

Influence of mechanical milling conditions on the dispersity of lithium ferrite

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Abstract. The effect of mechanical milling in a planetary ball mills on dispersity of the synthesized lithium ferrite powder was investigated by laser diffraction technique. The mechanical milling of powder was carried out by two planetary ball mills: SPEX 8000M and Fritsch Pulverisette 5 with different time period. The lithium ferrite powder was milled for 30, 60 and 120 minutes in the first ball mill. The powder milled for 60 minutes in the second ball mill was performed. The results showed that an increase in the milling time slightly decreases the average particle size of the ferrite powder in case both ball mills. However, only milling in a SPEX planetary mill increases homogeneity of the ferrite powder, while the milling in Fritsch causes intensive processes of particle aggregation.

1. Introduction

Lithium ferrites have currently of great interest to researchers due to their technological applications as a low cost magnetic material for microwave techniques [1-5]. As was shown in [6-7], lithium ferrite is considered a promising cathode material due to its low environmental impact and lower cost compared to other commonly used materials such as Ni and Co. Also, LiFe_5O_8 can be used as an element of gas sensors because of high temperature and chemical stabilities [8].

The perfection of traditional methods of synthesis and the elaboration of new methods for lithium ferrites were implemented in order to improve the quality of production. In most practical applications, it is required to produce nanostructured LiFe_5O_8 of homogeneous composition [9-12].

Pre-milling operations in a planetary ball mill have become of great importance for production of ferrite materials by ceramic technology. This type of processing enables homogenization, milling and mechanical activation of the initial powder reactants. This leads to the stimulation of physico-chemical processes in powder materials [13-14].

In [15-19], it was shown that mechanical activation of the initial reactants allows great increase in its reactivity and enables to obtain lithium ferrites at significantly lower temperatures if compared to with those obtained by the conventional method.

In [20], inclusion of mechanical activation into the technological cycle enables to the produce of lithium ferrite ceramic with a higher density.

In the research, we studied the effect of the duration of mechanical activation in planetary ball mills of different types on the dispersion of synthesized lithium ferrite powders.

2. Experimental

Lithium ferrite samples were prepared by standard technology. Iron oxide and lithium carbonate were used as the initial reagents to produce lithium ferrite. The ratio of the initial components in the reaction



mixture was calculated by the equation: $\text{Li}_2\text{CO}_3 + 5\text{Fe}_2\text{O}_3 \rightarrow 2\text{LiFe}_5\text{O}_8 + \text{CO}_2\uparrow$. Before weighing, initial powders were dried for three hours at a temperature of 200°C in a drying oven. Synthesis of lithium ferrite was conducted at 800°C for 120 minutes in "HimLabo" standard laboratory oven with programmable thermostat "VARTA". The parameter of heating and cooling was $\sim 10^\circ\text{C}/\text{min}$.

The obtained powder was divided into two parts. The powder from the first part (sample A) was milled dry in SPEX 8000M planetary ball mill using tungsten carbide balls. The powder was consistently milled for 30, 60 and 120 minutes.

The milling of lithium ferrite from the second part (sample B) was performed for 60 minutes using Fritsch Pulverisette 5 ball mill with tungsten carbide balls.

For samples A and B, the particle size was analyzed by laser diffraction method using Fritsch Analysette 22 MicroTec Plus analyzer. Additionally for sample A, micrographs of non-milled and milled powders were obtained with Supra 50 VP scanning electron microscope (SEM).

3. Results and discussion

Figure 1 shows the SEM micrographs for a non-milled sample A (figure 1a), milled lithium ferrite 30 min (figure 1b), 60 min (figure 1c), and 120 min (figure 1d).

