

Mechanical Milling: Evolution of Crystal Parameter of Iron Powder

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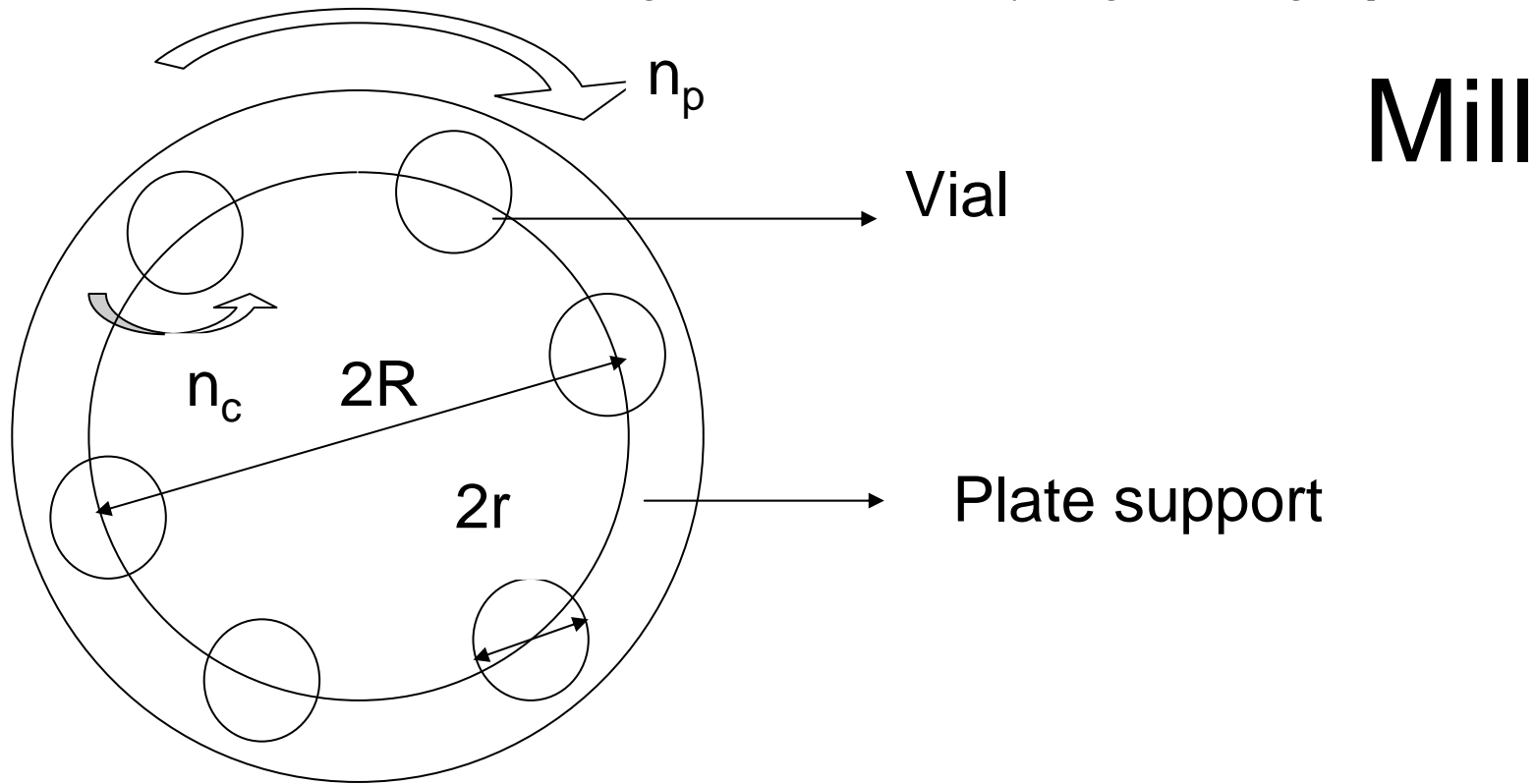
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Aim

- The scope of present research was to establish the efficiency of milling on iron powder, in a planetary mills varying the ball diameter.



