VILNIUS GEDIMINAS TECHNICAL UNIVERSITY
SEMICONDUCTOR PHYSICS INSTITUTE

Skirmantas KERŠULIS

MAGNETIC AND ELECTRICAL FIELD EFFECTS IN POLYCRYSTALLINE $La_{1-X}Sr_XMnO_3$ FILMS

SUMMARY OF DOCTORAL DISSERTATION

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VILNIAUS GEDIMINO TECHNIKOS UNIVERSITETAS
PUSLAIDININKIŲ FIZIKOS INSTITUTAS

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MAGNETINIO IR ELEKTRINIO LAUKO EFEKTAI POLIKRISTALINIUOSE $La_{1-X}Sr_XMnO_3$ SLUOKSNIUOSE

DAKTARO DISERTACIJOS SANTRAUKA

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Introduction

Topicality and problem of the work. Electronic properties of lanthanum manganites $La_{1-x}M_xMnO_3$ (*LSMO*), where M=Ca^{2+}, Sr $^{2+}$, Ba^{2+}, exhibiting phase transition from paramagnetic to ferromagnetic state, colossal negative magnetoressitance and negative electroresistance effects have been one focus of research in the past few decades. Despite that the most investigations were carried out on epitaxial manganite films the recent research of polycrystalline perovskite manganites has been renewed motivating by fundamental understanding as well as potential device applications. The investigations of magnetoresistance (*MR*) performed in films grown on bicrystal substrates introducing artificial grain boundaries (*GB*s), or ultrathin films grown on monocrystalline substrates having high mismatch of lattice constants, revealed advantages of such films if compare with epitaxial ones: significant value of *MR* over a wide temperature range, initial rapid rise with applied field (low field magnetoresistance, *LFMR*), and a slow almost linear increase up to very high fields (*HFMR*). It was demonstrated that *GB*s are mostly responsible for the observed effects despite that their formation methods could be different. The electrical transport properties studied in manganite films having artificial *GB*s revealed highly nonlinear current-voltage characteristics at high voltages (named electroresistance, *ER*) which were explained taking into account a multi-step inelastic tunnelling of charge carriers via several localized states in *GB*s. In order to gain an understanding of the *MR* and *ER* properties and the role of *GB*s in these phenomena, it is important to study transport mechanisms in polycrystalline *LSMO* films grown on lucalox substrate, especially focussing to randomly oriented crystallites with naturally formed grain boundaries.

The object of investigations. The object of investigation is $La_{1-x}Sr_xMnO_3$ polycrystalline films.

Aim of the work. The objective of this effort was to investigate electrical conductivity of $La_{1-x}Sr_xMnO_3$ polycrystalline films in low and high magnetic, and at strong electrical fields, to investigate how magnetoresistance and electroresistance effects depend on film preparation conditions such as deposition temperature, composition and thickness of the films.

Tasks of the work

1. To investigate how low magnetic field (0.001–1 T) magnetoresistance of $La_{1-x}Sr_xMnO_3$ films depends on film deposition temperature, composition and thickness of the films.

2. To study how high magnetic field (1–40 T) magnetoresistance of $La_{1-x}Sr_xMnO_3$ films depends on film deposition temperature, composition and thickness of the films.
3. To investigate how electroreisitance of $La_{1-x}Sr_xMnO_3$ films depends on film deposition temperature and composition at electrical field in the range 0–70 kV/cm.
4. To study how electroresistance of $La_{0.83}Sr_{0.17}MnO_3$ films depends on synchroniously applied magnetic field.
5. To investigate influence of Joule heating of polycrystalline $La_{0.83}Sr_{0.17}MnO_3$ manganite films using several tens of nanosecunde duration high electrical field pulses.
6. To analyze carrier transport mechanisms which determine magnetoresistance and electroresistance phenomena in manganite films.
7. To clear up possibility to develop isotropic magnetic sensor, contactless current sensor and limiter against electromagnetic pulse using polycrystalline $La_{1-x}Sr_xMnO_3$ films.

Methodology of research. Methods used for experimental investigations were based on generation and recording of low dc, high pulsed magnetic and electric fields. The results were analyzed using modified Mott's hopping conductivity, Glazman-Matveev inelastic tunnelling models as well as adiabatic Joule heating approach.

Scientific novelty. Novelties of this effort include the following results in the area of solid state physics and electronics.

