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НОВЫЕ ВОЗМОЖНОСТИ ПРИМЕНЕНИЯ МЕТОДА ЛА-ИСП-МС ДЛЯ ИССЛЕДОВАНИЯ СОСТАВА И ГОМОГЕННОСТИ ПОРОШКОВЫХ ПРОБ НА ОСНОВЕ ТАНТАЛА

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В статье рассмотрены возможности и особенности метода масс-спектрометрии с индуктивно связанной плазмой и лазерной абляцией (ЛА-ИСП-МС), применяемого для исследования распределения легирующей примеси и определения состава порошкообразных образцов прекурсоров Ta_2O_5 и шихты $LiTaO_3$, допированных редкоземельными элементами (TR = Pr, Nd, Sm, Eu, Gd, Dy). Для определения оптимальных параметров и настройки масс-спектрометра, лазерного испарителя и получения градуировочных характеристик использованы стандартное стекло SRM NIST 612, таблетированный образец апатитового концентрата 2462-82 и высокочистый аргон. Установлено, что при применении таблетированного образца 2462-82 можно получить более высокие пределы обнаружения, поэтому он был использован для анализа проб твердых прекурсоров $Ta_2O_5\langle TR\rangle$ и шихты $LiTaO_3\langle TR\rangle$ методом ЛА-ИСП-МС. Сканирование поверхности образцов осуществлялось лазерным пучком различного диаметра от 20 до 350 мкм с мощностью лазерного излучения более 1 Вт, плотностью энергии лазера $\geq$ 15 Дж/см^2, достаточной для эффективного испарения таблетированной пробы. Мощность лазерного импульса составляла 40-80% от возможной величины (1 Вт), частота следования импульсов – 5-8 Гц. На примере образцов $Ta_2O_5\langle Sm\rangle$ и $LiTaO_3\langle Sm\rangle$ показано, что содержание Sm, определенное методом ЛА-МС-ИСП, соответствовало заданному и имеет высокую прецизионность с рентгенофлуоресцентным методом. Равномерное распределение легирующей примеси TR в Ta_2O_5 и $LiTaO_3$ подтверждено найденными значениями относительного стандартного отклонения интенсивности аналитического сигнала Sr, которое для порошковых образцов $Ta_2O_5\langle TR\rangle$ составило 2-6, для шихты $LiTaO_3\langle TR\rangle$ – 4-7. Показано, что матричный эффект отсутствует в результатах анализа порошкообразных образцов методом ЛА-ИСП-МС; образцы $Ta_2O_5\langle TR\rangle$, изготовленные без связующего, могут быть использованы для количественного анализа шихты $LiTaO_3\langle TR\rangle$. Полученные результаты имеют важное значение в разработке эффективной технологии синтеза легированной редкоземельными элементами шихты танталата лития, используемой для получения однородных монокристаллов $LiTaO_3\langle TR\rangle$ высокого оптического качества. Новые возможности предложенного метода исследования могут быть использованы для других порошковых материалов, таблетированных без связующего компонента.

Ключевые слова: лазерная абляция, масс-спектрометрия с индуктивно связанной плазмой, пентаоксид тантала, танталат лития, легирование, редкоземельные элементы

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NEW OPPORTUNITIES OF THE LA-ICP-MS APPLICATION FOR RESEARCH OF THE COMPOUND AND HOMOGENEITY OF TANTALUM BASED POWDER SAMPLES

