

BRIEF COMMUNICATIONS

FORMATION OF MICROSTRUCTURE OF LITHIUM-TITANIUM FERRITE DURING ITS SYNTHESIS IN A 2.4 MeV ELECTRON BEAM

A. P. Surzhikov,¹ E. V. Nikolaev,¹ E. N. Lysenko,¹
S. A. Nikolaeva,¹ D. Zh. Karabekova,² and A. S. Ghyngazov³

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INTRODUCTION

Ferrite materials are the key elements of most modern radio-engineering, electronic and computational devices. [1, 2]. In particular, lithium ferrites are extensively applied in microwave devices and as cathode materials for lithium cells [3, 4]. Lithium-substituted ferrites, where Fe^{3+} ions are substituted for titanium ions are characterized by low dielectric loss values and high temperature stability and are therefore widely applied in microwave engineering [5–7].

Today, ceramic technology of ferrite production is quite common, including the synthesis of ferrite powders at high temperatures. Given the fact that ferrites generally have complex compositions, their synthesis involves the formation of transient products of solid-phase interaction including monoferrite phases and solid oxide solutions. Such solid-phase interactions occur in a diffuse regime and therefore for a complete synthesis reaction require an inclusion of multiple grinding and compaction procedures into the technological process, followed by many-hour annealing at high temperatures. It is for this reason that the ceramic technology of ferrite manufacturing belongs to multi-operation and power-consuming processes and does not yield a large percentage of useful products. Furthermore, using this technology it is impossible to manufacture ultrafinegrained ferrite powders because of high synthesis temperatures.

Within recent time, new methods of synthesizing ferrite materials have been extensively used, which rely on heating the reagents in a high-energy electron beam (1–10 MeV) [8–10]. In our works [11, 12] we showed that using this heating approach it is possible to accelerate solid-phase interaction in the reaction mixture by a few factors and considerably reduce the synthesis temperature and time. The ferrites manufactured via the electron-beam heating are characterized by high values of residual magnetization, saturation magnetization, electrical resistivity and Curie temperature.

It is well known that the properties of ferrites are largely determined by their microstructure. Therefore it is critical to know how heating of the initial mixture in the beam of high-energy electrons would affect the formation of the resulting ferrite microstructure. The use of electron microscopy for examination of microstructure of lithium ferrites is limited due to their high specific resistivity. Charging of the surface prevents from obtaining high-resolution images. The atomic-force microscopy (AFM) is void of this disadvantage. The purpose of this study is using the AFM method

¹National Research Tomsk Polytechnic University, Tomsk, Russia, e-mail: surzhikov@tpu.ru; nikolaev0712@tpu.ru; sal17@tpu.ru; lysenkoen@tpu.ru; ²Buketov Karaganda State University, Karaganda, Republic of Kazakhstan, e-mail: karabekova71@mail.ru; ³AO Sibkabel, Tomsk, Russia, e-mail: ghyngazov@mail.ru. Translated from *Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika*, No. 5, pp. 164–166, May, 2020. Original article submitted March 5, 2020.

to investigate the microstructure of lithium ferrite produced by heating in a beam of electrons with an energy of 2.4 MeV.

EXPERIMENTAL PROCEDURE

The experimental material was lithium-titanium ferrite $\text{Li}_{0.6}\text{Fe}_{2.2}\text{Ti}_{0.2}\text{O}_4$, which belongs to the main group of materials of advanced microwave engineering. The initial reagents were commercial powders Fe_2O_3 (AR), Li_2CO_3 (CP), TiO_2 (AR). The initial mixture of these powders in the prescribed proportion was subjected to milling in an AGO-2c planetary ball mill for 60 min at room temperature. The regime of maximum power level of the mill of 60g was used, for which the drum rotary velocity in the translator motion was found to be 2200 rev/min. The powder compacts were fabricated by the single-side cold-die compression in the shape of tablets.

Lithium ferrite was synthesized by the electron-beam heating of the powder compacts in an ILU-6 accelerator (Budker Nuclear Physics Institute, SB RAS), Novosibirsk) at the electron energy of 2.4 MeV [13]. The synthesis method is detailed elsewhere [11]. The synthesis reaction was performed at a temperature of 750°C for 60 min. These parameters are much lower than those used in the traditional synthesis in an oven (1000–1100°C, more than 10 h) [14].