1. It was obtained that in case of low magnetic fields the magnetoresistance (*MR*) phenomenon at low temperatures was larger for films prepared on substrates kept at lower temperatures (films with small size crystallites). In case of high temperatures ($T>T_m$), the *MR* phenomenon was larger if substrate was kept at higher temperatures. It was shown that in case of thin $La_{0.83}Sr_{0.17}MnO_3$ films at magnetic field lower than 200 mT the *MR* was less than 15%. It means that influence of spin polarized tunneling on carriers transport via grain boundaries was negligible.
2. It was shown that main features of *MR* phenomenon in thin polycrystalline $La_{0.83}Sr_{0.17}MnO_3$ films deposited on lucalox substrate can be explain assuming that charge carrier transport takes place mainly in magnetically and structurally disordered grain boundaries. It was suggested to explain main peculiarities of *MR* in these films in

temperature range from T_m to T_C using modified Mott's hopping conductivity model taking into account both ferromagnetic and paramagnetic phases.

3. It was obtained that in case of polycrystalline $La_{1-x}Sr_xMnO_3$ films the electroresistance (*ER*) phenomenon decreases for films grown at higher substrate temperatures and having higher Sr content x. The influence of grain boundaries on *ER* phenomenon was estimated. It was obtained that for films with larger size crystallites the main charge carriers transport mechanism is inelastic tunnelling via three localized states, while for films with smaller size crystallites it takes place only via two states.
4. Estimated Joule heating influence on electrical conductivity of polycrystalline $La_{0.83}Sr_{0.17}MnO_3$ films when these films were effected by strong electrical field pulses having duration of several ns. It was demonstrated that this influence was different for crystallites and grain boundaries.
5. It was shown that at low temperatures the magnetic field is able to increase the *ER* phenomenon in films with small size crystallites. For higher magnetic fields only the decrease of *ER* with increase of magnetic field was obtained.

Practical value. The obtained results enable to suggest two prototypes of electronic devices. The first is contactless electrical current sensor operating at room temperature and intended for 10–30 A electrical current sensing. The second is fast fault current limiter consisting of polycrystalline and epitaxial film operating at liquid nitrogen temperatures and generating 3.5 dB attenuation after 0.5 ns and 8 dB after several tens of ns. The results of investigation were also used for the improvement of high pulsed magnetic field sensor by decreasing value of *MR* anisotropy in the range of magnetic field 0.5–2 T.

Defended propositions

1. Low field magnetoresistance and its anisotropy in polycrystalline $La_{1-x}Sr_xMnO_3$ films grown at 650–750 ºC temperatures is determined by charge carrier transport in grain boundaries which properties could be guided by changing the dimensions of crystallites in the films. A decrease of their size results in increase of the *MR* in the ferromagnetic phase while in the paramagnetic phase the *MR* increases due to increased influence of the crystallites. The behavior of *MR* in high magnetic fields in the temperature range between T_m and T_C could be

explained by modified Mott's hopping model taking into account both ferromagnetic and paramagnetic phases.

2. *MR* anisotropy is highest in low magnetic fields, when *T* near to the T_m. It is proportional to the *MR* value and decreases at $T > T_m$ with decrease of composition *x*, film thickness or deposition temperature. In high magnetic fields (*B*>3 T) *MRA* does not depend on *x* and crystallite size and is negligible (<3%).
3. Carrier transport mechanism in $La_{1-x}Sr_xMnO_3$ polycrystalline films grown on lucalox substrate and having small size crystallites is mostly determined by inelastic tunneling through 3 localized states in *GB*s while for films having large size crystallites the tunneling through 2 states takes place. This determines higher value of electroresistance for films with small crystallites in the range $T<T_m$ while at $T>T_m$ the *ER* becomes smaller due to increased influence of crystallites interrior.
4. Low field magnetoresistance effect in polycrystalline $La_{1-x}Sr_xMnO_3$ films can be used for the development of contactless current sensor measuring 10–30 A currents and operating at room temperature. The electroresistance effect in these films and electrical bistability effect due to Joule heating in epitaxial films can be used for development of a fault current limiter against electromagnetic pulse operating at liquid nitrogen temperature.

Structure of the dissertation

The dissertation (in Lithuanian) includes introduction, 6 chapters, and main results and conclusions, references, list of publications.

The introduction contains relevance of the dissertation, aim of the work, tasks of the work, methodology of research, novelty and propositions to be defended.

1. Physical properties of manganite oxides

This chapter presents a literature review of the common physical properties of manganites. It describes their crystallographic structure, electrical structure, metal-insulator phase transition, paramagnetic and ferromagnetic phase separation. It also describes the main proposed charge transport models and their limitation. The results of manganites study in low and high magnetic and strong electrical fields are presented.

2. Preparation technique of the films and electrodes

This chapter describes a standard *MOCVD* and modified *PI CVD* film deposition techniques and electrical contacts formation. The contacts were made by thermal deposition of Ag using a Cr sub-layer, followed by standard negative photolithography. The substrate was cut into samples with area 0.5 mm^2.

