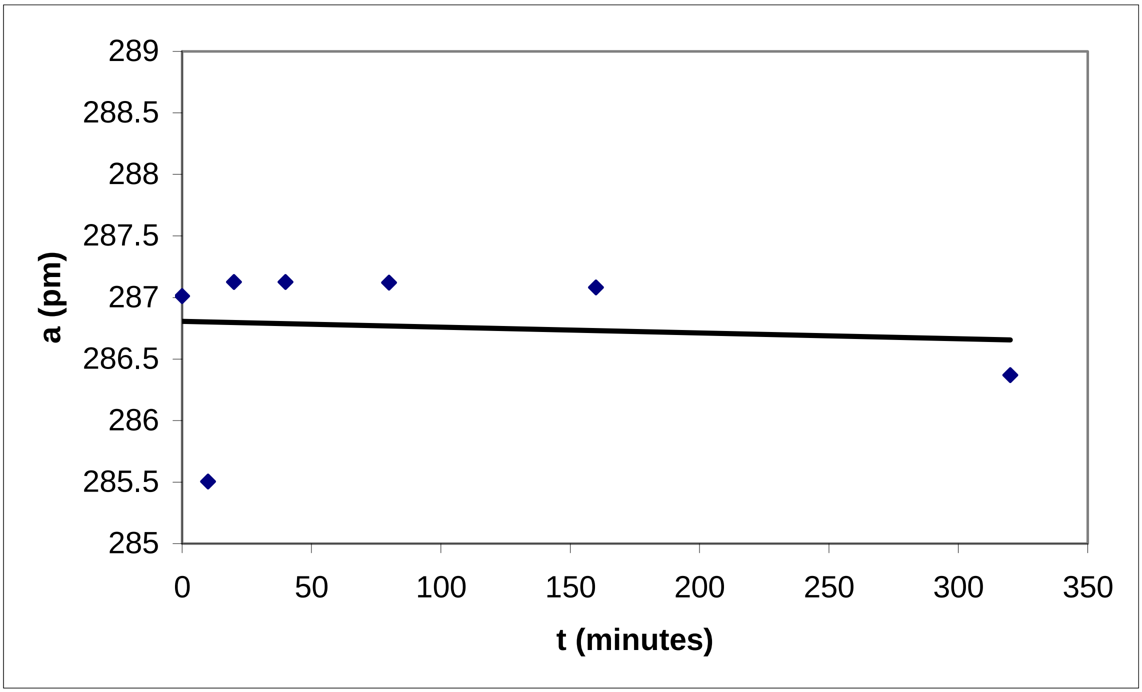

Fig. 2. Crystallographic parameter of iron powder

vial 4, radius D/2 = 6.3 mm vs. milling time

Figure 3 shows at 10 minute of milling the value of crystallographic parameter at 287.15 pm, at 20 minutes milling with 287.08 pm, at 40 minute of milling with 286.12 pm, at 160 minute of milling with 287.30 pm and at 320 minute of milling with 287.29 pm.

