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LASER ANNEALING OF OXIDE FILMS ON THE SAPPHIRE SURFACE

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Abstract

We investigate experimentally laser annealing of oxide films on the sapphire surface at the LIMO 100-532/1064-U facility. The treatment is performed using a Nd:YAG solid-state laser of a fixed wavelength of 1,064 nm. We present the results of morphology studies of iron-oxide film surfaces by atomic force microscopy. We obtain the dependence of the film-surface peak height on the laser irradiation power. We show that a laser irradiation power of the order of 90 W is sufficient for the formation of crystal grains and transition to the polycrystalline structure of the film. We show that, depending on the laser-irradiation parameters, the film-surface quality can be refined due to the recrystallization of amorphous layers.

Keywords: sapphire crystals, atomic force microscopy, laser treatment.

1. Introduction

An efficient method of modifying the surface of films is laser treatment (annealing). The use of laser annealing enables creating polycrystalline films promising for applications in thin-film transistors, liquid-crystal displays, sensors and solar cells due to the recrystallization of their amorphous structure. Besides, laser irradiation makes it possible to modify the electrophysical and optical characteristics of films, and their crystalline and defect structures. Laser irradiation ensures the highest energy density on the sprayed surface as compared to the existing methods of producing films (vacuum methods, chemical methods, combined methods). Laser annealing enables producing low-dimensional multicomponent homostructures and heterostructures [1, 2].

In this connection, an important task of this work is to study the effect of laser annealing on the morphology of films on the sapphire surface. Sapphire supports possess a set of certain physical properties (high melting temperature, chemical and radiation stability, high hardness and transparency), owing to which they find wide application in microelectronics, quantum electronics, high-resolution optics, and nanotechnologies [3–5]. The use of such materials has sharply increased in virtually all branches of production.

2. Experiment

We use a 3% solution of iron chloride $FeCl_3$ to develop a film of Fe_2O_3 on a sapphire support. We apply an iron-chloride solution under laboratory conditions with a doctor blade [6] through a screen printing mask, which is an adhesive tape (for instance, a polyimide film or Scotch tape) 40–50 μm thick, as shown in Fig. 1.

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Using the technique of applying an iron-chloride solution [6] shown in Fig. 1, we produced Fe_2O_3 films 0.25 cm^2 (0.5×0.5 cm) in area on the surface of a sapphire support 1×2 cm in size. After application, the films were kept for several minutes at room temperature before the screen printing masks were removed. Then they were dried on a Saturn ST-EC1161 electric oven at 120–150°C for 10–15 min and sintered on a LIMO 100-512/1064 laser facility (the Scientific Educational Center "Laser Technologies" of the Southern Federal University) by a diode-pumped pulsed solid-state Nd:YAG laser with a wavelength of 1,064 nm (pulse duration 80 ns, pulse recurrence frequency 10 kHz, maximum pulse energy 110 mJ, average power 110 W) [7–12].

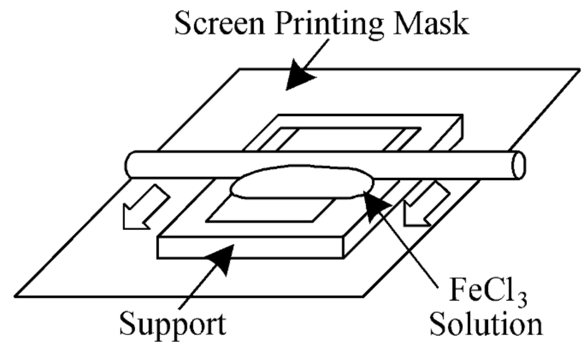

Fig. 1. Application of an iron–chloride solution to a sapphire support with a doctor blade.

In the process of annealing Fe_2O_3 films, the laser beam was aimed directly at the film applied on the sapphire support, which was placed onto the work table of the laser facility. Thus, the process of forming a Fe_2O_3 film using laser irradiation can be represented by the scheme in Fig. 2.