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The article discusses the capabilities and features of the method of mass spectrometry with inductively coupled plasma and laser ablation (LA-ICP-MS), which is used to study the distribution of the dopant and determine the composition of powder samples of Ta_2O_5 precursors and a charge $LiTaO_3$ doped with rare earth elements (TR = Pr, Nd, Sm, Eu, Gd, Dy). To determine the optimal parameters and adjust the mass spectrometer, laser evaporator and obtain calibration characteristics, we used standard SRM NIST 612 glass, a tableted sample of apatite concentrate 2462-82, and high-purity argon. It has been established that when using a tableted sample 2462-82, higher detection limits can be obtained, therefore it was used for the analysis of samples of solid precursors $Ta_2O_5\langle TR\rangle$ and a charge $LiTaO_3\langle TR\rangle$ by the LA-ICP-MS method. The surface of the samples was scanned with a laser beam of various diameters from 20 to 350 μm with a laser radiation power of more than 1 W, a laser energy density of ≥ 15 J/cm^2, sufficient for efficient evaporation of a pelleted sample. The laser pulse power was 40-80% of the possible value (1 W), the pulse repetition rate was 5-8 Hz. Using the $Ta_2O_5\langle Sm\rangle$ and $LiTaO_3\langle Sm\rangle$ samples as examples, it has been shown that the Sm content determined by the LA-ICP-MS method corresponded to the specified one and has a high precision with the X-ray fluorescence method. The uniform distribution of the TR dopant in Ta_2O_5 and $LiTaO_3$ is confirmed by the found values of the relative standard deviation of the intensity of the analytical signal Sr, which was 2-6 for the $Ta_2O_5\langle TR\rangle$ powder samples, and 4-7 for the $LiTaO_3\langle TR\rangle$ charge. It is shown that the matrix effect is absent in the results of the analysis of powder samples by the LA-ICP-MS method; $Ta_2O_5\langle TR\rangle$ samples prepared without a binder can be used for quantitative analysis of the $LiTaO_3\langle TR\rangle$ charge. The results obtained are important in the development of an effective technology for the synthesis of a charge of lithium tantalate doped with rare-earth elements, which is used to obtain homogeneous $LiTaO_3\langle TR\rangle$ single crystals of high optical quality. New possibilities of the proposed research method can be used for other powder materials tableted without a binder.

Key words: laser ablation, inductively coupled plasma mass spectrometry, tantalum pentoxide, lithium tantalite, doping, rare earth elements

INTRODUCTION

Creation of optical materials technology based on optically nonlinear crystals doped by rare-earth elements is a task of great practical importance. Single crystal lithium tantalate is of the most perspective ferroelectric materials. It arises such scientific interest because it combines unique electrooptical, optically nonlinear and laser properties [1-3]. It is a well known fact that doping with lithium tantalate changes the optical and electro-physical properties of single crystals. The research of lithium niobate single crystals [4] revealed the following: not only concentration and type of dopant influence crystals characteristics, but also doping method. Today the problem stays unsolved for lithium tantalate crystals. The method of lithium tantalate crystals doping by rare-earth elements is described in detail in [5]. The research highlights importance of chemical uniformity at a distribution of the dopant, starting from making of a precursor.

The dopant concentration in Ta_2O_5 and $LiTaO_3$ is usually detected in their acid solutions [6]. This analysis technique is able to obtain results for a usual sample amount 50-100 mg, i.e. a macro-volume. However, control of distribution of a single component in a micro-volume sample (less than 0.1 mg) is still an unsolved problem. This is the reason why it should be actual.

Today the amount of laboratories equipped by laser ablation inductively coupled plasma mass spectrometers (LA-ICP-MS method) rises. LA-ICP-MS is able to obtain instrumental response along the depth profile (up to 0.2-0.3 μm during one laser impulse) and on the flat sample surface at the analysis of a sample dry aerosol, evaporated by energy of a laser beam 20-350 μm in diameter. A structure of a solid sample, chemical compound, surface condition and the laser ablation parameters are crucial at laser evaporating of a sample. The parameters are radiation power, duty cycle and a laser beam diameter [7]. The research [7] concluded that laser energy parameters, repetition speed and the crater size bounded by an aperture determine the speed of the material evaporation. A change of the power by an energy attenuator controls the laser energy. A change in a repetition frequency programmed by software influences the speed of the evaporation. The frequency less 10 Hz is used for “soft” materials, such as powders, to provide optimal intensity of the evaporated material flux. For example, glasses or geological samples can be analyzed with the frequency 10 Hz. Authors of [7] emphasize that the repetition frequency should be adjusted for each matrix type. The authors of [8] used the laser ablation method to create textures on the surface of polytetrafluoroethylene. The works [9-11] describe the possibilities of using LA-ICP-MS for the analysis of powdered samples of tablet form with and without a binder.

Authors [9, 12] stress that effects of an element fractioning have a significant impact on instrumental response of the device at the stage of melting, evaporating, a sample transport to the plasma torch of mass spectrometer. In the works [13-18] confirm the previous conclusions for research of such transparent materials as calcite, silicate glass etc.

