Processing line for industrial radiation-thermal synthesis of doped lithium ferrite powders

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Abstract. The paper considers the issues of industrial production of doped lithium ferrite powders by radiation-thermal method. A technological scheme of the processing line is suggested. The radiation-thermal technological scheme enables production of powders with technical characteristics close to the required ones under relatively low temperature annealing conditions without intermediate mixing. The optimal conditions of the radiation-thermal synthesis are achieved isothermally under irradiation by the electron beam with energy of 2.5 MeV in the temperature range of 700–750 ^{0}C within~ 120 min.

Keywords: lithium ferrites, solid-state synthesis, electron beam, radiation-thermal annealing

1. Introduction

Modern technological electron accelerators (for example, ILU and ELV developed at BINP SB RAS, Novosibirsk [5–6]) are sufficiently small to be easily integrated in the production cycle. The design of these accelerators provides their continuous round-the-clock operation in the industrial production. In addition, electron beams provide broad options to control the radiation mode and do not induce radioactivity. They are cost-beneficial compared to other ionizing radiation sources, and provide high efficiency of conversion of the electric energy into the electron beam energy.

The studies which have been carried out in Russia showed that RT effects intensify a number of solid-phase reactions such as synthesis and sintering of some oxide compounds, Portland cement clinker, opening and processing of minerals, etc. [7]. RT proved to be effective for synthesis and sintering of ferrite ceramics [8–10]. In the latter case, the RT effect was characterized by increased speed of compact compression, particularly in the early stages of sintering. RT sintering reduced the processing time tenfold and enabled production of polycrystalline ferrites with a required combination of properties. The radiation was found to intensify the formation of the magnetic parameters for ferrite both in terms of time and maximum achievable values.

This suggests the existence of a number of physical effects which include the phenomenon of multiple acceleration of the diffusion mass transfer in crystalline and ceramic materials under excitation by power beams of charged particles and homogenization of heterogeneous structures in radiation fields. The phenomenon of radiation intensification of sintering of ferrite powder systems under the combined action of high temperatures and intense fluxes of accelerated electrons was unambiguously identified.

This paper addresses the problems to be solved for industrial production of doped lithium ferrite powders via radiation-thermal method and suggests a technological scheme of the production line.

2. Experimental techniques

Initial technical requirements claimed in the technological scheme design are as followers:

1) The heater for RT-annealing of reaction mixtures is pulsed electron accelerator ILU-6.

2) The homogeneity of the temperature field on the irradiated substrate with samples is not less than 3%.

3) The temperature-time mode of irradiation provides the content of the basic phase in the synthesized product not less than 70%.

The electron accelerator ILU-6 with sufficiently high parameters and relatively small dimensions has been chosen as a heat source. The electron beam is released into the air through the foil, and it can provide the power density of up to 400–500 W/cm^2 to allow heating of the process material up to ~2000 ^{0}C. A protected area with internal dimensions of 3×4×5 m^3 is sufficient to install the accelerator.

The maximum energy of accelerated electrons was E = 2.5 MeV, pulse current was 400 mA, pulse duration was 500 μs, the pulse repetition rate varied from 4.5 to 11.5 Hz, and the rate of compact heating was 130 ^{0}C/min. The durations of the nonisothermal annealing stages (heating and cooling) in the radiation-thermal synthesis were ~ 3 min.

Irradiation was carried out in air in the heat insulating cell [11] of lightweight chamotte with a mass thickness of the horizontal plates of ~ 0.16 g/cm^2. The electron energy loss in the plates of that thickness did not exceed 8% and could be neglected. The annealed samples inside the cell were placed on a thin plate of lightweight chamotte 3 mm thick located on ceramic tubes. To reduce the temperature gradient, the cell was covered with a radiation transparent cover 0.1 g/cm^2 thick. The temperature was monitored by the platinum/platinum-rhodium (90% of platinum, 10% of rhodium) thermocouple with the measuring junction in the test sample located in the immediate vicinity of the annealed compacts. To suppress the interference generated by the injected charge, a platinum earthing electrode was welded to the measuring junction. The platinum/ platinum-rhodium thermocouple was chosen due to its chemical resistance to most substances in the corrosive ionized medium at high temperatures, ease of producing sufficiently pure measuring junction, flexibility and a wide range of operating temperatures [12]. The thermocouple ceramic tube was located in the shadow of the metal radiation shield to improve the accuracy of the measured temperature. Mixture heating and maintenance of the given temperature were carried out without external heat sources due to the energy of decelerated electrons. The relation of the electron extrapolated paths to the sample mass thickness ensured the minimum temperature gradients on the pellet.

