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FABRICATION AND INVESTIGATION OF HETEROSTRUCTURES BASED ON LANTHANUM MANGANITES

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VILNIAUS UNIVERSITETAS
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ĮVAIRIALYČIŲ LANTANO MANGANITŲ SANDŪRŲ GAMINIMAS IR TYRIMAS

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Introduction

During the last few decades, great progress in electronics has been achieved mainly due to the developing of modern technologies and complex research of electronic transport phenomena in thin semiconductor films and related device structures. Up to now, the silicon still remains the most important material of modern electronics. However, increasing attention is paid also to a number of new materials exhibiting specific electrical and magnetic properties which are not typical for Si.

New ferromagnetic materials and particularly ferromagnetic oxides such as mixed valence manganites referred to by the general formula $R_{1-x}A_xMnO_3$ (here R = La, Nd, and A = Ca, Sr, Ba, Pb, Ce) and exhibiting spin-polarized carriers are of key importance for the development of spin-dependent electronics (spintronics). To meet increasing needs of spintronics there were attempts to prepare various multilayered device structures composed of ferromagnetic oxides, semiconducting and dielectric thin films. In addition, attention was paid also to various hybrid device structures possessing integration of the ferromagnetic oxides into Si-based electronic circuits. A number of experiments have been carried out to elaborate high crystalline quality manganite films and related heterostructures containing traditional semiconductors. Use of the manganite-based heterostructures has been demonstrated recently for the development of magnetic field sensors, magnetic memory elements and other devices.

New promising possibilities for future spintronics could be expected due to the integration of the so-called multiferroic oxides into various multilayer device structures. This kind of materials exhibit simultaneously ferroelectric and ferromagnetic (antiferromagnetic) properties and unusually strong correlation between polarization vector and internal magnetic moment. Bismuth ferrite ($BiFeO_3$) with perovskite-like structure demonstrating multiferroic properties at room temperature is one of the most promising materials belonging to this group of compounds, however electrical and magnetic properties of the compound has not yet been fully understood and technology of thin $BiFeO_3$ films and related heterostructures with other magnetic oxides such as manganites has not been elaborated.

Up to now, major attention of researchers was focussed mainly on magnetic and electrical properties of thin oxide films. Individual manganite films and their properties have been studied relatively well meanwhile their heterostructures continue to surprise us with new phenomena. One can conclude that following needs of rapidly developing spintronics more attention needs to be focused on both preparation and complex investigation of multilayered structures and heterostructures based on magnetic oxides as manganites and multiferroic compounds.

Major goals of this work

• To prepare high crystalline quality lanthanum manganite thin films doped by divalent (Ca^{2+}, Sr^{2+} and Ba^{2+}) and tetravalent (Ce^{4+}) ions and to investigate electrical and magnetic properties of the heterostructures formed between different manganite films.
• To investigate electrical and magnetic properties of the heterojunctions formed by growing thin films of lanthanum manganites on highly conductive $SrTiO_3$ <Nb> substrates.
• To investigate crystalline structure and electrical properties of thin manganite films doped by various ions, when grown on silicon and to investigate electrical and magnetic properties of the prepared "manganite-Si" heterostructures.
• To clarify functionality of the heterostructures containing constituent layers of manganites and multiferroic bismuth ferrite, $BiFeO_3$.

Novelty of scientific investigation

• It was found for the first time that thin intermediate layer of high electrical resistance formed at the interface between $La_{2/3}Ca_{1/3}MnO_3$ and $La_{2/3}Ce_{1/3}MnO_3$ thin films is responsible for non-linear current-voltage characteristics of the $La_{2/3}Ca_{1/3}MnO_3$ / $La_{2/3}Ce_{1/3}MnO_3$ heterojunction.
• Comparative study of the $La_{2/3}Ba_{1/3}MnO_3$ / STON, $La_{2/3}Ca_{1/3}MnO_3$ / STON and $La_{2/3}Ce_{1/3}MnO_3$ / STON heterostructures has been performed for the first time. Negative magnetoresistance has been measured for all these heterostructures at low voltage values. Meanwhile positive magnetoresistance values have been indicated for the heterojunctions when forward bias voltage exceeded a certain critical value corresponding to a steep increase of a forward current**.**
• Dominating electrical transport mechanisms have been clarified for the $La_{2/3}A_{1/3}MnO_3$ / STON (A ≡ Ba, Ca, Ce) heterojunctions. It was found that current versus voltage relations of the prepared "manganite/STON" heterostructures at high temperatures (when the manganite layer is in a paramagnetic state) can be described by Schottky thermionic emission model.

Importance for application:

• $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ba, Ca, Sr, Ce) thin films grown heteroepitaxially on lattice-matched $SrTiO_3$(100) and $SrTiO_3$<Nd>(100) substrates by dc magnetron sputtering and pulsed laser deposition demonstrating high crystalline quality can be used for the fabrication of multilayer structures of various spintronic devices.

• The $La_{2/3}Ce_{1/3}MnO_3$ / $La_{2/3}Ca_{1/3}MnO_3$ bilayer structures with magnetic field-dependent current versus voltage characteristics could be used for the fabrication of magnetic field sensors.

• The prepared $La_{2/3}Ba_{1/3}MnO_3$ / STON, $La_{2/3}Ca_{1/3}MnO_3$ / STON and $La_{2/3}Ce_{1/3}MnO_3$ / STON heterostructures, with magnetic field dependent current-voltage relationships can be used for measuring of magnetic field.

• The prepared LCaMO / *n*-Si, LSrMO / *n*-Si and LBaMO / *n*-Si heterostructures demonstrating nonlinear and asymmetric magnetic field-dependent current versus voltage relationships typical for semiconductor *p-n* diode structures show promising possibilities to integrate the manganite films into Si-based integrated circuits.

• A role of the doping on structural stability and electrical properties of multiferoic $BiFeO_3$ thin films has been elucidated. The performed investigations revealed promising opportunities for the integration of this compound into manganite-based heterostructures and related spintronics devices.

Statements carried out for defence:

1. $La_{2/3}Ce_{1/3}MnO_3$ thin films grown on $SrTiO_3$ and *n* - $SrTiO_3$ <Nb> (100) substrates show hetergeneous state in the vicinity of the manganite-substrate interface characterized by certain amount of CeO_2 precipitates surrounded by cation-deficient regions with reduced carrier density and lower Curie temperature values compared to those in the bulk.

2. The $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Sr, Ce) / *n* - $SrTiO_3$<Nb>(100) heterojunctions, as well as those formed by growing polycrystalline manganite films on n-Si substrates exhibit rectifying behavior, characterized by strongly asymmetric *I* - *U* curves. The current-voltage characteristics of the heterojunctions with manganite layers being in a paramagnetic state, can be described by Schottky thermionic emission model.

3. The $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Sr, Ce)/(STON) and $La_{2/3}Ca_{1/3}MnO_3$ / *n*-Si heterstructures are characterized by negative magnetoresistance values at low forward bias U ($eU << \Phi$, where Φ is a barrier height) and positive magnetoresistance values at $eU > \Phi$.