3. Analysis of the experimental setups

This chapter presents a review of experimental setups used in the dissertation.

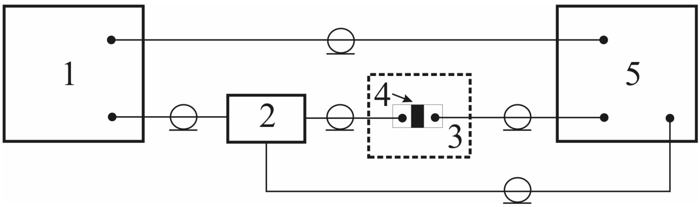


Fig. 1. Schematic diagram of ns pulse measurement equipment

The experimental setup of ns pulse measurement equipment is represented in Fig. 1. The sample 4 was connected in series to a 50 Ω impedance 10 GHz frequency transmission line. Rectangular shaped single electrical pulse with amplitude up to 1.2 kV, 5–18 ns duration and 0.15–0.5 ns rise time was generated with special generator 1. The devider 2 was used to divide pulse into two parts with ratio 1:80. The small part of the signal was used to record the applied voltage, while the larger one was transmitted to the sample. Oscilloscope 5 was used for signal recording. The sample was mounted in a closed cycle helium cryocooler 3 in order to perform measurements in the temperature range 4.2–300 K.

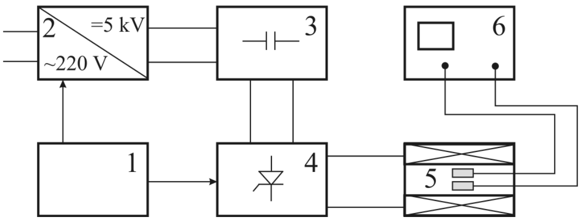


Fig. 2. Schematic diagram of setup used for measurements in the strong pulsed magnetic fields

The experimental setup used to investigate a *LSMO* films in low permanent magnetic field consisted of closed cycle helium cryocooler electromagnet which enabled to measure resistance in the range 1 mT–1 T.

The schematic diagram of strong pulsed magnetic field generation and measurement setup is presented in Fig. 2. It consists of control unit 1, capacitor bank charging unit 2, capacitors bank 3, high current semiconductors switch 4 and coil 5. Sample and magnetic field sensor were mounted inside the coil and responses were recorded by oscilloscope 6. System generates monopolar sine shape magnetic field pulse with length of 0.6 ms. Measurements where performed over temperature range 77–320 K. This chapter also presents measurement methods of manganite film resistance in pulsed magnetic field, and describes inductive and optical Faraday rotation methods used for magnetic field measurement and calibration. Final part contains a description of measurement setup of synchronized pulsed high electric and magnetic fields. The samples were connected in series to 50 Ω impedance and a 10 GHz bandwidth transmission line and then mounted inside the bore of a pulsed magnetic field coil. A 5 ns long electrical pulse was synchronized with a halfsine shaped magnetic pulse having a pulse length of 0.6 ms at the moment in time when it reached its maximal value. Due to the jitter in the synchronization of the electrical and magnetic pulses which was about 10 μs, the change in the magnetic field amplitude during *ER* measurement did not exceed 0.1%. Measurements were performed at temperatures 100–300 K.

4. Morphology, microstructure and elektrical properties of the samples

This chapter is intended for sample characterisation. The investigated samples were made from polycrystalline $La_{1-x}Sr_xMnO_3$ films having 25–400 nm thickness, deposited on lucalox (99.9% Al_2O_3 + 0.1% MgO) substrates at temperatures T_{sub} ranged from 650 °C up to 825 °C using the *PI MOCVD* technique. Chemical composition x was changed from 0.17 to 0.3.

Surface morphology and structure analysis of the films were performed by *RHEED*, *AFM*, *SEM* and *XRD* methods. *AFM* analysis of 400 nm thick films showed that decrease of T_{sub} from 825 °C to 650 °C results in a decrease of sizes of crystallites D from 170 nm to 110 nm. An increase of film thickness from 25 nm up to 360 nm resulted in increase of D from 75 nm up to 175 nm.

Fig. 3 presents resistivity dependence on temperature for films grown at different T_{sub} (a) and with different composition x (b). It shows typical for manganite film metal-insulator transitions at a certain critical temperature T_m. A decrease of T_{sub} resulted in increase of the resistivity maximum ρ_m and the shift of T_m to lower temperatures indicating that more *GB*s material is present in the film with smaller crystallites and they are more defected. An increase of x (Fig. 3b) results in the shift of the T_m to higher temperatures and decrease of the ρ_m.