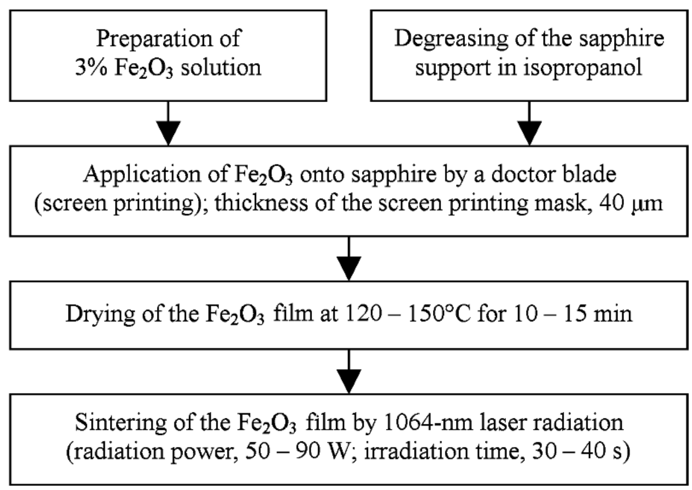

Fig. 2. Scheme of the Fe_2O_3 film formation process.

The universal laser facility LIMO 100-512/1064 shown in Fig. 3 consists of a Nd:YAG laser (wavelength 1,064 nm), a galvanometric two-coordinate mirror scanner, an optical system including a homogenizer to form the spatial profile of the laser beam and to achieve uniform illumination of the support, the focusing system (F-Theta lens), the control computer, and a movable work table (object table), which displaces the support in the vertical direction with respect to the position of the laser beam's focusing region. Using the computer, the main working parameters of the laser are controlled (power and pulse recurrence frequency), and the scanner is operating (the spatial position of the laser spot is determined, as well as the scanning rate and the interval between the lines along which the laser beam moves).

The surface of the Fe_2O_3 film was studied by atomic force microscopy (AFM) at the NTEGRA Vita probe nanolaboratory of the Common Use Center "Nanotechnologies" (Southern Federal University). The produced AFM images were processed using the Image Analysis 3.5 software package. The wide distribution of the AFM method for studies of various objects is due to the fact that, unlike other methods of (light and electron) microscopy, it does not require long, complex, and expensive preparation of specimens [13–15].

Figure 4 presents AFM images of the iron-oxide film surface at a laser irradiation power of 50–90 W. The AFM measurements showed the specimens to have polycrystalline (Fig. 4 a, b) or amorphous (Fig. 4 c–f) structures. The polycrystalline (grainy) structure is formed upon treatment of the film by laser irradiation of 90 W. In Fig. 4 b, the grain diameter is 654±187 nm, surface roughness 87 nm, and peak height 776 nm. Upon treatment of the film by laser irradiation of 50–70 W, the film preserves its