4. Substitution of 10 % Bi atoms by La and replacement 10 % of both Bi and Fe by La and Mn atoms in the parent $BiFeO_3$ compound enables synthesis of multiferroic compounds with reduced amount of secondary phases and higher electrical resistance values. The heterostructures containing layers of mixed valence manganites and multiferroic $BiFeO_3$ provide new opportunities for the fabrication of novel spintronics devices.

Publication and approbation of scientific results

Major results of the thesis have been published in 7 referred scientific papers; they were also announced in 15 national and international conferences.

Content of the thesis

The thesis is divided into six chapters including short introduction and the conclusion's chapter. Main goals of the investigations, scientific novelty, practical importance and main statements are presented in the **Introduction.** The main goals and tasks of the work have been formulated. Short introduction of the problems to be solved and structure of the thesis are also presented.

The first chapter presents a review of main properties of the manganese oxides including their crystalline structure, phase diagrams. Major attention is paid to electrical and magnetic properties of the manganites used in this work for the preparation of heterostructures. This chapter also presents an analysis of the most important publications related to the investigations performed in this scientific work.

In the second chapter one can find general description of the methods used for the preparation, characterization and investigation of lanthanum manganese ferromagnetic thin films and related heterostructures.

In the third chapter attention is paid to the formation and investigation of the LCaMO / LCeMO bilayer films. In the beginning of these investigations there were indications in literature that partial substitution of La^{3+} by tetravalent cations such as Ce results *n*-type conductivity of the lanthanum manganite [1]. The main goal of our investigations was to certify presence of *n*-type conductivity of Ce-doped lanthanum manganite films by forming *p* - *n* structure based on Ca- and Ce-doped manganite thin films.

Diode is one of the most important devices of current electronics. Therefore, preparation of a magnetic field driven diode based on *n*- and *p*-type manganite films could be of greatest importance for the development of new device structures for future spintronics [2]. A lot of research interest has been devoted recently for the fabrication of various heterojunctions based on CMR manganite films [3].

The LCaMO / LCeMO bilayer films were patterned in the cross-stripe geometry to investigate transverse electrical transport of the prepared manganite heterojuctions. The patterned tape-like underlying LCaMO film (200 μm in width) and the top LCeMO film (1 mm in width) were deposited by PLD at 750°C onto lattice-matched crystalline $SrTiO_3$ (STO) (100) substrate through special masks. Low resistance electrodes used for the electrical measurements were prepared by sputtering coatings of metallic Ag onto a top surface of the LCaMO and LCeMO films. The drawing of the samples used for investigations is presented in Fig.1.

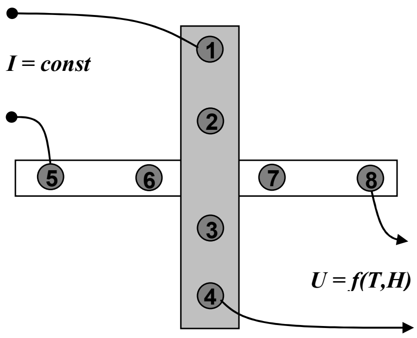

Fig.1. Cross-stripe geometry of the LCaMO/LCeMO heterostructure.

The suggested geometry made it possible to measure parameters of both individual layers of the manganite films as well as electrical properties of the heterojunction formed between different manganite films. The interface resistance has been estimated by investigating vertical electrical transport between the adjacent LCaMO and LCeMO manganite layers, i. e. by passing current between leads 1, 5 and measuring voltage between leads 4, 8 (see Fig.1).

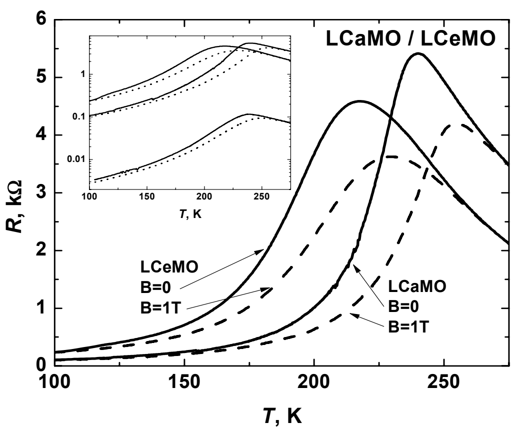

Fig.2. Resistance versus temperature of the LCaMO and LCeMO films and that of the LCaMO / LCeMO interface (the lowest curve in the inset).

All the measurements were performed at T = 300 K and T = 78 K. Major results of electrical measurements of LCaMO and LCeMO films and heterojunction are presented in Fig. 2. It can be seen from this figure that both LCaMO and LCeMO films and the LCaMO / LCeMO interface exhibit similar peak-like resistance versus temperature behavior with the peak temperatures (corresponding to characteristic resistive transitions of manganite films) at 235 K, 210 K, and 215 K, respectively. Note significant decrease of the interface resistance, $R_{\perp}$, with applied magnetic field ($B \approx 1$ T) indicated below the characteristic peak temperatures T_C. The observed decrease of the interface resistance with magnetic field demonstrates negative magnetoresistance of the LCaMO/LCeMO interface.

Fig. 3 demonstrates nonlinear *I* - *U* characteristics of the LCaMO / LCeMO interface at $T = 78$ K and $T = 300$ K. Nearly symmetrical characteristics (in respect to current direction change) avoid presence of *n*-type conductivity of the LCeMO layer. Resistance of the LCaMO / LCeMO interface was found to exceed total resistance of the bilayer film (with current passing perpendicular to the interface plane) at least by about one order of magnitude. So our investigations reveal formation of thin interlayer at the interface characterized by increased resistivity compared to that in a bulk of the LCaMO or LCeMO layers.

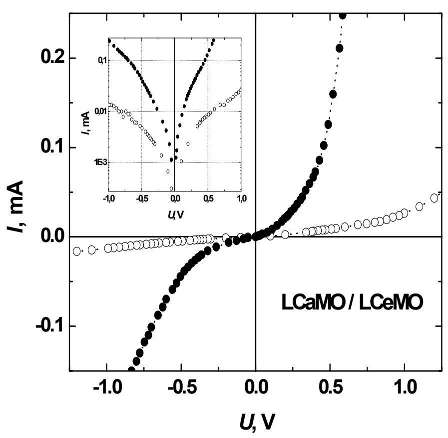

Fig.3. Current versus voltage plots of the LCaMO/LCeMO interface measured at 300 K (solid circles) and 80 K (circles).

The origin of this interlayer may be associated with reduced Ce concentration leading to a reduced carrier density at the interface. One can expect that instability of the LCeMO compound in respect to formation of CeO_2 impurity phase should be enforced at the interface due to existence of interface strain.