An examination of a specimen in an atomic-force microscope involves progressive probing of the specimen surface area with a sharp tip. The surface is scanned with a piezo-scanner moving the specimen precisely with respect to the sharp tip. The results of measurement of the probe displacement with respect to the specimen are recorded by the software modeling the specimen surface relief.

The surface microstructure of the synthesized ferrites was investigated using a Solver P47 atomic-force microscope. The microrelief of the synthesized ferrite was examined by the AFM method in a tapping mode. The ferrite tablet was fixed on the specimen stage using double-sided adhesive tape. The surface was analyzed using the HA_HR probes of the ETALON series (NT-MDT, Russia). It should be underlined that no specific artefacts, such as sticking of the probe or anomalous generation, were observed during scanning.

RESULTS AND DISCUSSION

Figure 1 presents three- and one-dimensional images of surface microrelief of lithium-titanium ferrite specimen synthesized by the method of electron-beam heating.

An analysis of the $\text{Li}_{0.6}\text{Fe}_{2.2}\text{Ti}_{0.2}\text{O}_4$ ferrite structure images presented in Fig. 1 demonstrated that the average ferrite particle size ~ 250 nm. The relief variation occurs within the range ~ 100 –600 nm. The measurement error was found to be 5 nm. According to the data presented elsewhere [11, 12], the average particle size of lithium ferrite during high-temperature synthesis is 1–2 μm .

Summing up, the AFM results imply that the particle size of the synthesized ferrite is by a factor of 4–8 smaller than that of ferrite manufactured by the traditional high-temperature synthesis. This can account for the high values of specific electrical resistivity and residual magnetization of ferrites synthesized by the method of electron-beam heating.

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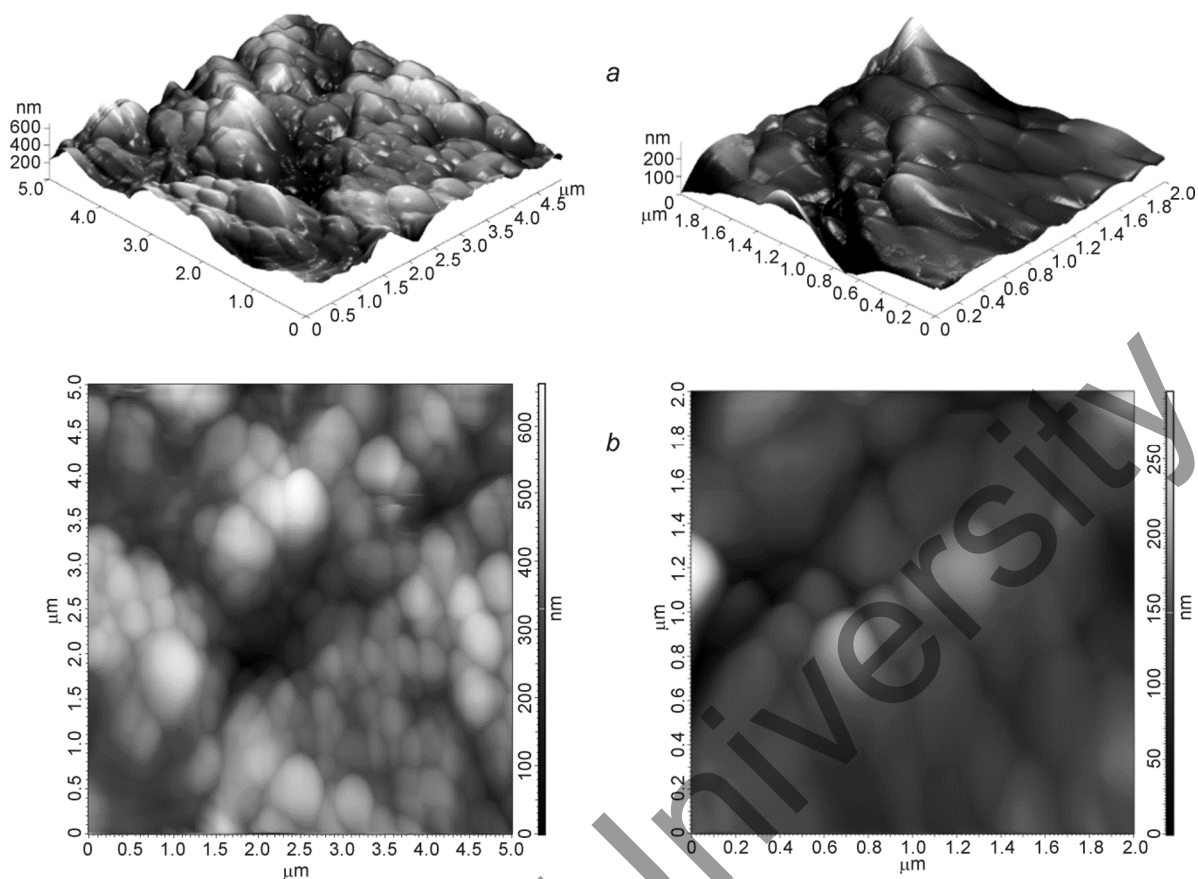