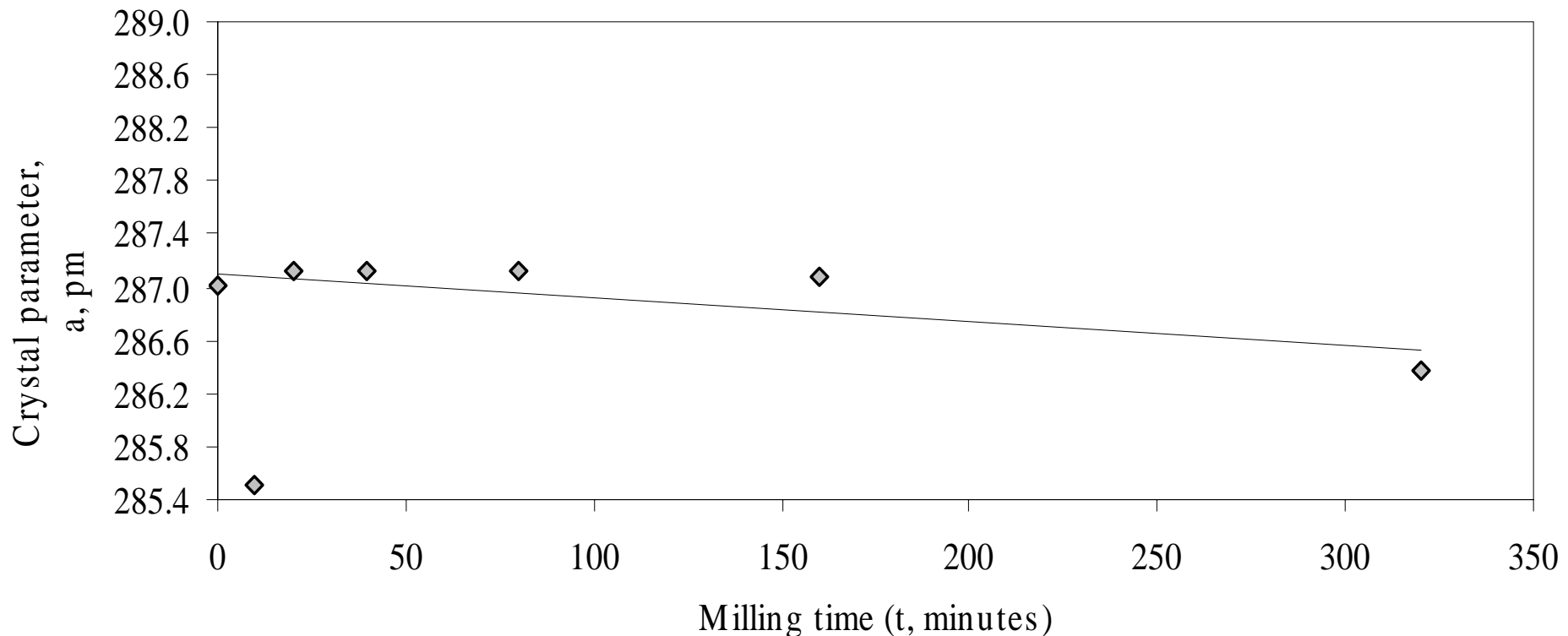
- The planetary mill has 6 vials, mounted on a plate support; velocity of rotation of plate support of planetary mill is $n_p = 1200$ rpm, velocity of rotation of vials is $n_c = -1.25 n_p$ and is produced by construction. Initial parameters are: radius of disk, R (250 mm), and radius of vials, r , vials have a mean weight of 1.750 kg approximately. Each vial contained a certain mass of balls: m_b with a diameter D .

The experiment

- The vials were of OLC 45 steel and the balls of RUL 2 steel.
- In vial number one there were introduced 32 balls with diameter $D = 18.6$ mm.
- In vial number two there were introduced 13 balls with diameter $D = 22$ mm, in vial number three there were introduced 13 balls with diameter $D = 22$ mm and in vial number four there were 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 $200 \mu\text{m}$. 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 warms balls, vials, and powder and some is producing disorder of structure [3, 4].
- The thermal effect gives an intensifying of diffusion. The increasing of density dislocations resulted of plastic deformations accelerates the process of volume diffusion in particle powder.