Almost symmetrical voltage-current relations of the LCaMO / LCeMO heterojunction (in respect to current direction change) observed in this work certify hole doping of the LCeMO compound in accordance to Ca, Sr and Ba-doped manganites. Up to now both *n*- [4] and *p*-type [5] conductivity has been reported for the LCeMO system. Thus, further research is needed to elucidate finally a role of cerium ions on doping properties of lanthanum manganites.

The forth chapter presents main results of preparation and investigation of high crystalline quality Ca-, Ba- and Ce-doped lanthanum manganite thin films as well as related heterojunctions prepared by growing heteroepitaxially these manganite films on highly conducting *n*-type Nb-doped strontium titanate (STON) substrates. The heterojunctions formed between *p*-type manganites and *n* - STON can be very promising for applications, especially for the development of magnetic field sensors and information storage devices. However, the research and formation of these heterojunctions are still in the initial stage.

It is worth noting also that band theory of conventional semiconductors is not valid for these systems and the underlying physics of manganite-based heterojunctions is still unclear. Studies on this kind of heterojunctions could improve our understanding of the interface between strongly correlated systems and promote application in novel spintronics devices.

The manganite heterojunctions formed by growing LCaMO, LBaMO and LCeMO films onto 0.1 % Nb-doped $SrTiO_3$ (100) were fabricated either by the magnetron sputtering (MS) or pulsed laser deposition (PLD) methods. Crystalline structure of the prepared films was examined by high resolution XRD using Cu K_α radiation. High crystalline quality of LCaMO and LBaMO films when deposited on lattice matched STON substrates has been certified by measuring their θ - 2θ X-ray diffraction spectra. Clearly defined reflection peaks corresponding to the (*l*00) plane family were observed in θ-2θ spectra of all the films investigated. Similar XRD patterns have been measured for the LCeMO films (see Fig.4), although X-ray diffraction spectra in this case revealed traces of additional reflexes at about 28° and 33° demonstrating presence of negligible amount (<<1%) of CeO_2 impurity phase (see Fig.4 inset).

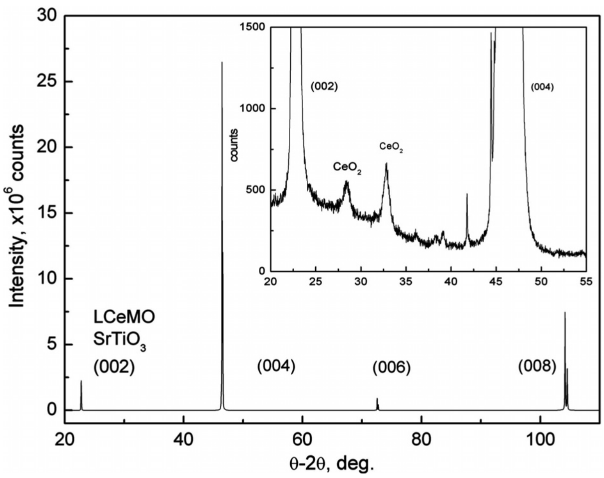

Fig.4. Θ-2Θ patterns of XRD scans measured for LCeMO thin film grown on $SrTiO_3$ (100) substrate. Inset presents an enlarged view of XRD scan demonstrating weak reflexes of CeO_2.

Lattice constant of CeO_2 is about 0.541 nm, and the corresponding lattice spacing in [110] crystallographic direction is close to that of the lattice constant of both LCeMO film and STO substrate. It means therefore that CeO_2 inclusions can coexist in the LCeMO film as intergrowths with a rotation angle of 45°.

Considering that the LCeMO film is inhomogeneous in structure at the LCeMO / STON junction, one can conclude that there are cation-deficient regions and Ce-rich regions in LCeMO film at the interface with STON. Since the Ce-rich regions are of low conduction, the transport behavior of the junction is mainly determined by the cation-deficient regions in the LCeMO film to form many *p* - *n* junctions in parallel. In this case, the behavior of the LCeMO / STON junction is similar to that of a junction based on typical manganites [6].

In-plane resistance of the films, $R_{//}$, versus temperature, was investigated by applying a standard four point-probe method with DC current of about 0.01 mA passing in a film plane (CIP geometry) (Fig.5 upper inset). Ag coatings (2.0 × 2.0 mm^2) sputtered onto the tape-like manganite films were used as low contact resistance electrodes for the electrical measurements. The $R_{//}(T)$ curves with clearly defined resistance maxima seen at the characteristic temperatures T_m, of about 280 K, 250 K and 275 K have been measured for the LBaMO, LCaMO and LCeMO films, respectively.

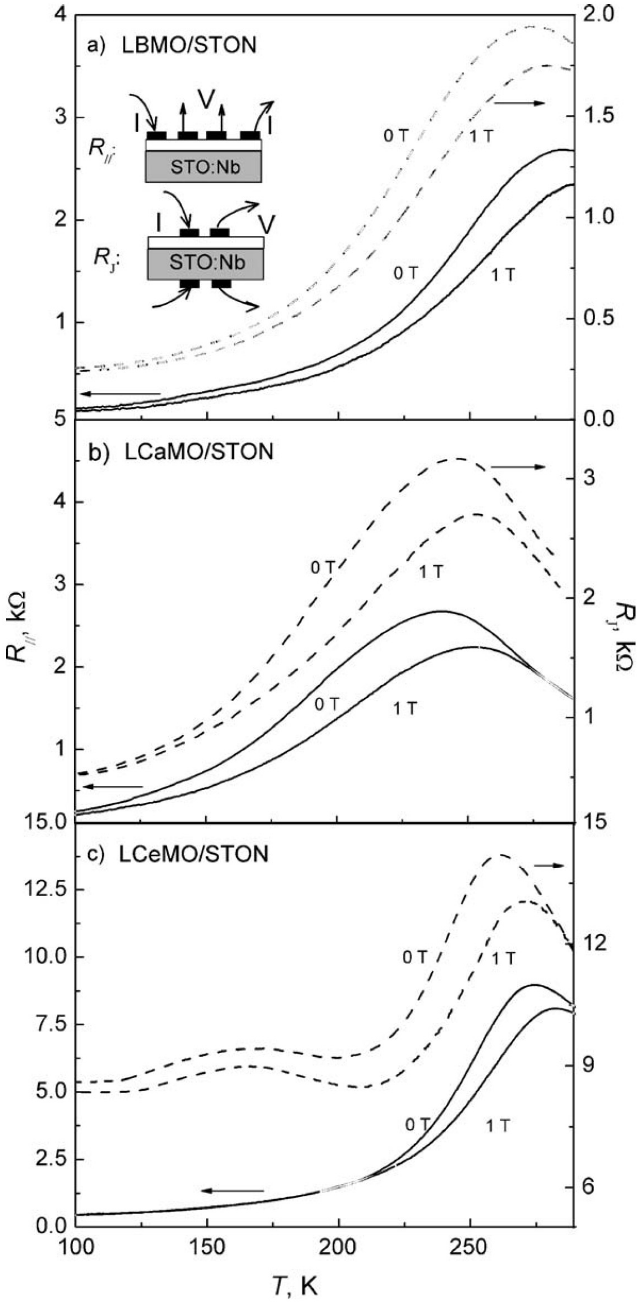

Fig.5. In-plane resistance $R_{II}(T)$ and zero-field junction resistance $R_J(T)$ measured for the LBaMO / STON, LCaMO / STON and LCeMO / STON heterostructures at zero magnetic field and under magnetic field (B = 1.0 T). Insets show geometry of the contact leads used in this work for R_{II} and R_J measurements.