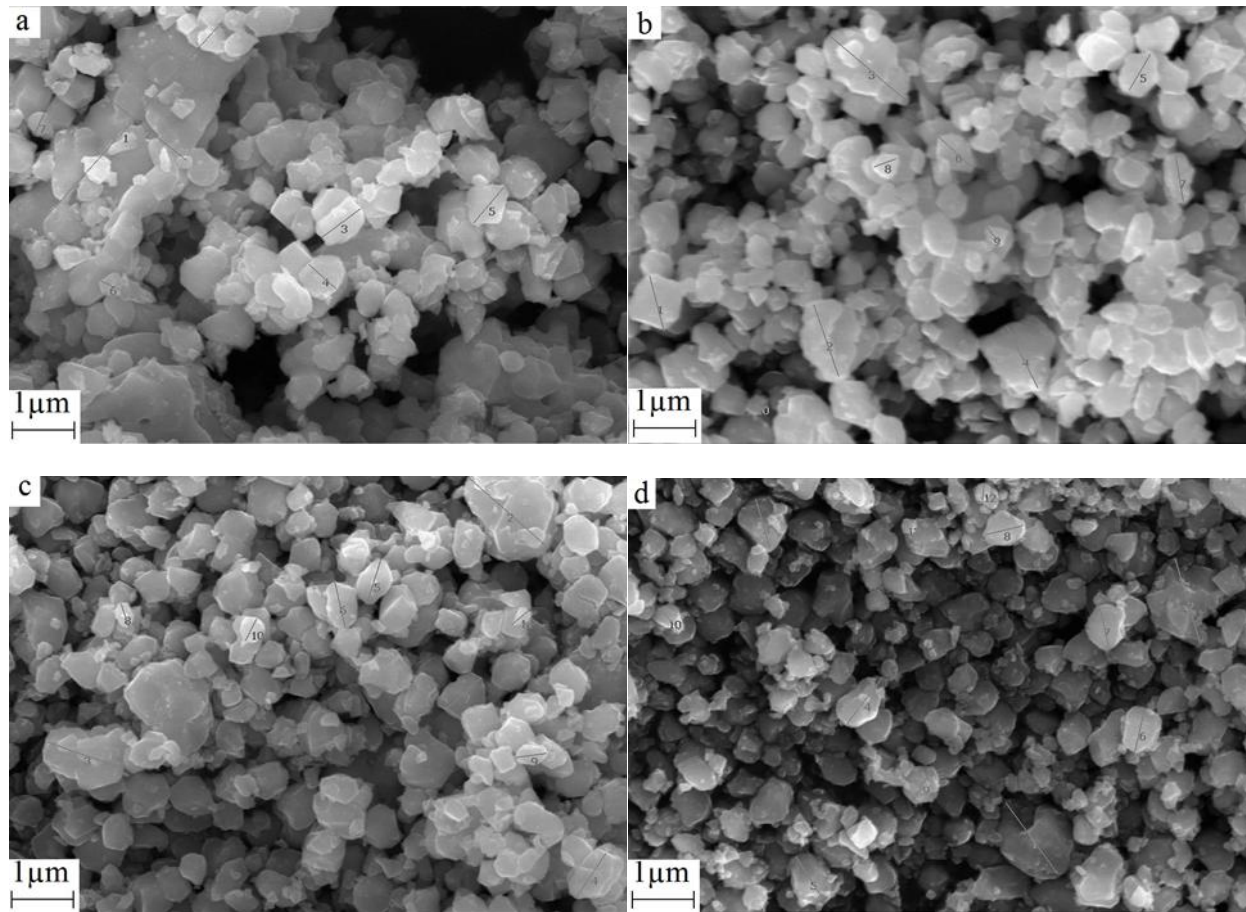


Figure 1. SEM micrographs for non-milled sample (a), milled for 30 min (b), 60 min (c), and 120 min (d).

The average particle size was 0.59 μm for a non-milled sample. After milling, the average particle sizes were 0.57 μm in milling for 30 min, 0.54 μm in milling for 60 min, 0.42 μm in milling for 120 min.

As can be seen in figure 1, mechanical activation of lithium ferrite powder leads to a slight reduction in particle size and higher powder homogenization. Thus, to obtain a homogeneous composition of the lithium ferrite powder, the time of mechanical treatment should be at least 2 hours.