LA-ICP-MS is sensitive towards compound and micro-homogeneity of a standard sample. Such sample could be used for setting and optimizing mass spectrometer operating parameters and for obtaining of calibration curves. Authors of [5, 19-20] solved the problem of sample choice to calibrate laser evaporator and mass spectrometer parameters. The authors revealed that a tableted sample made of a geological standard (for example, an apatite concentrate) is suitable for these purposes.

Thus a complex approach to the laser evaporation parameters choice must take into account the requirements of the analytical problems to be solved and particularities of an object of study. This is necessary to achieve the best accuracy of analysis and the lowest detection limits. The experience in use of LA-ICP-MS gives an ambiguous choice of conditions for research of compound and chemical homogeneity of samples. This fact determines actuality of solving the problem of choice of standard and reference samples for the LA-ICP-MS analysis. This problem has been little studied for powdered tableted samples.

This paper tests the possibility to use an LA-ICP-MS in a completely new application – research of a micro-homogeneity of a sample with a simultaneous detection of a dopant concentration.

MATERIALS AND METHODS

Concentrations of analytes were detected by a mass-spectrometer method (ELAN-9000 DRC-e, Perkin Elmer, USA). A sample material was taken locally from the surface by a laser sampler UP-266 MACRO (New Wave Research, USA), using a laser ablation process. Laser radiation used in experiments was Nd:YAG ($\lambda 0$ = 266 nm). Determination of optimal conditions for a standard silicate sample (glass) SRM NIST 612 [21] sampling was necessary to provide stability of instrumental response of an interrelated complex that includes a laser evaporator and a mass-spectrometer. Al and Si were chosen as analytes for LA-ICP-MS parameters optimization. Al and Si concentration in SRM NIST 612 were 33.6% and 1.1%, respectively. Laser beam diameters were chosen to be 20-350 μm among all possible values. Use of bigger diameters is undesirable because drift of intensities appears. Tableted samples $Ta_2O_5\langle TR\rangle$ и $LiTaO_3\langle TR\rangle$ (TR = Pr, Nd, Sm, Eu, Gd, Dy**)** without the binder 12 mm in diameter were obtained under identical and repeatable conditions by pressing in a metal shell under pressure ~106 Pa. The powder was not mixed or rubbed before tableting. A mass-spectrometer parameters were the following: Neb. Gas Flow = 0.08 – 1.00 l/min, ICP RF Power = 1250.00 – 1350.0 W, Analog Stage Voltage = -1800.00 – -1900.00 V, Pulse Stage Voltage = 850.00 – 950.00 V. High-purity argon (not less than 99,995 wt. %) was used as a blank. A mode “jump on peaks” was used during analysis automatically, which means that method file contained: 3 jumps on the peak, 3 scans on the peak, 3 replicas of mass spectrum scanning. A microscope Leica DM2500 was used to measure LA craters diameter and evaluate their shape.

RESULTS AND DISCUSSION

We have detected a ration between physical parameters of LA and their influence on intensity of the instrumental response (i.e a number of electrical impulses registered by a detector per second). The instrumental response intensity was determined to be connected with a laser beam diameter, laser impulses frequency and laser radiation power. A change in a laser impulses frequency fails to influence energy of laser beam that reaches the sample. Whereas an increase in a laser beam diameter does increase this parameter. Laser radiation energy follows a saturation curve at a power increase to 100%. Energy that reaches the sample increases with an increase in laser beam diameter in both cases. Such interrelation reflects technical parameters of UP-266 MACRO. Obtained dependences helped to choose a laser beam diameter and power necessary to supply a sample by enough energy at analyses.

We adjusted the device using LA attachment both at mass-spectrometry analysis and analysis of solutions. We have detected that functional dependence of the ion lens voltage on the analyte mass is free of fundamental differences from the same dependence obtained at use of calibration solutions. The differences are absent at adjusting of resolving power (mass scale) in LA-ICP-MS and ICP-MS experiments.

During adjusting of parameters of mass-spectrometer and laser evaporator, and obtaining of calibration characteristics we have replaced the standard glass SRM NIST 612 with a tableted sample from a powder geological standard sample of apatite concentrate 2462-82 (composition and determination error of analytes are presented in Table 1). An adjustment procedure was carried out according to the geological standard according to the scheme recommended by the manufacturer. Use of tableted sample enables achieving of higher detection limits [19]. We assume that this is a result of more active emission of material from powder samples, but the reasons are a matter of future study.