The current-perpendicular-to plane (CPP) geometry of electrical contacts was applied to investigate the characteristic resistance of the heterojunctions, R_J. The $R_J(T)$ curves of the LBaMO / STON, LCaMO / STON and LCeMO / STON heterojunctions are displayed in Fig.5 by dashed lines for the comparison. Similarity of the measured $R_J(T)$ and $R_{II}(T)$ curves for oxygen saturated LCaMO / STON heterojunction can be seen from Fig.5b.

Following this figure, it is worth noting slight shift of the PM / FM transition temperature corresponding to the LBaMO film material at the interface compared to that in a bulk (see Fig.5a). In addition, we point out presence of additional resistance maximum for the LCeMO / STON junction at about 160 K (Fig.5c) indicating, probably, the largest content of CeO_2 inclusions at the interface due to existence of an interfacial strain.

Magnetoresistance of the all prepared manganite films and the "manganite/STON" heterojunctions was investigated by applying magnetic field ($B \approx 1.0$ T) normal to a film plane while current was passing both in-plane (CIP geometry) and perpendicular to plane (CPP geometry. Main results of these measurements are presented in Figure 5.

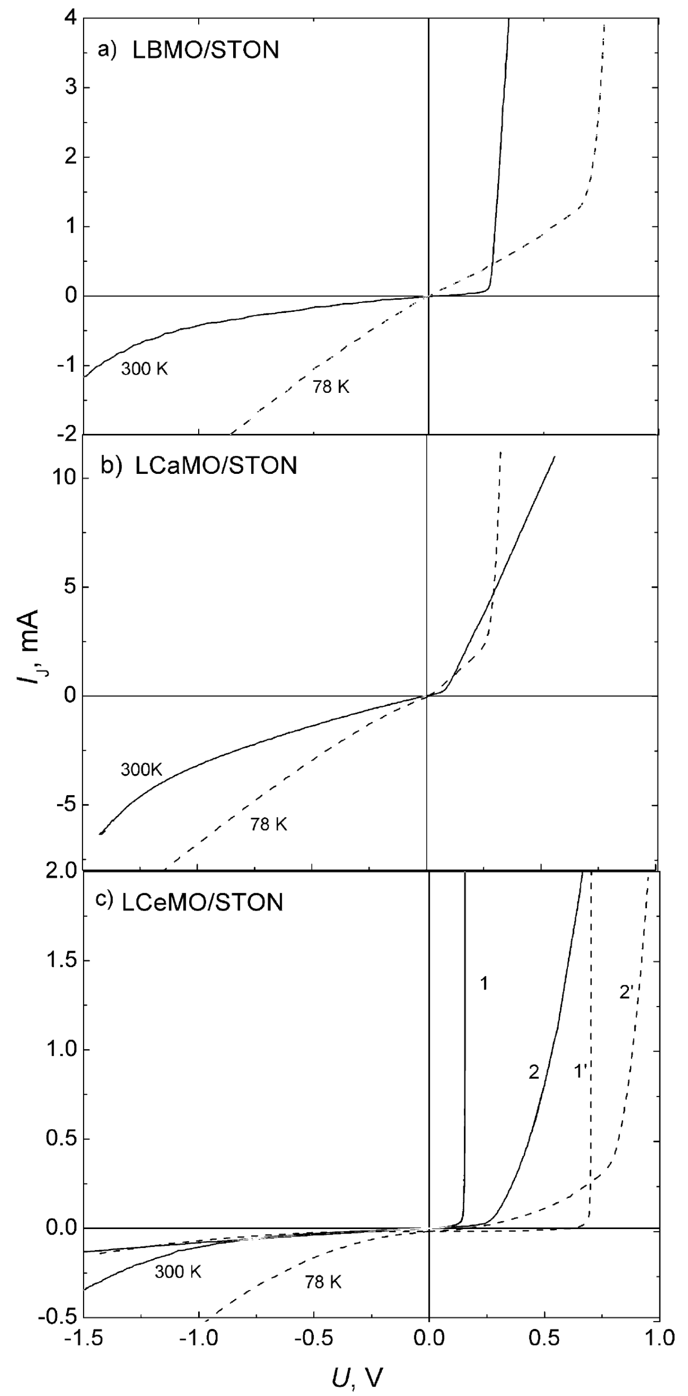

Fig. 6. Current – voltage characteristics of oxygen-saturated LBaMO / STON, LCaMO / STON and LCeMO / STON heterojunctions measured at $T = 300$ K (solid lines) and $T = 78$ K (dashed lines) in the absence of magnetic field.

Current-voltage (I - U) relations of the heterojunctions formed by growing LBaMO, LCaMO and LCeMO manganite films on STON (see Fig.6) revealed clearly defined rectifying behavior both at T = 78 K and 300 K. However, all these heterojunctions showed features different from conventional semiconductor p-n junctions. It can be seen from the Fig.6 that differential resistance of the LBaMO / STON, LCaMO / STON and LCeMO / STON heterojunctions, $R_J = d(U) / d(I)$, at zero bias decreases significantly with T decreasing from 300 K down to 78 K.

Fig.7 demonstrates a set of $I - U$ curves in a case of a forward bias measured for the LBaMO / STON heterojunction at various temperatures. Similar characteristics were obtained for other heterojunctions. At $U \ll U_d$, (U_d is the interfacial potential, corresponding to a steep current increase at a forward bias) the measured $I - U$ curves were almost linear in the whole temperature range. The estimated U_d values for the LBaMO / STON and LCeMO / STON heterojunctions were found to increase monotonously with temperature decreasing.

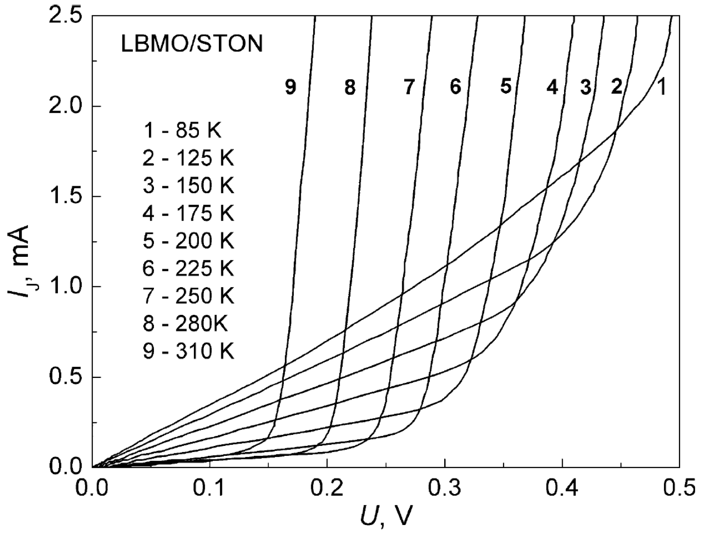

Fig.7. Set of current – voltage characteristics measured in a case of forward bias for the LBaMO / STON heterostructure at various temperatures.