The characteristics of particle size distributions by laser diffraction method are summarized in table 1. For the milled powders, a narrow monomodal particle size distribution are observed as a result of higher homogeneity of the ferrite powder. The results showed that as the duration of milling increases, the average particle size decreases.

Table 1. Particle size distribution for sample A.

Sample	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)
Initial sample	1.28±0.02	1.95±0.02	3.00±0.10
Milled for 30 min	1.13±0.09	1.80±0.06	2.88±0.04
Milled for 60 min	1.00±0.01	1.54±0.05	2.38±0.18
Milled for 120 min	1.90±0.05	1.32±0.02	1.91±0.03

The differential and integral particle size distribution for sample B is constructed and compared with the samples milled in a ball mill SPEX 8000M (figure 2).

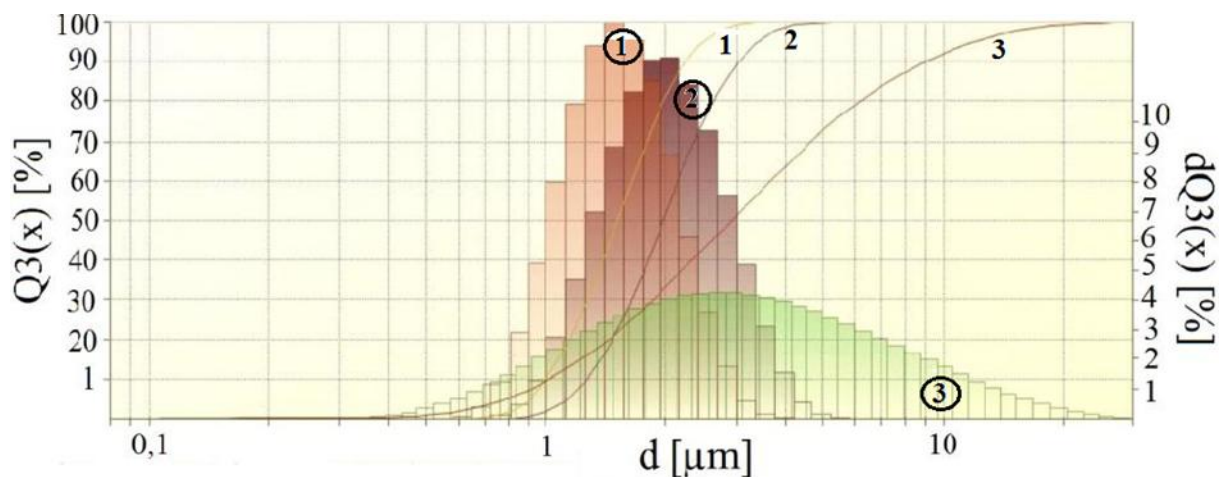


Figure 2. Differential and integral particle size distribution for samples B milled for 60 min: 1- initial sample, 2- milling in SPEX 8000M ball mill; 3- milling in Fritsch Pulverisette 5 ball mill.

The characteristics of the particle size distributions are shown in table 2. The data indicate, enhances both of the process and more intensive process of particle aggregation occur after powder milling in Fritsch Pulverisette 5 ball mill.

Table 2. Particle size distribution for sample B.

Sample	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)
Initial sample	1.28±0.02	1.95±0.02	3.00±0.10
After milling in Fritsch Pulverisette 5	1.13±0.09	1.80±0.06	2.88±0.04
After milling in SPEX 8000M	1.00±0.01	1.54±0.05	2.38±0.18

4. Conclusion

The results showed that mechanical activation of lithium ferrite powder in air at room temperature causes slight reduction of the particle size and increases the homogeneity of the ferrite powder.

In this case, the optimal conditions to obtain a homogeneous composition of the lithium ferrite powder are:

- mechanical activation in SPEX 8000M planetary ball mill;
- at least 2 hour of milling.

The obtained powders can be used for further sintering of ceramic from lithium ferrite.

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