The final stage of routine daily adjustment of the mass spectrometer tuning is determination of the LenzDac parameter, which links the pulse and analog segments of the detector to the mass of isotopes selected for tuning. Adjustment characteristics were similar to the ones obtained at adjustment using SRM NIST 612.

Table 1

Composition of geological standard sample of apatite concentrate 2462-82, certified by Institute of mineralogy, geochemistry and crystal chemistry of rare elements (Moscow, Russia)

Таблица 1. **Состав геологической эталонной пробы апатитового концентрата 2462-82, аттестованной Институтом минералогии, геохимии и кристаллохимии редких элементов (Москва, Россия)**

The component	Weight fraction of component. %	An absolute measurement error (%) with a 0.95 confidence interval
SiO_2	2.07	0.15
TiO_2	0.44	0.01
CaO	50.66	0.28
MgO	0.081	0.012
P_2O_5	39.27	0.27
MnO	0.039	0.004
Na_2O	0.43	0.04
K_2O	0.18	0.02
SrO	2.61	0.18
Fe	0.46	0.02
F	3.14	0.04
ΣTR_2O_3	0.83	0.04
La_2O_3	0.24	0.03
CeO_2	0.36	0.03
Pr_2O_3	0.040	0.006
Nd_2O_3	0.14	0.01
Sm_2O_3	0.021	0.004
Eu_2O_3	0.0058	0.0011
Gd_2O_3	0.022	0.006
Tb_2O_3	0.0018	0.0006
Dy_2O_3	0.0078	0.0017
Yb_2O_3	0.0014	0.0004
Y_2O_3	0.040	0.005

A laser beam of different diameters scanned surfaces of tableted samples. Diameter was from 20 to 350 μm, laser radiation power was upper than 1 W, laser energy density ≥ 15 J/cm^2. Such density is enough for effective evaporation of a tableted sample. Pulse power was 40-80% of a possible value (1 W), pulses frequency was 5-8 Hz.

The sample evaporates under the laser beam. Power, energy and the laser beam spot area are determined by LA processes parameters. The choice of parameters influences quality, repeatability of the shape and volume of the crater; thus it influences the amount of material evaporated during one pulse. This determines intensity of the instrumental response, thus limits of elements detection. It is important to ensure that consecutive pulses form strictly cylindrical craters of the exact diameter. The beam shape can be other than a circle, but it is crucial that the shape and proportion should stay the same. Otherwise reproducible results and calibration dependencies are impossible to achieve because amount of evaporated during one sampling act material will considerably vary.

In the process of examining samples $Ta_2O_5\langle TR\rangle$ и $LiTaO_3\langle TR\rangle$ laser ablation is most effective in comparison with other methods of their decomposition (acidic, melting followed by leaching, autoclave). Standard samples of similar composition necessary for obtaining of calibration curves are absent. This is the main problem of LA-ICP-MS analysis of solid precursors $Ta_2O_5\langle TR\rangle$ and $LiTaO_3\langle TR\rangle$ charge. In order to solve this problem we considered a possibility to use tableted samples of an apatite concentrate 2462-82 with certified values (wt.%): Pr_2O_3 = 0.040, Nd_2O_3 = 0.14, Sm_2O_3 = 0.021, Eu_2O_3 = 0.0058, Gd_2O_3 = 0.022, Dy_2O_3 = 0.0078. Besides, study of micro-homogeneity and composition of samples can be carried out by scanning of the sample surface with a laser beam or a layer-by-layer analysis during laser sampling in one point. Such analysis is carried out firs of all to detect a dopant concentration. The method minimizes experiment method due to absence of decomposition or dissolving stage. The stage provides possibility to loose or add something. Most of spectral poly-atomic overlap is excluded when liquid phase is absent from the sample flow in a plasma torch of a mass-spectrometer.

Unlike analysis of tantalum-containing samples solutions, at use of LA-ICP-MS for analysis of solid tableted samples of precursor and charge the matrix effect was absent, or at least we did not detect it. At analysis of tableted tantalum pentoxide sample containing 1 wt. % TR this ratio never exceeded 40, even at use of 100% of laser power. Thus we have detected that the tantalum amount exceeding TR amount did not suppress instrumental response of TR. The absence of the matrix effect at is another advantage of LA-ICP-MS analysis of tableted samples.