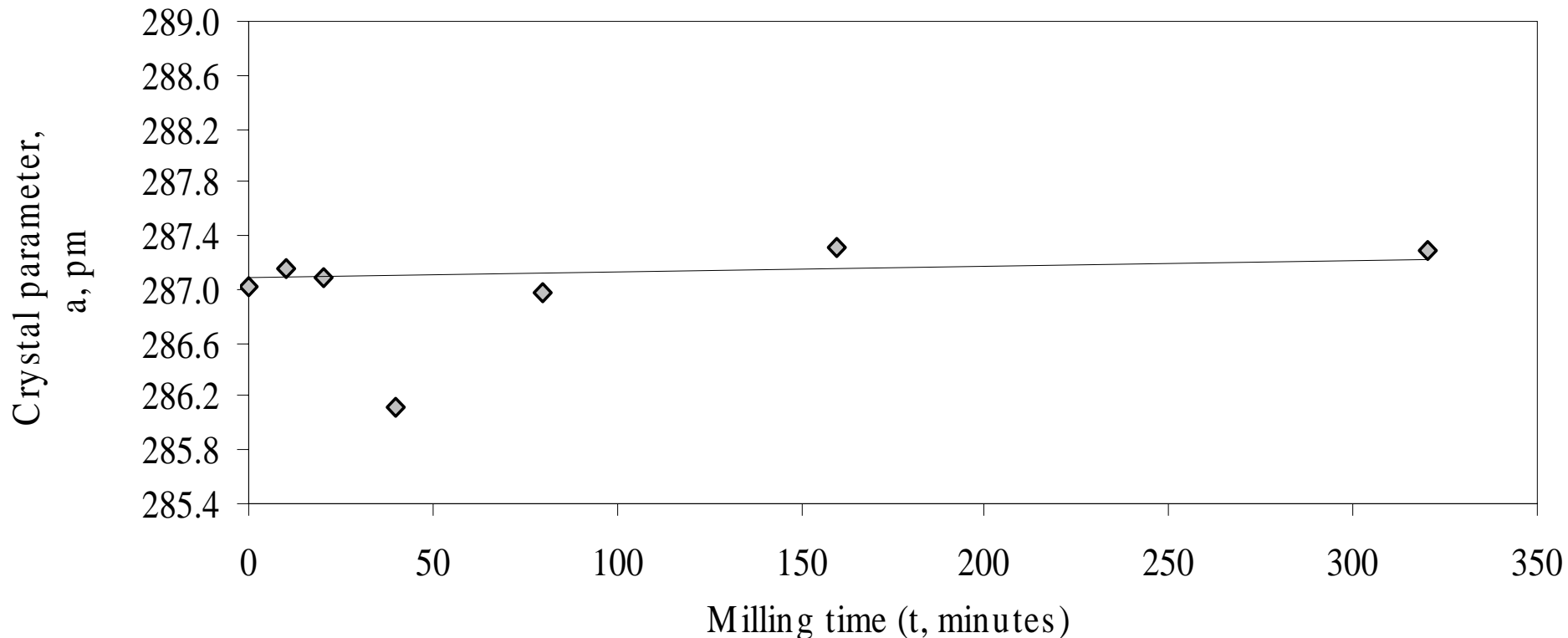
Results

- Crystallographic parameter of iron powder, vial 4, radius ($D/2 = 6.3$ mm) versus milling time
- The cut of experimental results line with abscissa gives $a_{00} = 286.8$ pm and the line slope $b = -0.0005$. It is noticed oscillations of values until 20 minutes of milling, after which there is a tendency of steadily decreasing of crystallographic parameter