Fig. 1. Structure images of the $\text{Li}_{0.6}\text{Fe}_{2.2}\text{Ti}_{0.2}\text{O}_4$ -ferrite obtained by the AFM method: *a* – 3D-image of the surface relief, *b* – surface micrograph.

REFERENCES

1. V. A. Zhuravlev, V. I. Itin, R. V. Minin, *et al.*, Russ. Phys. J., No. 11, **60**, 1946–1954 (2018); <https://doi.org/10.1007/s11182-018-1307-8>.
2. V. A. Zhuravlev, A. V. Zhuravlev, V. V. Atamasov, and G. I. Malenko, Russ. Phys. J., No. 10, **62**, 1926–1936 (2020); <https://doi.org/10.1007/s11182-020-01924-9>.
3. K. Mahmood and M. A. Khan, Physica B, **567**, 45–50 (2019); <https://doi.org/10.1016/j.physb.2019.05.014>.
4. S. A. Mazen and N. I. Abu-Elsaad, Appl. Nanosci., **5**, 105–114 (2015); <https://doi.org/10.1007/s13204-014-0297-2>.
5. Y. Gao, Z. Wang, R. Shi, *et al.*, J. Alloy. Compd., **805**, 934–941 (2019); <https://doi.org/10.1016/j.jallcom.2019.07.173>.
6. M. Kavanlooe, B. Hashemi, H. Maleki-Ghaleh, and J. Kavanlooe, J. Electron. Mater., **41**, 3062–3066 (2012); <https://doi.org/10.1007/s11664-012-2235-y>.
7. Q. Yin, Y. Liu, Q. Liu, *et al.*, J. Mater. Sci.: Mater. Electron., **30**, 5430–5437 (2019); <https://doi.org/10.1007/s10854-019-00836-w>.
8. U. V. Ancharova, M. A. Mikhailenko, B. P. Tolochko, *et al.*, IOP Conf. Ser.: Mater. Sci. Eng., **81**, Article No. 012122 (2015).

9. V. G. Kostishin, V. G. Andreev, V. V. Korovushkin, *et al.*, *Inorgan. Mater.*, **50**, 1317 (2014).
10. V. A. Zhuravlev, E. P. Naiden, R. V. Minin, *et al.*, *IOP Conf. Series: Mater. Sc. Eng.*, **81**, Article No. 012003 (2015).
11. E. N. Lysenko, A. P. Surzhikov, V. A. Vlasov, *et al.*, *Nucl. Instrum. Methods Phys. Res. B*, **392**, 1–7 (2017).
12. A. P. Surzhikov and A. M. Pritulov, *Radiation-Thermal Sintering of Ferrite Ceramics* [in Russian], Energoatomizdat, Moscow (1998).
13. V. L. Auslender, *J. Nucl. Instrum. Methods Phys. Res. B*, **89**, 46–48 (1994).
14. M. S. Ruiz and S. E. Jacobo, *Physica B*, **407**, 3274–3277 (2012).

Buketov University