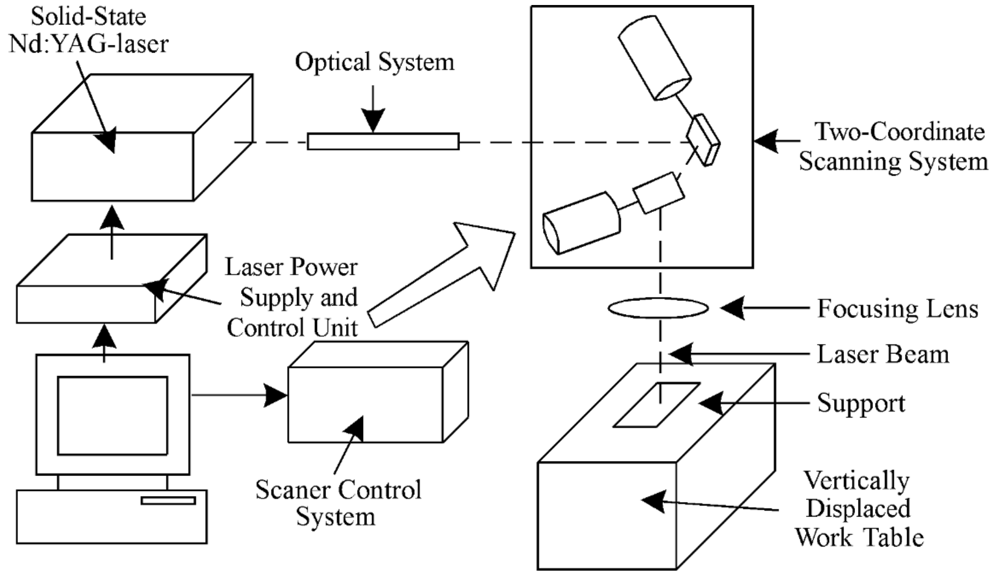

Fig. 3. Scheme of the laser sintering of a Fe_2O_3 film (wavelength 1,064 nm) at a LIMO 100-512/1064 laser facility.

amorphous structure. Thus, a laser irradiation power of the order of 90 W is sufficient for formation of crystal grains and transition to the polycrystalline structure, which refines the quality of the film surface and makes it possible to modify its electrophysical and optical characteristics.

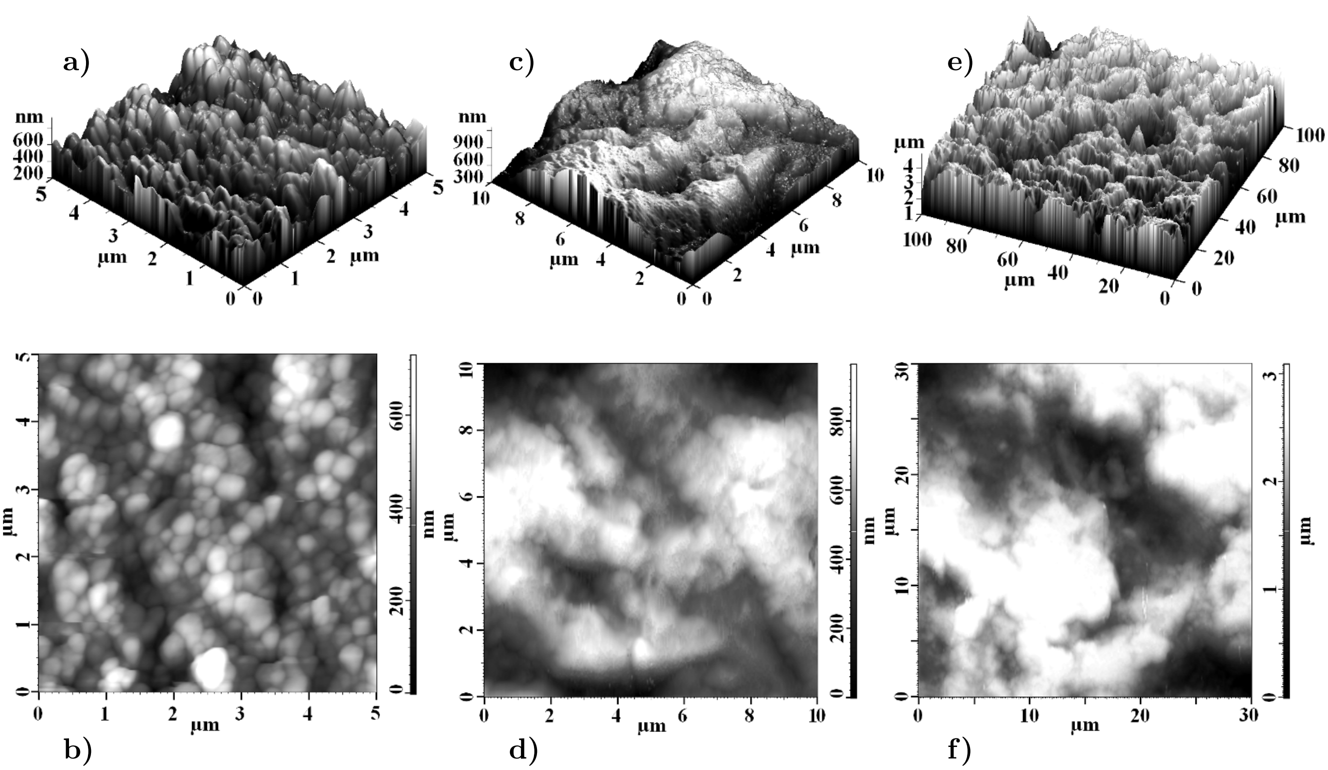

Fig. 4. Atomic force microscopy images (3D and 2D) of a Fe_2O_3 film at a laser irradiation power of 90 W (a, b), 70 W (c, d), and 50 W (e, f).