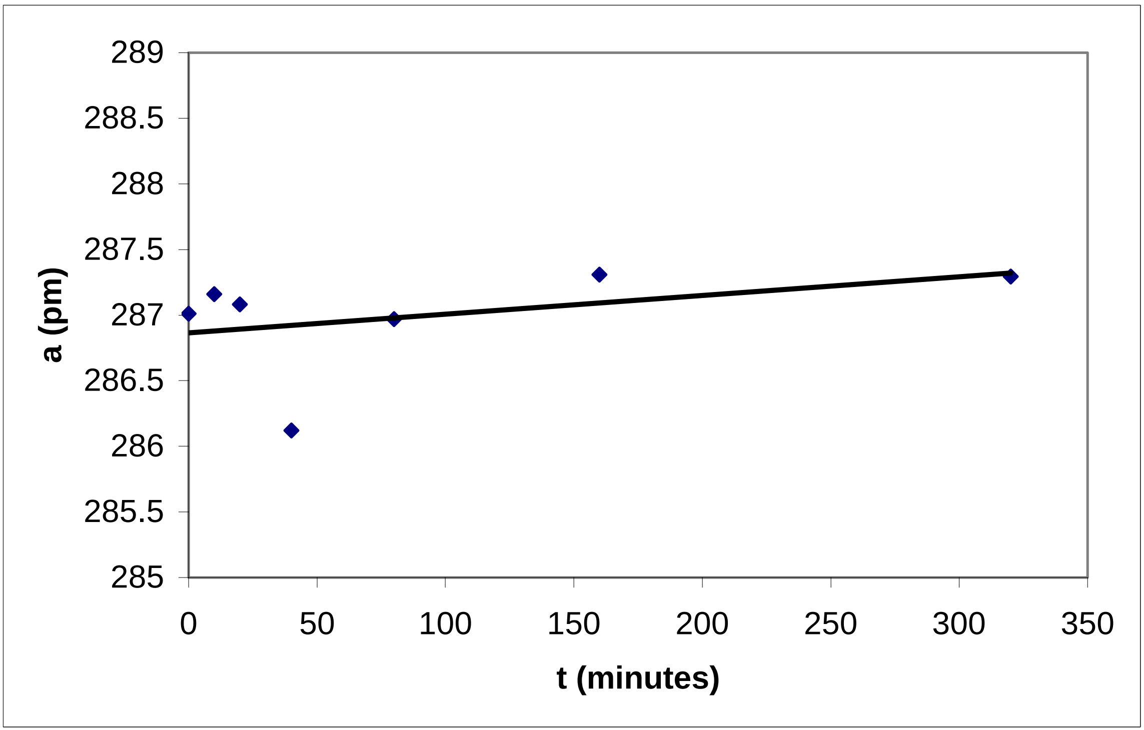

Fig. 3. Crystallographic parameter of iron powder

vial 3, radius D/2 = 7.5 mm vs. milling time

It is noticed an easily increasing of crystallographic parameter till 40 minute, after that an easily decreasing. The investigation of figure 3 it shows that a_{00} = 286.88 pm, b = -0.00133 (sloping of straight line).

Figure 4, vial 1 the crystallographic parameter at 287.09 pm at 0 minute of milling, at 20 minute of milling with 287.15 pm, at 80 minute of milling with 287.56 pm, at 160 minute of milling with 287.84 pm and at 320 minute of milling with 288.80 pm.