The approached were identical at analyses of precursors $Ta_2O_5\langle TR\rangle$ and $LiTaO_3\langle TR\rangle$ charge. We have demonstrated them on the examples containing Sm as a dopant. Fig. 1 demonstrates craters after the LA of a tableted sample of an apatite concentrate 2462-82 and $Ta_2O_5\langle Sm\rangle$ (1.0 wt. %) at an increase in the pulse amount from 10 to 1000.

We found that an increase in the pulse amount does not lead to a distortion of cylindrical shape of craters and their diameters. Measurements on a microscope Leica DM2500 P has shown that craters have average diameters 105±2.97 μm (relative standard deviation 1.72). Use of 100% power of laser radiation leads to over-heating of a sample's upper layers, spreading, cracking and crumbling of material which leads to an increase in dispersity of reproducibility of results.

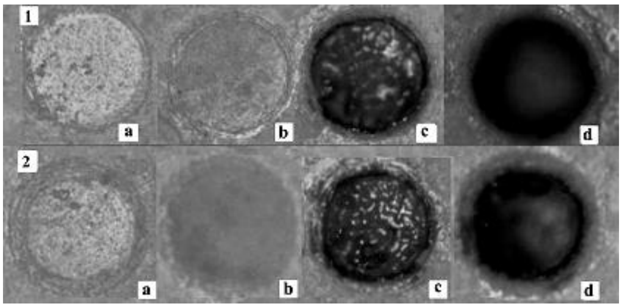

Fig. 1. Craters after laser ablation of tableted samples: 1 – apatite concentrate 2462-82, 2 – $Ta_2O_5\langle Sm\rangle$ (1.0 wt. %); a – a single impulse, b – 10, c – 50, d – 1000 pulses. Power and frequency of impulses were 60% and 5 Hz, laser beam diameter was 100 μm

Рис. 1. Кратеры после лазерной абляции таблетированных образцов: 1) ГСО 2462-82 (апатитовый концентрат), 2) $Ta_2O_5\langle Sm\rangle$ (1,0 мас.%); a – единичный импульс, b – 10, c – 50, d – 1000 импульсов. Мощность и частота следования импульсов, соответственно, 60% и 5 Гц, диаметр лазерного пучка 100 мкм

A dependence on Fig. 2 was obtained due to the use of tableted apatite concentrate 2462-82 for drawing of a calibrating characteristics (a number of measurements n = 4) during registration of a mass-spectrum in the mode "jumping from peak to peak" (3 jumps on the peak, 3 scans on the peak, 3 replicas of mass spectrum scanning). A quantitative LA-ICP-MS analysis of a tantalum pentoxide doped by samarium sample was carried out using a calibration dependence on Fig. 2 and other calibration dependences obtained for ^{147}Sm, ^{149}Sm, ^{150}Sm, ^{154}Sm. We used GSO 2462-82 tableted powder for calibration and then analyzed a sample of Ta_2O_5 doped with samarium. That is, the analysis involved samples of different phase and chemical composition. New possibilities of the proposed research method can be used for other powder materials tableted without a binder. Table 2 demonstrates the results.

The LA-ICP-MS analysis has demonstrated a high precision (the degree of proximity of the results of the analysis obtained by independent methods in specific regulated conditions) with a X-ray fluorescence method: the results corresponded to given values of samarium concentrations detected due to the weight of dopant added at the $Ta_2O_5\langle Sm\rangle$ precursor synthesis stage (Table 2).

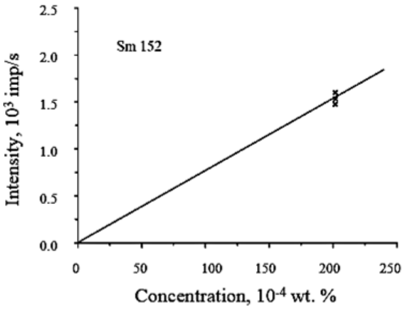

Fig. 2. Calibration dependence for ^{152}Sm, obtained using tableted apatite concentrate 2462-82 with the content 0.021 wt.% of Sm_2O_3

Рис. 2. Градуировочная зависимость для ^{152}Sm, полученная с использованием таблетированного ГСО 2462-82, содержащего 0,021 мас.% Sm_2O_3

We have confirmed chemical composition of $Ta_2O_5\langle Sm\rangle$. This is the reason why these samples were used as standards for the construction of calibration characteristics at detection of samarium concentration in the $LiTaO_3\langle Sm\rangle$ charge samples. A $Ta_2O_5\langle Sm\rangle$ nature is close to the one of $LiTaO_3\langle Sm\rangle$, this determines why we prefer a $Ta_2O_5\langle Sm\rangle$ standard to apatite concentrate 2462-82.