The U_d (T) plots shown in Fig.8 by solid and empty triangles were obtained for the same LCaMO / STON heterojunction measured just after film growth and after several cooling-heating cycles, respectively. Thus, we point out instability of the LCaMO / STON heterojunction.

Taking into account relatively large lattice mismatch for the LCaMO / STON heterostructure (of about 1.2% compared to 0.025% and 0.2% for the LBaMO / STON and LCeMO / STON, respectively) we associate this effect with possible epitaxial strain at the LCaMO / STON interface and possible reduction of the strained state in the manganite film by generating dislocations during film heating and cooling.

Magnetoresistance of the LCaMO / STON, LBaMO / STON and LCeMO / STON heterojunctions was found to depend strongly on temperature, magnetic field and current flowing through the junctions. Negative magnetoresistance under applied magnetic field has been indicated for the heterostructures at low forward bias values ($U < U_d$). However, a certain (almost parallel) shift of the I - U curves to higher voltages with applied external magnetic field (at $U > U_d$) resulted positive junction magnetoresistance values.

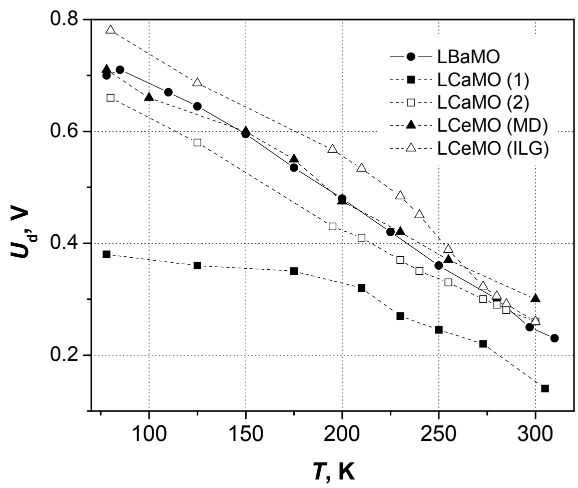

Fig.8. Diffusion voltage U_d as function of temperature estimated for the LBaMO / STON, LCaMO / STON and LCeMO / STON heterojunctions. Solid and opened squares show U_d versus T for the same LCaMO / STON heterostructure measured just after preparation and after several cooling and heating cycles, respectively.

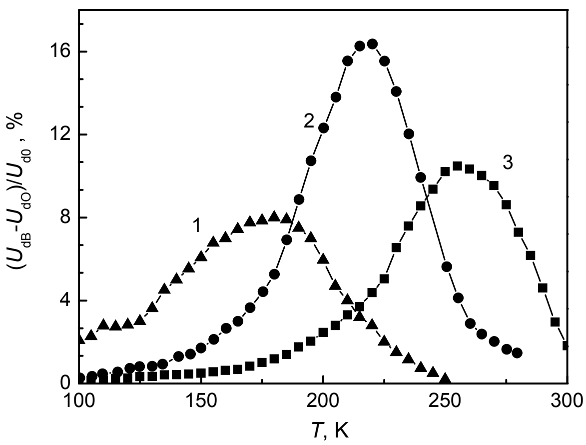

Fig.9. Relative increase of the interface potential U_d under applied magnetic field ($B = 1$ T) measured for the LCaMO / STON (1), LCeMO / STON (2) and LBaMO / STON (3) heterojunctions.

Relative increase of the characteristic U_d values ($\Delta U_d / U_{d0}$) with applied magnetic field $B = 1$ T measured in this work for the LCaMO / STON, LCeMO / STON and LBaMO / STON heterojunctions is shown in Fig.9. Peak-like ($\Delta U_d / U_{d0}$) versus T curves seen from the figure have been explained taking into account unusually strong dependence of the interfacial potential on magnetic field at temperatures just below the ferromagnetic to paramagnetic phase transition temperature of the manganite films.

In a case of a forward bias, the I - U relations measured for the heterojunctions at room temperature were fitted well by applying the well known Schottky diode model $j = j_s \exp(qU/nk_BT)$ (here n is the ideality factor).

At high temperatures, the I - U curves of the heterojunctions plotted in a semi-logarithmic scale were almost parallel to each other and the ideality factor values were close to unit (see Fig.10). Significant increase of n values at low temperatures shows that additional transport mechanisms should be applied to explain experimental I - U characteristics of the "manganite / STON" heterojunctions at low temperatures i. e. below Curie temperature of the manganites.

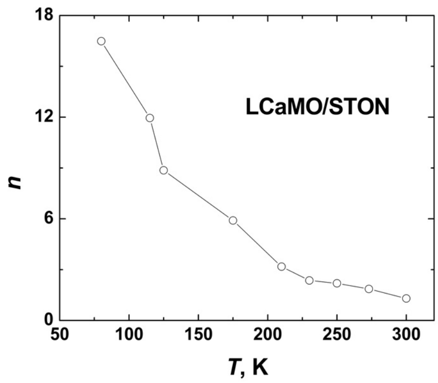

Fig.10. LCaMO/STON heterostructures ideality factor n as function of temperature.

Summarizing our results we conclude that the "manganite / STON" p - n heterojunctions could be promising for the fabrication of planar magnetic field sensor arrays due to sufficiently large values of MR and the ability to change the junctions' properties by applied electrical and magnetic fields.

In the fifth chapter we present the main results of preparation and investigation of LCaMO, LSrMO and LBaMO thin films and heterojunctions composed of these lanthanum manganites and Si substrates. Integration of manganites onto semiconducting materials such as on Si remains a challenging task for potential applications that utilize both information processing and data storage in the same device [7].

XRD measurements revealed presence of single phase LSrMO, LCaMO and LBaMO films when grown onto *n*-type Si substrates although crystalline structure of all these films was polycrystalline. Typical surface roughness of the films investigated by atomic force microscopy (AFM) was found to be in the range of 20 ÷ 35 nm.

The "manganite / *n* - Si" heterostructures exhibited significantly higher resistance values at 78 K than at 300 K compared to similar heterojunctions formed between manganites and STON and demonstrated semiconductor-like junction resistance increase with *T* decreasing in the whole temperature range from 300 K to 78 K.

The current-voltage curves, measured for the LCaMO / Si heterojunctions at room temperature and at $T = 80$ K, are displayed in Fig.11. Similar characteristics were measured also for the LBaMO / Si and LSrMO / Si heterojunctions. All these heterostructures exhibited clearly defined rectifying current-voltage characteristics. Differential resistance of the heterostructures (at zero bias) was found to increase monotonously with cooling from 300 K down to 78 K.