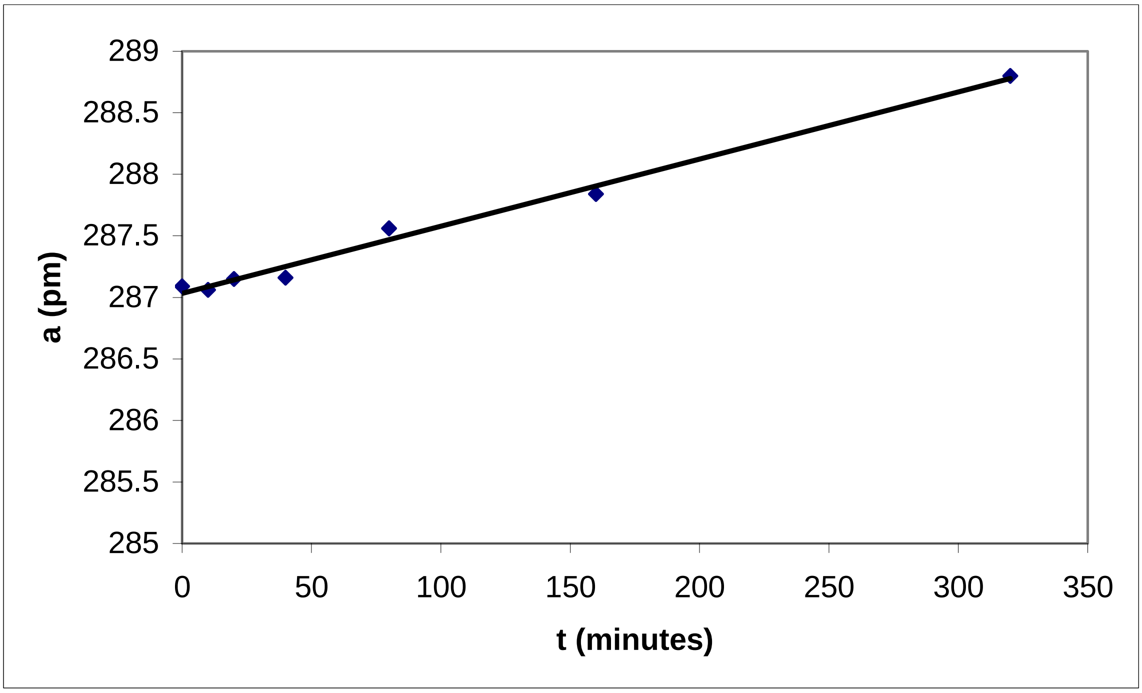

Fig. 4. Crystallographic parameter of iron powder vial 1, radius D/2 = 9.1 mm vs. milling time

It is noticed a tendency of linear increasing of crystal parameter of milling time noticing that thing at fourth figure after that. The investigation of figure 4 it shows that a_{00} = 286.225 pm, b = -0.00546 (sloping of straight line).

Figure 5 present the value of crystallographic parameter at 287.10 pm at 10 minute of milling, at 20 minute of milling with 287.39 pm, at 40 minute of milling with 287.08 pm, at 160 minute of milling with 286.99 pm and at 320 minute of milling with 286.37 pm.

It is noticed an easily increasing of crystallographic parameter till 20 minutes, after which an easily decreasing occur. The investigation of figure 4 it shows that a_{00} = 287.225 pm, b = -0.00242 (sloping of straight line).

In all figures 2-5 are noticed a linearity with time of increasing and decreasing of crystallographic parameter.