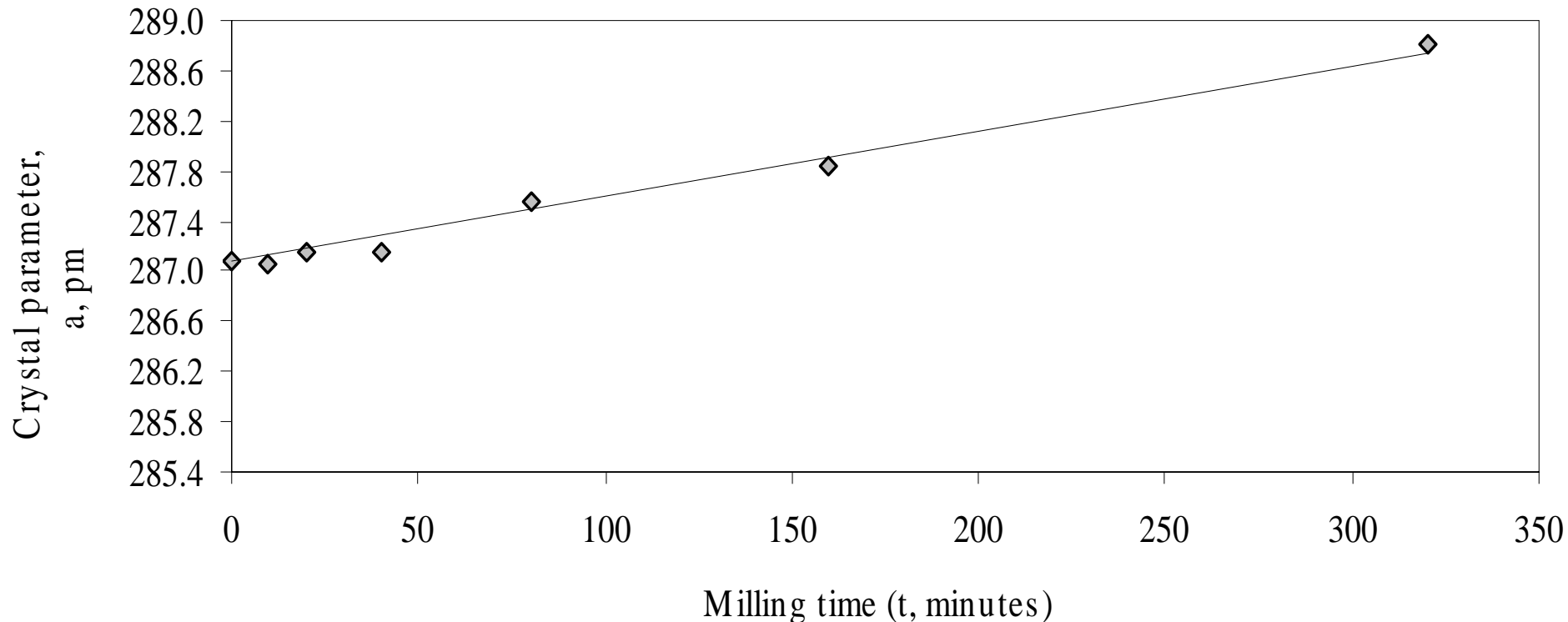
Results

- Crystallographic parameter of iron powder, Vial 3, radius ($D/2 = 7.5$ mm) versus milling time
- It is noticed an easily increasing of crystallographic parameter until 40 minute, after that an easily decreasing.
- $a_{00} = 286.9$ pm, $b = 0.0014$