3. Result and discussion

The experimental data obtained in [11, 13] was used as the basis for development of the technological scheme of radiation-thermal synthesis of lithium ferrites (Fig. 1).

The working chamber is a container of lightweight chamotte with a compacted reaction mixture inside. The containers are placed on the remotely operated conveyor system "loading-unloading" opposite the accelerator outlet port. The measurements obtained with a system of thermocouple sensors showed that the required temperature uniformity in the range of 600–800 ^{0}C can be reached if the irradiated surface is located at a distance of 16 cm from the accelerator outlet port and the accelerator operates in the nominal mode with the electron beam scanning. In this case, the size of the irradiated area is 6×30 cm^2. In the area of the reaction mixture preparation, mixing and briquetting of the initial reagents is carried out under the pressure of 200 MPa. The compact thickness is to be about two-thirds of the extrapolated electron path for uniform depth heating of the compact.

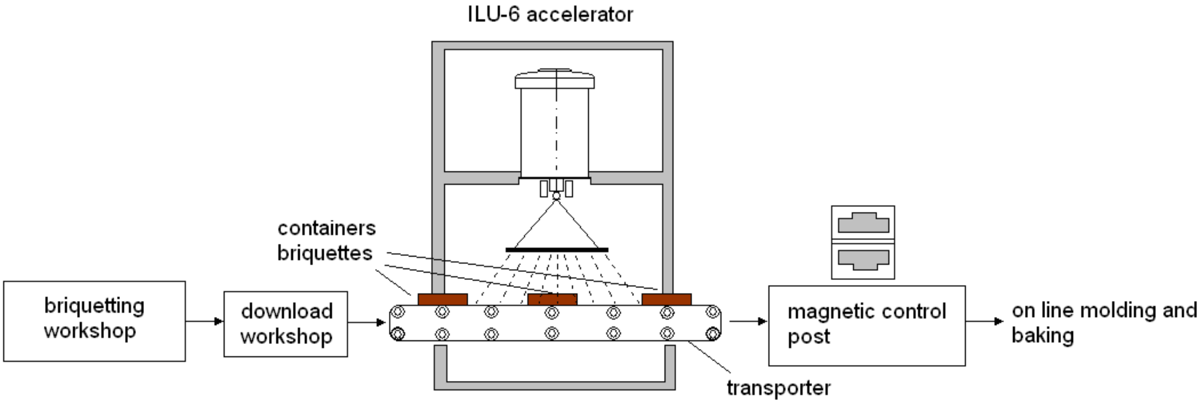

Fig. 1. Technological scheme of radiation-thermal synthesis of ferrite powders

The calculation shows that if the electron energy is 2.4–2.5 MeV, the optimum thickness of the compacted mixture is 3 mm. Under these parameters, a single container loading is ~55 cm^3. The mixture weight is ~170 g for the compact density of ~ 3 g/cm^3.

The proposed scheme was used to perform synthesis of a batch of lithium substituted samples of all compositions in a single container within 120 minutes at isothermal excerpt temperatures of 600 ^{0}C, 700 ^{0}C and 750 ^{0}C [14].

The comparison of sample batches shows that the developed scheme of radiation-thermal technology provides the production of powders with specifications close to the required ones at relatively low temperature conditions of annealing without intermediate mixing.

4. Conclusions
A technological scheme for radiation-thermal synthesis of substituted lithium ferrites is proposed. It includes heating of the compacted reaction mixtures by intense electron beam with energy of 2.5 MeV without external heat sources. The optimum mode of radiation-thermal synthesis is achieved through isothermal excerpt under irradiation in the temperature range of 700–750 ^{0}C within ~ 120 min.

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