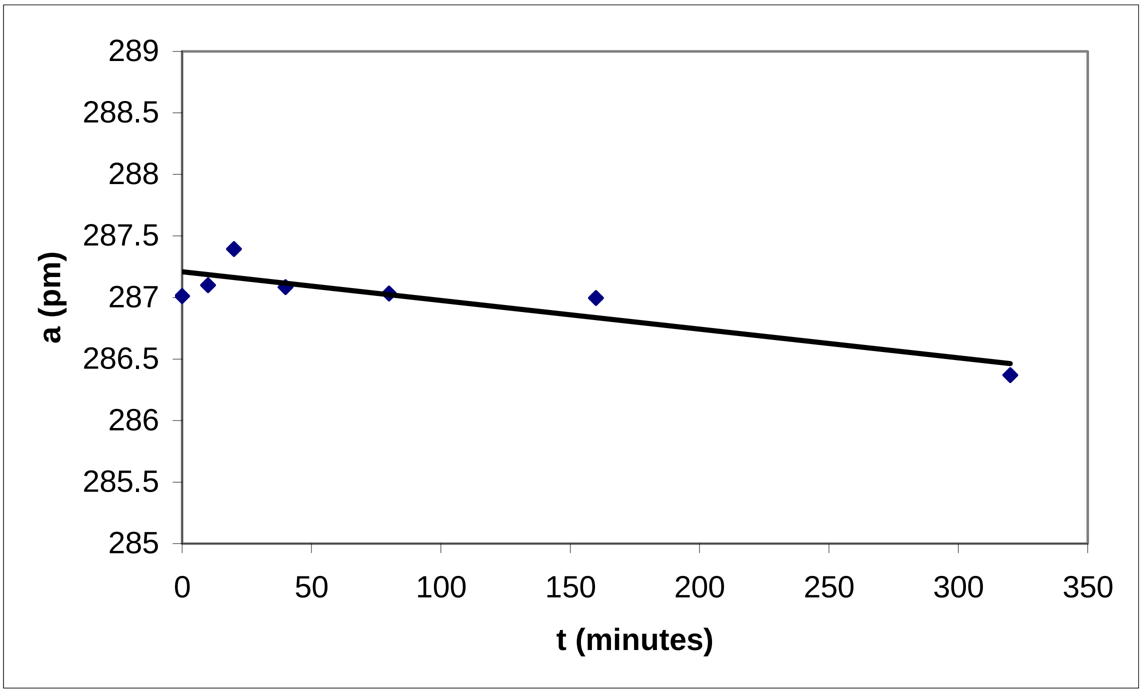

Fig. 5. Crystallographic parameter of iron powder vial 2, radius D/2 = 11 mm vs. milling time

The milling powder at mention time has been subjected diffractions with raze X to obtain crystallographic parameters using the last square method. In case of milling process, the energy transferred in system is the base the influence of parameters this energy depends of mill type. It produces mechanical disorder which leads to an increasing of Gibbs free energy.

The following was parameters in our investigation: ball mass and velocity, and ratio mass balls/mass powder, time of process, and volume filling of vial.

Table.1. Parameter a_0 versus of milling time (minutes), number at balls and radius of balls

No.	a_o, pm	minutes	balls	D/2, mm		No.	a_o, pm	minutes	balls	D/2, mm
1	287.09	0	73	6.3		15	287.09	0	32	9.1
2	285.55	10	73	6.3		16	287.06	10	32	9.1
3	287.12	20	73	6.3		17	287.15	20	32	9.1
4	287.12	40	73	6.3		18	287.16	40	32	9.1
5	287.12	80	73	6.3		19	287.56	80	32	9.1
6	287.08	160	73	6.3		20	287.84	160	32	9.1
7	286.37	320	73	6.3		21	288.8	320	32	9.1
8	287.09	0	46	7.5		22	287.09	0	13	11
9	287.15	10	46	7.5		23	287.1	10	13	11
10	287.08	20	46	7.5		24	287.39	20	13	11
11	286.12	40	46	7.5		25	287.08	40	13	11
12	286.97	80	46	7.5		26	287.03	80	13	11
13	287.3	160	46	7.5		27	286.99	160	13	11
14	287.29	320	46	7.5		28	286.37	320	13	11

The experimental X-ray results used for figures 2-5 are presented in table 1. In base of table 1 and Figures 2-4, and results is the last square method computed obtained, the crystallographic parameter at 0 time of milling a_{00} and line slope b.

Results are in table 2.

Table 2. Crystallographic parameter a_{00} line slope b
of milling of iron powder with balls of different radii

No.	a_{00}, pm	b	Radius of balls, mm
1	286. 83	-0.00059	6.3
2	286.88	0.00133	7.5
3	286.28	0.00546	9.1
4	287.22	-0.00242	11

In during the milling time take place:

- the increasing density of dislocation and internal stress;
- the possibility of plastic deformation and o strong plastic distortion;
- the tendency to morphological and structural equilibrium [4].

The energy of impact corresponds in the some time for peak of pressure and an increasing of local temperature which may be approximate over the activation energy barrier.

During milling time a deformation of powders, a increasing of dislocation mobility and an increasing of temperature take place.

A ratio between mass balls and mass powders, of high value, reduces the mean size of impact balls, while a small ratio reduces the impacts frequently because unit efforts action at atomic level elongates atomic net, connection energy of iron atoms increases and arrives at a maxim value in case at broken particles that tension atomics network, energy of connection of iron atoms increasing and arriving at maxim value in case broken particles, consequent leads at increasing of distance between atoms like in figure 3, and in case of overlapping of some slipping plane decreasing their distance similar with the net stress as noticed in Figures 3, 4 and 5, where these parameters increase respectively figure 5 decrease with milling time.