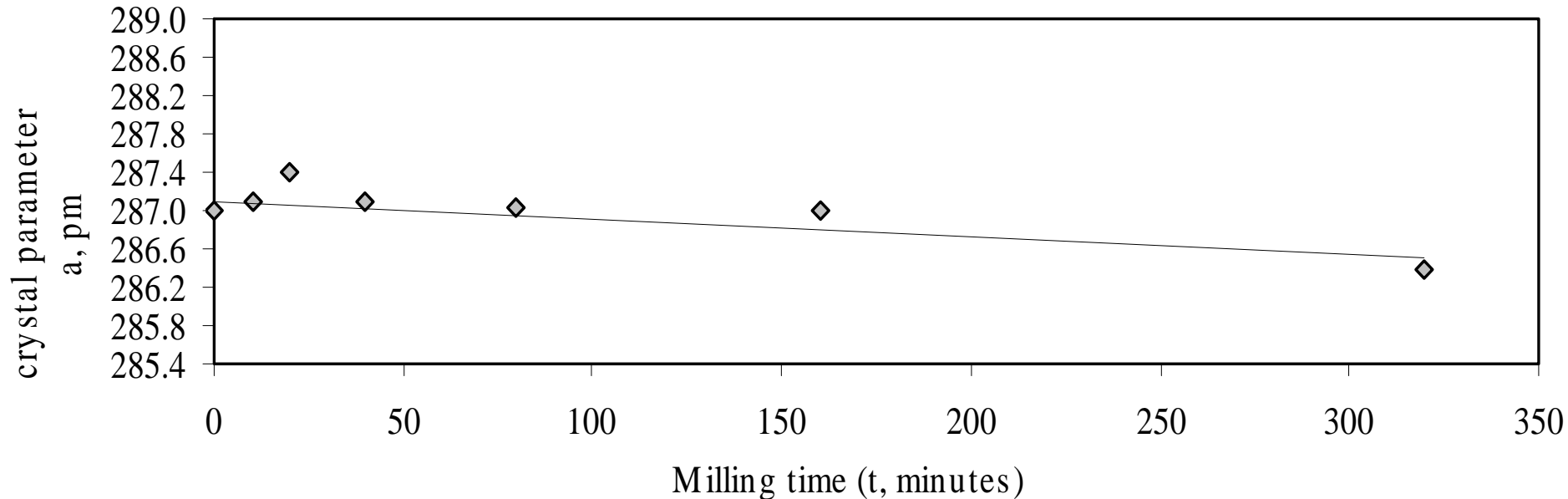
Results

- Crystallographic parameter of iron powder, vial 1, radius ($D/2 = 9.1$ mm) versus milling time
- It is noticed a tendency of linear increasing of crystal parameter with milling time.
- $a_{00} = 287.03$ pm, $b = 0.0055$



Results

- Crystallographic parameter of iron powder, vial 2, radius ($D/2 = 11$ mm) versus milling time
- It is noticed an easily increasing of crystallographic parameter until 20 minutes, after which an easily decreasing occur.
- $a = 287.2$, $b = -0.002$



Discussions

- The milled powder at mention time has been subjected to X-ray diffraction to obtain crystallographic parameters. It was applied the last square method in computing of a_{00} .
- A high value ratio between balls mass and powder mass reduces the frequency of impact balls, while a small ratio frequently reduces the impact, because unit efforts action at atomic level elongates atomic net, connection energy of iron atoms increases and arrives at a maxim value in case of broken particles that tension atomic network, consequently it leads at increasing of distance between atoms like in Vial 4, and in case of overlapping of some slipping plane decreasing their distance, similar to the net stress, where these parameters increase (Vial 1) or decrease (Vial 2) with milling time.