Figure 5 presents the dependence of the effect of laser irradiation power on the height of Fe_2O_3 film surface peaks.

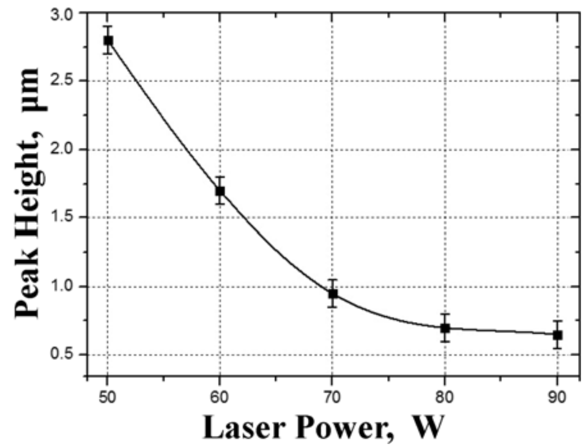

Fig. 5. Effect of the laser irradiation power on the height of film surface peaks.

The AFM phase image (Fig. 6) shows the distribution of the relative value of the cantilever phase failure in the scanning field coinciding with the topographic AFM image. An important use of the AFM phase technique is the possibility of identifying chemical compounds on the surface; also, it enables expanding the possibilities of visualizing surface defects.

From the analysis of the phase-contrast AFM image, it follows that the iron oxide film treated by 90 W laser irradiation has a homogeneous and grainy structure. The film texture depends on the texture of the support (which is cubic), but data on the crystallographic structure of the formed film would have to be obtained in further work. The same algorithm can be used to produce and investigate various oxide films on sapphire or glass supports, in particular, titanium dioxide films, manganese oxide films, etc.

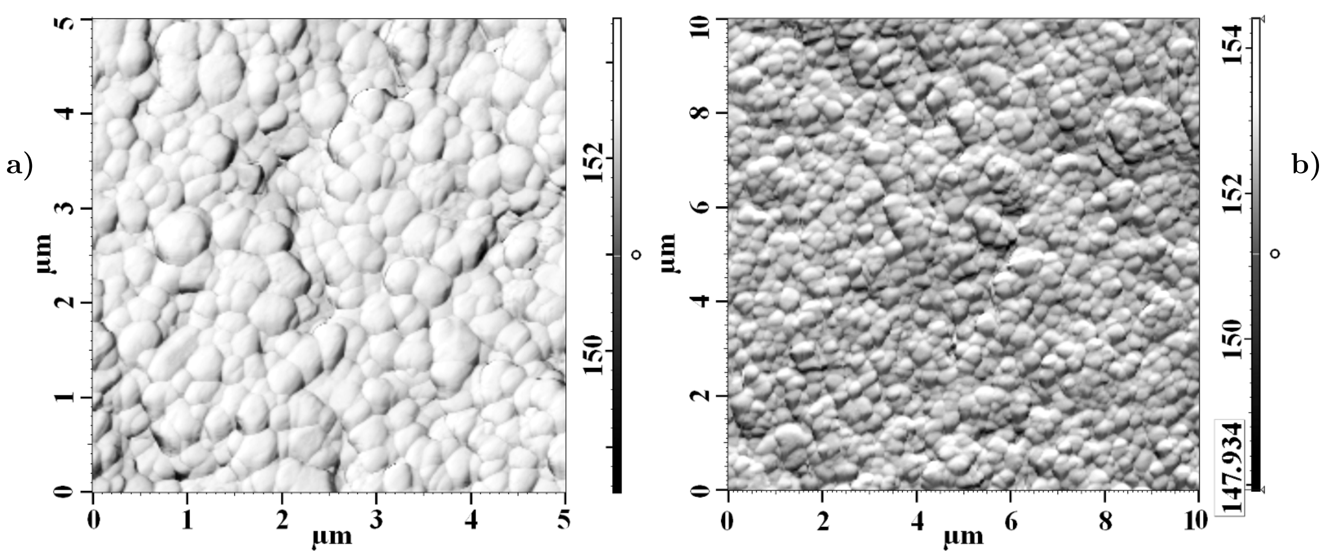

Fig. 6. Phase contrast for a specimen treated at a laser irradiation power of 90 W.