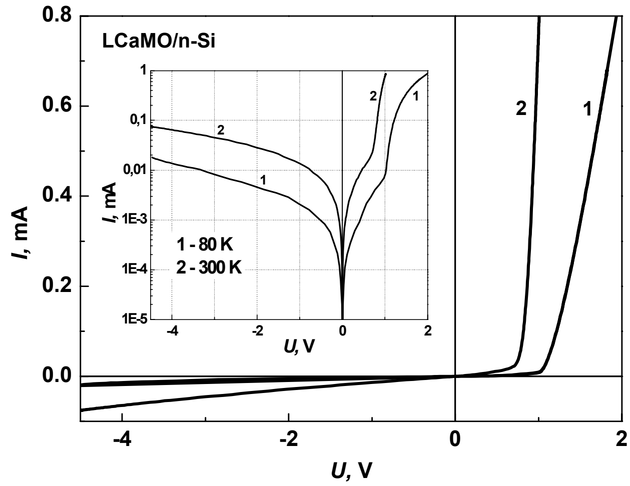

Fig.11. Current – voltage characteristics measured in a case of forward and reverse bias for the LCaMnO / *n*-Si junction at $T = 300$ K and $T = 80$ K ($B = 0$). Inset shows lg(I) = $f(U)$ characteristics of this heterojunctions.

Summarizing results of this chapter we point out the possibility of *p* - *n* heterojunction formation by "in-situ" depositing hole-doped (Ca, Ba, Sr) lanthanum manganite films directly onto electronically doped Si substrate with preliminary removed native SiO_2. Studies of electrical properties showed that the as prepared "manganites / Si" heterostructures exhibit rectifying *I* - *U* characteristics both at room temperature and at 78 K. It was found in this work that the *I* - *U* characteristics of the heterojunctions depend slightly on magnetic field. The LBaMO / Si and LSrMO / Si heterojunctions demonstrated negative magnetoresistance both at 78 K and 300 K.

Meanwhile change of magnetoresistance values from positive to negative with forward bias has been indicated for the LCaMO / Si heterojunction in accordance to similar phenomenon reported earlier for the "manganite / STON heterojunctions (Fig.12).

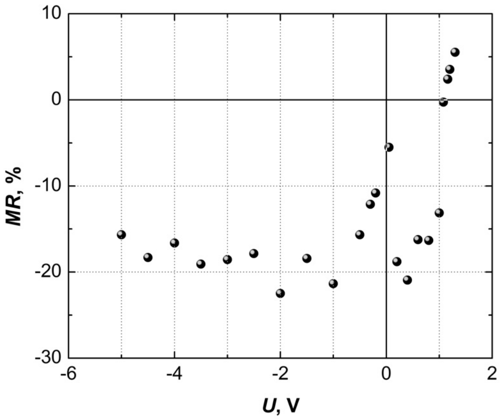

Fig.12. Magnetoresistance of the LCaMnO / *n*-Si heterojunction versus applied voltage ($T = 80$ K, $B = 0.9$ T).

The demonstrated possibility to grow single phase manganite films on Si substrates, clearly defined rectifying properties of the prepared "manganite/Si" heterojunctions and their magnetoresistance make the manganites promising both for their integration in Si based electronics circuits and fabrication of new device structures for spintronics.

In the sixth chapter we present our recent studies on preparation and investigation of non-doped and doped thin films of multiferroic $BiFeO_3$ (BFO) and their heterojunctions with STON, *n*-Si and manganites. Multiferroic $BiFeO_3$ with a pseudo-cubic unit-cell parameter ($a \approx 3.96$ Å) of a rhombohedrally distorted perovskite structure is a typical ferroelectric below 1103 K and slightly canted antiferromagnet below 643 K [8].

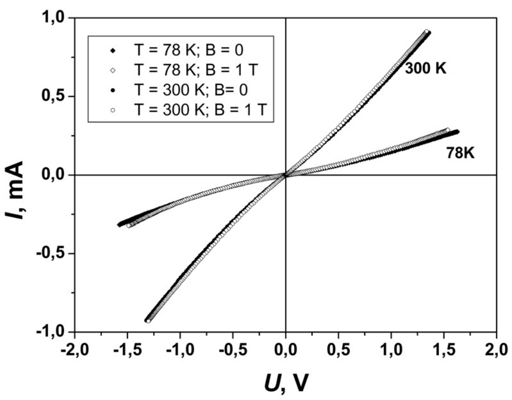

Fig.13. Current – voltage characteristics of the $La_{2/3}Sr_{1/3}MnO_3$ / $BiFeO_3$ / *n* - Si heterojunction measured at $T = 300$ K ir $T = 78$ K.

This compound is very promising for the fabrication of new types of devices for spintronics, ferroelectric memory applications and sensors due to unique electrical and magnetic properties. Furthermore, it may be used also for the integration into heterojunctions containing ferromagnetic manganites due to a rather good matching of lattice parameters with a number of perovskite oxides. BFO heterostructures with hole-doped manganite layers on STON, *n* - Si substrates exhibit nonlinear magnetic field-dependent current-voltage relations (Fig.13). La*A*MO / BFO / *n* - Si heterostructures provide new opportunities for the fabrication of novel spintronics devices.

MAIN RESULTS AND CONCLUSIONS

1. $La_{2/3}Ce_{1/3}MnO_3$ / $La_{2/3}Ca_{1/3}MnO_3$ heterojunctions prepared by heteroepitaxial overgrowth of the $La_{2/3}Ce_{1/3}MnO_3$ films on the underlying $La_{2/3}Ca_{1/3}MnO_3$ layers exhibited magnetic field-dependent electrical properties. Nonlinear voltage-current characteristics of the $La_{2/3}Ce_{1/3}MnO_3$ / $La_{2/3}Ca_{1/3}MnO_3$ heterojunction have been explained assuming carrier transport through a self-formed thin depletion layer, i. e. tunnelling of carriers through the interfacial barrier and electric field-dependent hoping of carriers in the depletion region following from Pool Frenkel theory.

2. Presence of CeO_2 impurity phase in the $La_{2/3}Ce_{1/3}MnO_3$ film at the LCeMO / STON interface results formation of the interfacial region with reduced carrier concentration and lower Curie temperature values compared to those in a bulk of LCeMO film. Results of the investigations confirm hole-type electrical conductivity of the $La_{2/3}Ce_{1/3}MnO_3$ layer.

3. Heterojunctions formed by heteroepitaxial growth of thin $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Ce) layers on crystalline n - STO<Nb>(100) substrates as well as by deposition of polycrystalline $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Sr) layers on n - Si substrates exhibit nonlinear voltage-current characteristics in a wide temperature range (80 K ÷ 300 K). Presence of an interfacial potential barrier resulting formation of a depletion layer at the interfaces is responsible for magnetic field-dependent rectifying properties of the heterojunctions.