Fig. 3 demonstrates calibration function of ^{152}Sm. It was built using a number of $Ta_2O_5\langle Sm\rangle$ charge powder tablets. We have obtained analogues dependences for isotopes ^{147}Sm, ^{149}Sm, ^{150}Sm, ^{154}Sm. We have also found equations of calibration dependences:

for ^{147}Sm Int (imp/s)=(5.6818±0.0567) · C_{Sm}(ppm), R=0.999255,

for ^{149}Sm Int (imp/s)=(5.1059±0.0553) · C_{Sm}(ppm), R=0.999120,

for ^{150}Sm Int (imp/s)=(2.6770±0.0242) · C_{Sm}(ppm), R=0.999388, (1)

for ^{152}Sm Int (imp/s)=(9.3591±0.0715) · C_{Sm}(ppm), R=0.999563,

for ^{154}Sm Int (imp/s)=(7.6194±0.0702) · C_{Sm}(ppm), R=0.999363,

where R is a coefficient of approximation of a linear calibration function, 1 ppm=10^{-4} wt.%.

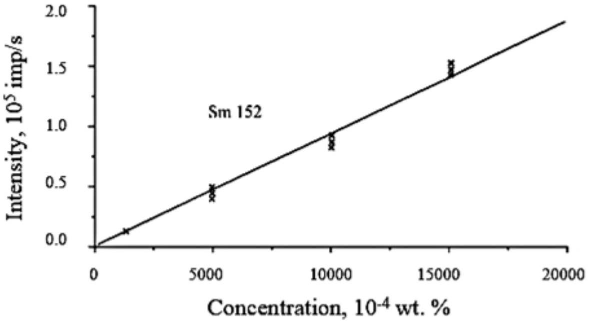

Fig. 3. A dependence of instrumental response intensity for ^{152}Sm on samarium concentration in tableted samples $Ta_2O_5\langle Sm\rangle$

Рис. 3. Зависимость интенсивности аналитического сигнала ^{152}Sm от концентрации самария в таблетированных пробах $Ta_2O_5\langle Sm\rangle$

Table 2

Results of samarium detection in researched samples

***Таблица 2*. Результаты обнаружения самария в исследованных образцах**

$Ta_2O_5\langle Sm\rangle$				$LiTaO_3\langle Sm\rangle$			
Given concen-tration, wt.%	According to LA-ICP-MS analysis, wt.%	Relative standard deviation S_r, %	According to X-ray fluorescence analysis, wt.%	Given concent-ration*, wt.%	According to LA-ICP-MS analysis, wt.%	Relative standard deviation S_r, %	According to X-ray fluorescence analysis, wt.%
0.1	0.105	3.25	0.093	0.094	0.095	6.69	0.104
0.5	0.494	4.19	0.480	0.470	0.446	5.08	0.453
1.0	0.988	2.00	0.972	0.940	0.918	4.00	0.894
1.5	1.560	2.55	1.429	1.410	1.375	4.09	1.420

Note: * Concentration values were calculated according to the given Sm concentration in Ta_2O_5 in $LiTaO_3$ charge of congruent composition (molar ratio [Li]/[Ta]=0.9417)

Примечание: * Значения концентрации рассчитаны по заданной концентрации Sm в Ta_2O_5 в шихте $LiTaO_3$ конгруэнтного состава (мольное отношение [Li]/[Ta]=0,9417)

A ration of natural samarium isotopes in a row ^{150}Sm:^{149}Sm:^{147}Sm:^{154}Sm:^{152}Sm corresponds to 1:1.87:2.03:3.08:3.62. A ratio between angular coefficients of dependences (1) was 1:1.91:2.12:2.85:3.50 in this row. This fact together with extrapolation to the coordinate basic origin confirmed the correctness of obtained approximations and the correctness of use of tableted powder apatite concentrate 2462-82 for the LA-ICP-MS analysis. We have used equations (1) for a quantitative LA-ICP-MS analysis of $LiTaO_3\langle Sm\rangle$.