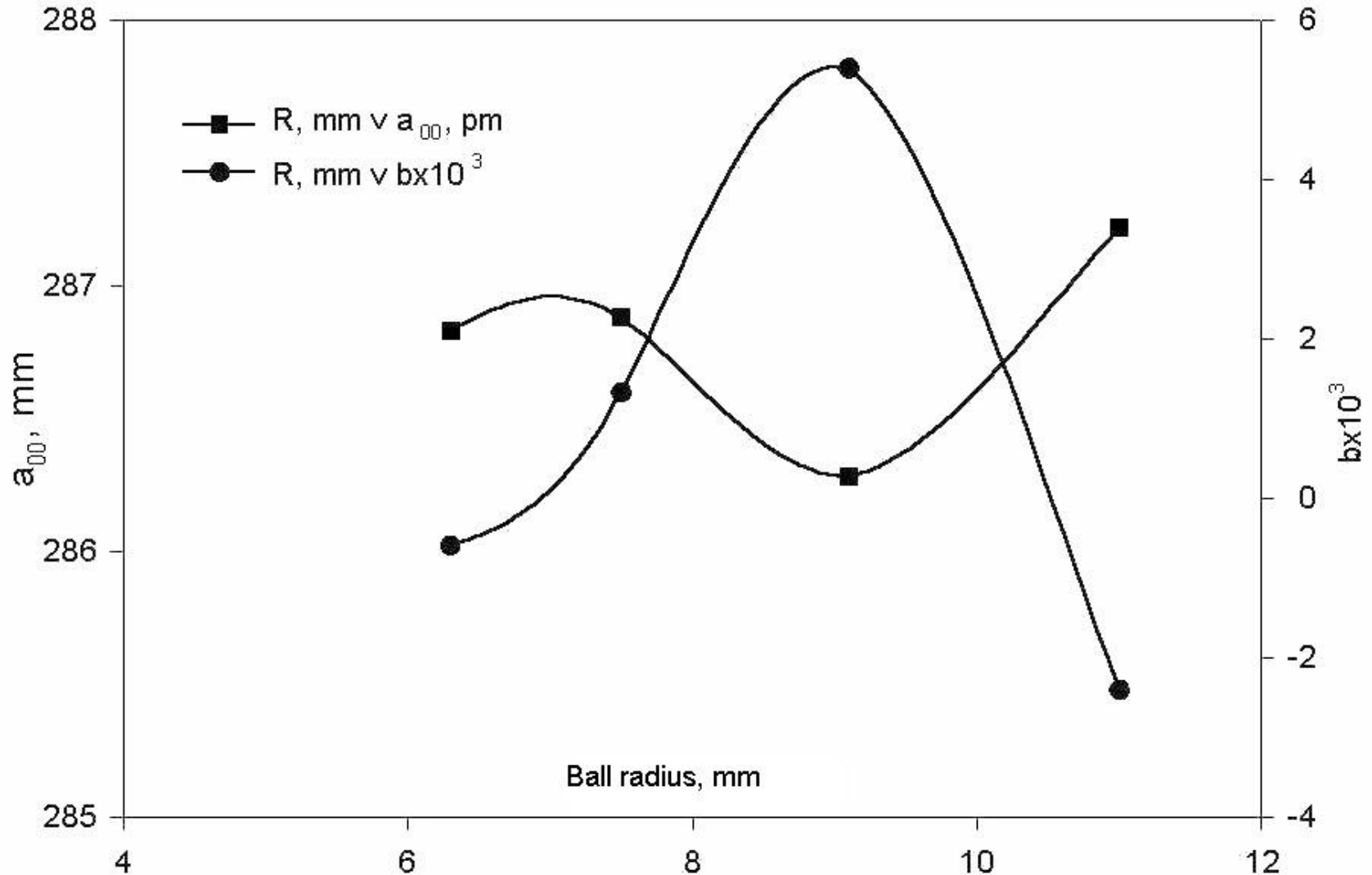
X-ray analysis results

Cr.No.	a_0 , pm	Milling time, minutes	Nr. of balls	D/2, mm
1	287.09	0	73	6.3
2	285.55	10	73	6.3
3	287.12	20	73	6.3
4	287.12	40	73	6.3
5	287.12	80	73	6.3
6	287.08	160	73	6.3
7	286.37	320	73	6.3
8	287.09	0	46	7.5
9	287.15	10	46	7.5
10	287.08	20	46	7.5
11	286.12	40	46	7.5
12	286.97	80	46	7.5
13	287.30	160	46	7.5
14	287.29	320	46	7.5

X-ray analysis results

Cr.No.	a_0 , pm	Milling time, minute	Nr. of balls	D/2, mm
15	287.09	0	32	9.1
16	287.06	10	32	9.1
17	287.15	20	32	9.1
18	287.16	40	32	9.1
19	287.56	80	32	9.1
20	287.84	160	32	9.1
21	288.80	320	32	9.1
22	287.09	0	13	11
23	287.10	10	13	11
24	287.39	20	13	11
25	287.08	40	13	11
26	287.03	80	13	11
27	286.99	160	13	11
28	286.37	320	13	11

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



Discussion

- It can be noticed, in case of Vial 2, which has smaller number of big balls and mass 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.
- Vial 4 has 73 balls of small size and mass, which produce a high number of impacts between balls, is producing of big number of cold welding during its milling time

Final remarks

- With balls with radius of 6.3 mm there is a small tendency of decreasing of crystallographic parameter from 287.1 pm to 286.5 pm during milling of 320 minutes.
- At balls with radius of 7.5 mm there is a small tendency of increasing of crystallographic parameter from 287.1 pm to 287.2 pm during milling of 320 minutes.
- At balls with radius of 9.1 mm there is a tendency of increasing of crystallographic parameter from 287.1 pm to 288.9 pm during milling of 320 minutes.
- At balls with radius of 11 mm there is a small tendency of decreasing of crystallographic parameter from 287.1 pm to 287.0 pm during milling of 320 minutes
- From this results, it is noticed that in the interval at $7.5 \div 9$ mm there a tendency of increasing of crystallographic parameter. From experimental data, it may said that this interval is more efficiently for milling, from the evolution of crystallographic parameter.