4. It was found in this work that Shottky diode model can be applied to explain forward voltage-current characteristics of the $La_{2/3}Aa_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Ce) / STON and $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Sr) / n - Si junctions in the high temperatures range, when manganites layers are in a paramagnetic state. A role of tunnelling through a barrier on I - U characteristics of the heterojunctions has been determined by fitting experimental data with those of numerical calculations following thermoelectronic emission and Newman theory models.

5. The “manganite / STON” heterojunctions studied in this work exhibited negative magnetoresistance at low forward voltage values ($U < U_d$, where U_d is the interfacial potential of the barrier) while positive magnetoresistance values have been measured at $U > U_d$.

6. Changing of magnetoresistance values from negative to positive has been indicated for the LCaMO / *n* - Si junction at 80 K with forward bias increasing. Positive magnetoresistance values of the heterojunction have been explained assuming carrier transport through the interfacial barrier and probable increasing of the barrier height with applied magnetic field.

7. The influence of doping by Nd, La, Mn ions on crystalline structure, surface quality and electrical conductivity of thin multiferroic $BiFeO_3$ films grown on $SrTiO_3$, *n*-$SrTiO_3$<Nb> and *n*-Si substrates has been investigated. It was found that substitution 10% of Bi atoms by La and replacements 10 % of both Bi and Fe by La and Mn atoms in the parent $BiFeO_3$ compound enable synthesis of multiferroic films with reduced amount of secondary phases and higher electrical resistance values. Resistivity values of about 2×10^6 Ωcm and 10^7 Ωcm, at 300 K and 78 K temperature, respectively, have been evaluated for the $Bi_{0.9}Nd_{0.1}FeO_3$ film.

8. It was found that heterostructures formed by growing $Bi_{0.9}Nd_{0.1}FeO_3$ and $Bi_{0.9}La_{0.1}Fe_{0.9}Mn_{0.1}O_3$ films on *n* - STON, *n* - Si substrates and hole-doped manganite layers exhibit nonlinear and rectifying current-voltage relations. Heterostructures containing layers of mixed valence manganites and multiferroic $BiFeO_3$ provide new opportunities for the fabrication of novel spintronics devices.

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REZIUMĖ

Disertacijoje nagrinėjamos įvairios galimybės panaudoti feromagnetinių oksidų – manganitų sluoksnius bei jų darinius naujų spintronikos prietaisų gaminimui. Šio darbo pagrindiniu tyrimo objektu pasirinktos sandūros, sudarytos tarp dvivalenčiais (Ca, Ba, Sr) ir keturvalenčiais (Ce) jonais legiruotų lantano mangano oksidų plonųjų sluoksnių, o taip pat tarp manganitų ir elektroninio laidumo $SrTiO_3$<Nb> (STON) bei *n* - Si padėklų. Darbe pateiktas išsamus minėtų manganitų sluoksnių ir jų darinių gaminimo magnetroninio dulkinimo ir impulsinio lazerinio garinimo būdais aprašymas. Disertacijoje pateikti plonųjų manganitų sluoksnių, užaugintų ant skirtingų padėklų, kristalinės struktūros bei paviršiaus kokybės tyrimo duomenys, aprašytas magnetiniu lauku valdomų diodinių darinių formavimas, jų elektrinių bei magnetinių savybių tyrimai, įvertinti svarbiausi atskirų manganitų sluoksnių bei jų diodinių darinių elektrofiziniai parametrai.

Atlikus kompleksinius keturvalenčiais Ce^{4+} jonais legiruotų lantano manganito sluoksnių kristalinės sandaros bei elektrinių savybių tyrimus nustatyta, kad šie sluoksniai pasižymi ne elektroniniu, kaip buvo skelbta anksčiau, o skyliniu elektriniu laidumu. Skylinis junginio elektrinis laidumas paaiškintas nežymiu šalutinės CeO_2 fazės ir katijonų vakansijų susidarymu auginamuose sluoksniuose .

Pateikti Ca, Ba, Sr ir Ce jonais legiruotų manganitų įvairialyčių darinių palyginamieji tyrimai, įvertinta padėklo įtaką kristalinės manganitų sandaros tobulumui, paviršiaus šiurkštumui taip pat jų elektrinėms bei magnetinėms savybėms.

Visapusiškai ištirta sandūrų, sudarytų tarp įvairiais jonais legiruotų manganitų sluoksnių ir elektroninio laidumo STON bei *n* - Si padėklų elektrinių ir magnetinų savybių priklausomybė nuo išorinio magnetinio lauko. Ištyrus šių sandūrų voltamperines charakteristikas plačiame temperatūrų ruože buvo pastebėtas jų magnetovaržos ženklo pasikeitimas didėjant pralaidžiosios krypties įtampai. Nurodytos galimos manganitų sandūrų neigiamos ir teigiamos magnetovaržos atsiradimo priežastys.

Papildomai šiame darbe buvo sprendžiamos multiferoinio bismuto ferito $BiFeO_3$ junginio, pasižyminčio tuo pačiu metu tiek magnetinėmis tiek ir feroelektrinėmis savybėmis, struktūrinio stabilizavimo problemos bei galimybes panaudoti unikalias šios medžiagos savybes įvairiuose lantano manganitų dariniuose. Šių tyrimų rezultatai yra svarbūs tuo, kad gauti nauji rezultatai bei jų analizė turėtų pasitarnauti ateityje gaminant magnetiniu lauku valdomas p-n sandūras, kurios labai praverstų kuriant naujus magnetiniu bei elektriniu lauku valdomus spintronikos prietaisus.

Darbo tikslai

- Ištirti dvivalenčiais (Ca^{2+}, Sr^{2+} ir Ba^{2+}) ir keturvalenčiais (Ce^{4+}) jonais legiruotų lantano manganitų plonųjų sluoksnių kristalinę sandarą bei įvairialyčių sandūrų, sudarytų tarp šių lantano manganitų sluoksnių, elektrines ir magnetines savybes.
- Ištirti įvairialyčių sandūrų, sudarytų tarp įvairių lantano manganitų sluoksnių ir laidžiųjų $SrTiO_3$<Nb> padėklų, elektrines ir magnetines savybes.
- Ištirti įvairių cheminių sudėčių lantano manganitų sluoksnių, užaugintų ant elektronikos pramonėje naudojamų Si padėklų, kristalinę sandarą, o taip pat įvairialyčių sandūrų, sudarytų tarp šių lantano manganitų ir Si, elektrines bei magnetines savybes.
- Išaiškinti manganitų funkcines galimybes panaudojant jų daugiasluoksnius darinius su multiferoiniu bismuto feritu ($BiFeO_3$).