Notice vial 1 has 32 balls, which is the mean number of balls having the approximately average radius. It is noticed in case of vial 2, which has smaller number of big balls and big mass is reduced is reduced:

- number of impacts and cold welding of particles as a results at plastic deformation;

- impacts between balls and particles produce a big kinetic energy, because of less welding which produce the less agglomerations.

In case of vial 4 which has 73 balls of smalls size and mass which produces a high number of impacts between balls, to be producing of big number of cold welding during its milling time [5].

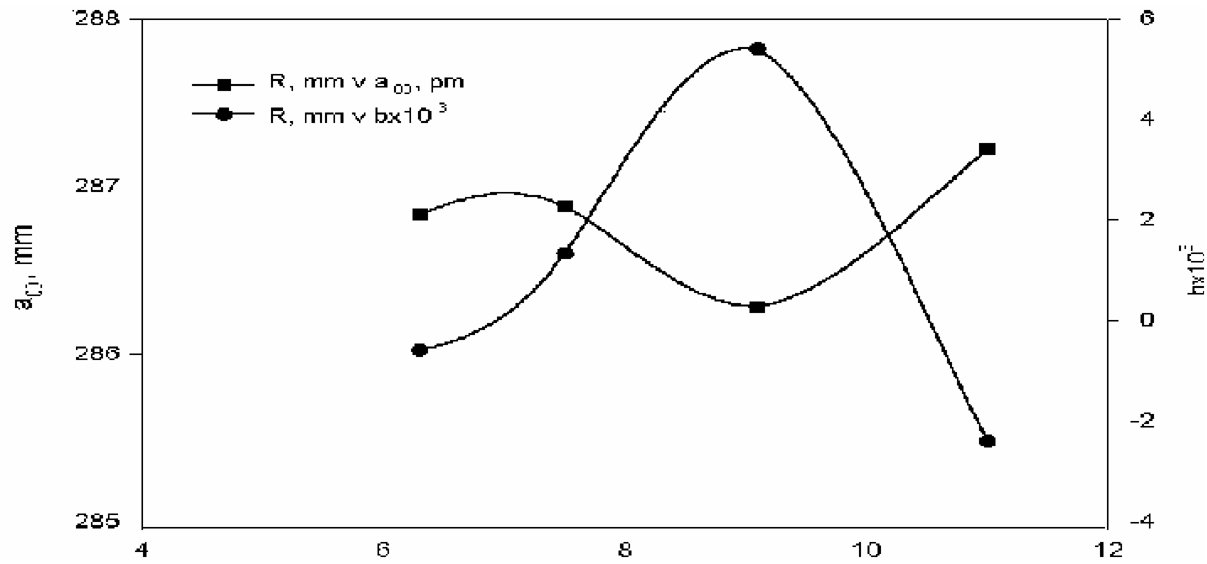

Fig. 6. Crystal parameter a_{00} at time 0 of milling results from the slope of straight line passed and b (slope of straight line) of iron powder milling with balls of different radius

It is noticed from table 2 and figure 6 that increasing of crystallographic parameter takes place only at radius at 7.5 mm and 9.1 mm, in interval from 7.5 to 9 mm.

4. Conclusions

Some final remarks can be makes:

- At balls with radius of 6.3 mm there is a tendency of easily decreasing of crystallographic parameter.
- At balls with radius of 7.5 mm there is a tendency of easily increasing of crystallographic parameter.
- At balls with radius of 9.1 mm there is a tendency of accentually increasing of crystallographic parameter.
- At balls with radius of 11 mm there is a tendency of accentually decreasing of crystallographic parameter.

- From this results is noticed that in the interval at 7.5 with 9 mm there a tendency of accentually increasing of crystallographic parameter.
- From experimental data someone may say that in that interval there is more efficiently milling from the evolution of crystallographic parameter.

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