3. Results

After conducting the experiments, we conclude that

1. The action of laser irradiation on the structure of a Fe_2O_3 film enables modification of its crystal and defect structures.

2. Depending on the laser irradiation parameters, the quality of the film surface can be refined due to

the recrystallization of amorphous layers. In particular, laser irradiation of the order of 90 W leads to phase transition of the film from an amorphous to a polycrystalline state.

3. Laser annealing of films on the sapphire surface is of undoubted applied interest, as it is a method of creating microstructures of a characteristic size, which require further detailed investigation of their properties and prospects of use in thin-film transistors, displays, sensors, and solar elements.

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References

1. R. V. Konakova, A. F. Kolomys, O. B. Okhrimenko, et al., *Semiconductors*, **48**, 621 (2014).
2. V. V. Voronov, S. I. Dolgayev, and G. A. Shafeyev, *Dokl. Akad. Nauk*, **58**, 465 (1998).
3. D. I. Cherednichenko, S. P. Malyukov, and Yu. V. Klunnikova, “Heat-physical processes at the sapphire crystals growth by horizontal directed crystallization,” in: I. Tartaglia (Ed.), *Sapphire: Structure, Technology and Applications*, Nova Science, New York (2013), p. 101.
4. S. P. Malyukov and Yu. V. Klunnikova, “Physical and technological fundamentals of sapphire production for electronics,” in: I. A. Parinov (Ed.), *Nano- and Piezoelectric Technologies, Materials, and Devices*, Nova Science, New York (2013), p. 133.
5. S. P. Malyukov and Yu. V. Klunnikova, “Complex investigations of sapphire crystals production,” in: S.-H. Chang, I. A. Parinov, and V. Yu. Topolov (Eds.), *Advanced Materials*, *Springer Proceedings in Physics* (2014), Vol. 152, p. 55.
6. G. P. Smestad, *Sol. Energy Mater. Sol. Cells*, **55**, 157 (1998).
7. S. P. Malyukov and A. V. Sayenko, *J. Russ. Laser Res.*, **34**, 531 (2013).
8. Yu. V. Klunnikova, S. P. Malyukov, and A. V. Sayenko, *Izv. Saint-Petersburg Electrotechnical University (LÉTI)*, **8**, 15 (2014).
9. Yu. V. Klunnikova, S. P. Malyukov, and A. V. Sayenko, *Izv. South Federal University, Tekh. Nauki*, **9**, 39 (2014).
10. M. N. Libenson, E. B. Yakovlev, and G. D. Shandybina, *Interaction of Laser Radiation with Matter (Power Optics)*, Saint-Petersburg State University of Information Technologies, Mechanics, and Optics (2008) [in Russian].
11. A. S. Kashin and V. P. Ananikov, *Dokl. Akad. Nauk, Ser. Khim.*, **12**, 2551 (2011).
12. V. V. Voronov, G. A. Shafeev, and A. M. Prokhorov, “Laser-assisted micro-fabrication of sapphire,” in: I. Tartaglia (Ed.), *Sapphire: Structure, Technology, and Applications*, Nova Science, New York (2013), p. 74.
13. P. E. West, *Introduction to Atomic Force Microscopy: Theory, Practice, Applications*, Pacific Nanotechnology, Santa Clara, CA (2007), p. 68.
14. A. P. Dostanko, O. A. Ageev, D. A. Golosov, et al., *Semiconductors*, **48**, 1242 (2014).
15. O. A. Ageev, E. Y. Gusev, E. G. Zamburg, et al., *Appl. Mech. Mater.*, **475–476**, 1266 (2014).