Atliktų tyrimų rezultatų naujumas:

- Pirmą kartą nustatyta, kad skiriamojoje riboje tarp $La_{2/3}Ca_{1/3}MnO_3$ ir $La_{2/3}Ce_{1/3}MnO_3$ plonųjų sluoksnių susidaro didelės elektrinės varžos tarpsluoksnis, lemiantis netiesines voltamperines charakteristikas ir netiesinę šios sandūros elektrinės varžos priklausomybę nuo elektrinio lauko.
- Pirmą kartą atlikti palyginamieji įvairialyčių $La_{2/3}Ba_{1/3}MnO_3$/STON, $La_{2/3}Ca_{1/3}MnO_3$ / STON ir $La_{2/3}Ce_{1/3}MnO_3$ / STON sandūrų elektrinių ir magnetinių savybių tyrimai. Išmatuota neigiama sandūrų magnetovarža, esant mažoms įtampos vertėms bei magnetovaržos ženklo pasikeitimas, įtampai viršijus kritinę vertę, atitinkančią staigų srovės didėjimą pralaidžiąja kryptimi.
- Išaiškinti vyraujantys $La_{2/3}A_{1/3}MnO_3$ / STON ($A \equiv$ Ba, Ca, Ce) sandūrų elektrinio laidumo mechanizmai. Parodyta, kad $I = f(U)$ priklausomybės, lantano manganitams esant paramagnetineje būsenoje, gali būti aprašytos panaudojant Šotkio termoelektroninės emisijos modelį.

Disertacijos praktinė vertė:

- Naudojant magnetroninio dulkinimo ir impulsinio lazerinio garinimo technologijas ant suderintų gardelių STO ir STON padėklų epitaksiškai užauginti kokybiški $La_{2/3}A_{1/3}MnO_3$

($A \equiv$ Ba, Ca, Sr, Ce) sluoksniai, tinkantis daugiasluoksnių struktūrų ir įvairių spintronikos prietaisų gaminimui.

- Pagamintos $La_{2/3}Ce_{1/3}MnO_3$ / $La_{2/3}Ca_{1/3}MnO_3$ sandūros, pasižyminčios magnetiniu lauku valdomomis elektrinėmis savybėmis, gali būti panaudotos magnetinio lauko jutiklių gaminimui.
- Pagamintos $La_{2/3}Ba_{1/3}MnO_3$, $La_{2/3}Ca_{1/3}MnO_3$ ir $La_{2/3}Ce_{1/3}MnO_3$ feromagnetinių oksidų heterosandūros, pasižyminčios magnetiniu lauku valdomomis lyginimo savybėmis.
- Suformuotos LCaMO / *n* - Si, LSrMO / *n* - Si ir LBaMO / *n*-Si sandūros, pasižyminčios netiesinėmis ir puslaidininkiniam diodui būdingomis bei magnetiniu lauku valdomomis lyginimo savybėmis plačiame temperatūrų ruože, parodė tradicinių puslaidininkių integravimo galimybes spintronikai vystyti.
- Įvertinta legiravimo įtaka multiferoinio $BiFeO_3$ junginio struktūriniam stabilumui bei parodyta šios medžiagos panaudojimo mišraus valentingumo manganitų heterostruktūruose galimybė, praplečiant šiuolaikinės elektronikos ribas.

Disertaciją sudaro 6 skyriai, iš kurių paskutinis – rezultatų apibendrinimas.

Įvadiniame skyriuje nagrinėjamas problemos aktualumas, formuluojamas darbo tikslas bei uždaviniai, aprašomas mokslinis darbo naujumas, pristatomi autoriaus pranešimai ir publikacijos, disertacijos struktūra.

Pirmajame skyriuje apžvelgta mišraus valentingumo manganitų kristalinė sandara ir fazinės diagramos, aprašytos elektrinės ir magnetinės šių medžiagų savybės, nurodyti pagrindiniai modeliai, paaiškinantys elektros transporto mechanizmus ir magnetovaržos prigimtį. Pabaigoje apžvelgti pagrindiniai literatūroje pateikti moksliniai darbai, aprašantys feromagnetinių manganitų pagrindu sudarytų heterosandūrų savybes.

Antrajame skyriuje trumpai aprašyti pagrindiniai šiame darbe naudojami plonųjų sluoksnių auginimo būdai: magnetroninis dulkinimas ir impulsinis lazerinis garinimas. Trumpai aprašyti darbe panauduoti kristalinės struktūros, elektrinių bei magnetinių savybių ir kiti plonųjų sluoksnių ir heterosandūrų tyrimo metodai.

$La_{2/3}Ca_{1/3}MnO_3$ / $La_{2/3}Ce_{1/3}MnO_3$ struktūra aprašyta **trečiajame skyriuje**. Šiame skyriuje patekta heterostruktūros geometrija, jos auginimo sąlygos, kristalinės struktūros bei paviršiaus tyrimo rezultatai. Taip pat nagrinėjama magnetinio lauko įtaka plonųjų sluoksnių bei sandūros elektrinėms savybėms

Ketvirtajame skyriuje pateikti gana išsamūs $La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Ce) / $SrTiO_3$:Nb heterostruktūrų tyrimų rezultatai. Skyriuje aprašomos elektrinės ir magnetinės sluoksnių ir sandūrų savybės. Nustatomos Šotkio barjero teorijos taikymo ribos. Aprašomas neigiamos ir teigiamos magnetovaržos atsiradimo efektas bei pateikiami keli šį efektą paaiškinantys modeliai.

$La_{2/3}A_{1/3}MnO_3$ ($A \equiv$ Ca, Ba, Sr) sandūrų, užaugintų ant silicio padėklų, gaminimas ir tyrimas aprašytas **penktajame skyriuje**. Suformuotų LCaMO / *n* - Si, LSrMO / *n* - Si ir LBaMO / *n* - Si sandūrų netiesinės ir puslaidininkiniam diodui būdingos bei magnetiniu lauku valdomos lyginimo savybės parodė tradicinių puslaidininkių integravimo galimybes į spintronikos struktūras.

Šeštajame skyriuje pristatyti šiuo metu vykdomi tyrimai, skirti bismuto ferito ploniesiems sluoksniams ir parodytos šios medžiagos integravimo galimybes į lantano manganitų struktūras. Pateikiami legiravimo įtaką BFO struktūros stabilizavimui patvirtinantys rezultatai.

Disertacijos pabaigoje pateikti pagrindiniai atlikto darbo rezultatai ir suformuluotos išvados, išvardinta cituojama literatūra bei darbo tema paskelbti darbai.

Darbo aprobacija

Darbo metu gauti ir disertacijoje aprašomi rezultatai atspausdinti 7 moksliniuose straipsniuose ir pristatyti 15 tarptautinių bei nacionalinių konferencijų.

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FABRICATION AND INVESTIGATION OF HETEROSTRUCTURES BASED ON LANTHANUM MANGANITES

Summary of Doctoral Dissertation
Technological Sciences, Materials Engineering (08T)

Jelena Devenson

ĮVAIRIALYČIŲ LANTANO MANGANITŲ SANDŪRŲ GAMINIMAS IR TYRIMAS

Daktaro disertacijos santrauka
Technologijos mokslai, medžiagų inžinerija (08T)