A uniformity of TR distribution was proved by a relative standard deviation S_r of the instrumental response intensity measured in the experiment on research of dopant microhomogeneity. A relative standard deviation characterized both homogeneity of the analyte distribution and a standard deviation of the instrumental measurements. Table 2 demonstrates S_r values for samples with Sm. A figure in [5] demonstrates a change in the instrumental response for different isotopes during the LA-ICP-MS analysis for samarium. Graphical data obtained due to an analogues method for dysprosium is demonstrated on Fig.4.

A dispersity of reproducibility of the instrumental response intensity detected from the value of relative standard deviation: did not depend on the velocity and step length of the scanning; did not exceed the dispersity obtained at analysis of a chemically uniform standard SRM NIST 612; did not exceed the dispersity obtained at LA-ICP-MS analysis of glasses and geological samples. The sum of these features confirms chemical uniformity the macro-dopant in researched samples Ta_2O_5 and $LiTaO_3$.

The graphs had the same character at analysis of all samples containing TR. For the Ta_2O_5⟨TR⟩ samples S_r (%) parameter was 2-6, for $LiTaO_3$⟨TR⟩ charge - 4-7, which is lower than analysis error (up to 10%). This confirms chemical uniformity of TR distribution in samples under consideration.

CONCLUSION

Working parameters of LA was shown to be interconnected at the LA-ICP-MS analysis. Powder samples of Ta_2O_5⟨TR⟩ precursor created without a binder can be used for the quantitative LA-ICP-MS analysis of doped lithium tantalate charge. Results obtained by the LA-ICP-MS analysis were confirmed by X-ray fluorescent method and coincide well with it. Ta_2O_5⟨TR⟩ precursors and $LiTaO_3$⟨TR⟩ charge have revealed that dopant distribution is chemically uniform. An absence of a matrix effect of tantalum on the instrumental response intensity was detected at analysis of tableted samples.

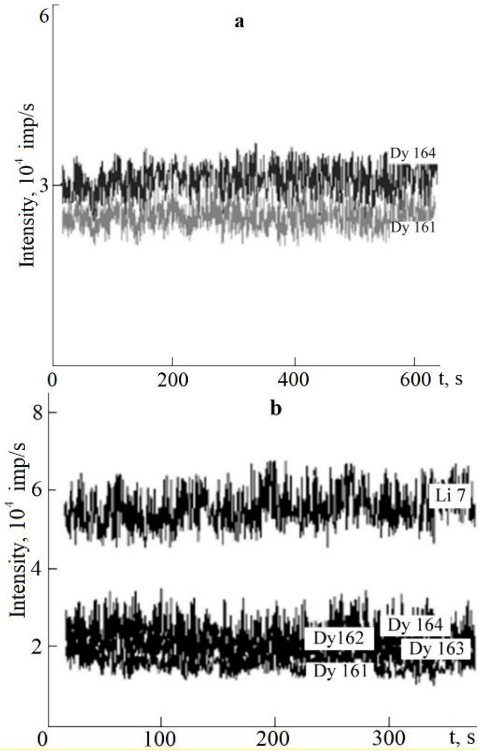

Fig. 4. The change in instrumental response intensity of Dy isotopes at stepwise laser ablation (a step was 7-9 μm) across the tablets sample surface: a - Ta_2O_5⟨1.5 wt.% Dy⟩, b – $LiTaO_3$⟨1.35 wt.% Dy⟩. A laser beam diameter was 100 μm, laser impulses frequency was 5Hz, laser pulse power was 60%

Рис. 4. Изменение интенсивности аналитического сигнала изотопов Dy при пошаговой лазерной абляции (шаг 7-9 мкм) по поверхности таблетированного образца: а - Ta_2O_5⟨1,5 мас.% Dy⟩, b - $LiTaO_3$⟨1,35 мас.% Dy⟩. Диаметр лазерного пучка 100 мкм, частота следования импульсов 5 Гц, мощность лазерного импульса 60%

CONFLICT OF INTERESTS

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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