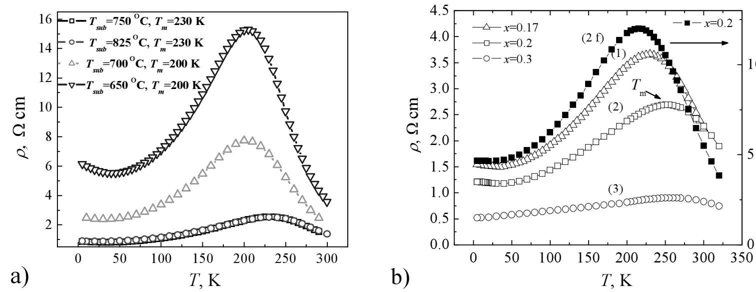


Fig. 3. Resistivity *vs*. temperature for films grown at different substrate temperatures (a) and chemical composition (b)

5. Colossal magnetoresistance and its anisotropy in polycrystalline LSMO films

This chapter is intended for studies of manganites properties in low and high magnetic fields. Magnetoresistance which is defined as $MR=100\%\cdot[R(B)-R(0)]/R(0)$, where $R(B)$ and $R(0)$ are resistance in magnetic field and without it, respectively, was measured.

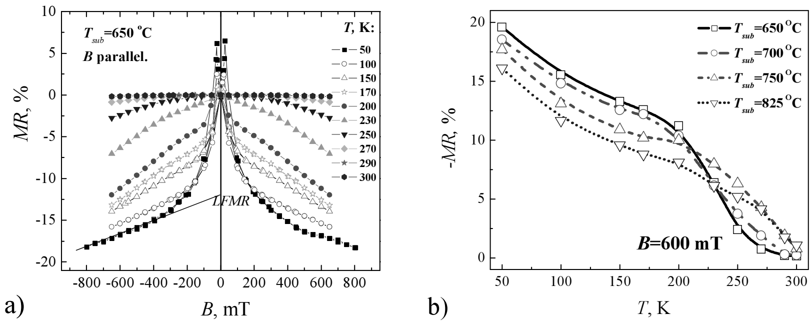


Fig. 4. *MR vs. B* characteristics at different temperatures (a), films grown at different substrate temperature *MR vs T* (b)

Low field magnetoresistance (*LFMR*), coercitivity, demagnetization field, magnetoresistance anisotropy (*MRA*) of *LSMO* films prepared at different conditions were also investigated. Fig. 4a presents *MR* dependence on magnetic field at different temperatures. One can see that increse of the tempterature results in decrease of *MR* of the film. *MR* dependence on *T* for various films is presented in Fig. 4b. It shows that films grown at lower substrate temperature have higher sensitivity to magnetic field at low temperatures, but smaller at high temperatures.

The *LFMR* estimation for various samples obtained from *MR vs. B* dependences (see Fig. 4a) showed that maximal *LFMR* value obtained at low temperatures was <15%, much less than 33% predicted for spin-polarized tunneling (*SPT*). Thus, in our investigated films the *SPT* is not prevailing transport mechanism. The *GB*s in our case can be analyzed as mesoscopic regions with lower magnetic order as compared with crystallites. The magnetic field increases magnetization of *GB* what leads to decrease of its resistance.

The investigations of the *MR* anisotropy showed that it is the highest in low magnetic fields close to temperature T_m. The *MRA* is proportional to *MR* and decreases at $T>T_m$ with decrease of composition *x*, film thickness or T_{sub}.

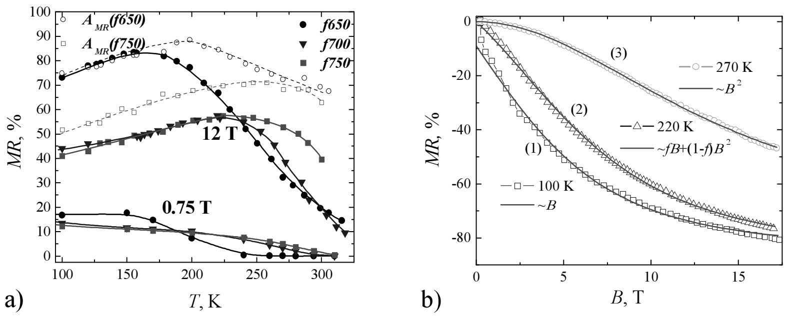


Fig. 5. Magnetoresistance *vs.* temperature at different substrate temperatures (a), *MR vs B* at different temperatures (b)

MR investigations in high magnetic field is one of priority for the development of high magnetic field sensors. The *MR* of films with different chemical composition *x* and grown at various T_{sub} was studied in the temperature range 100–320 K. It was obtained that the higher is ρ_m and lower T_m of the films the higher is maximal *MR* value. For films with smaller *x* the *MR* is larger at low temperatures and starts to decrease when *T* approaches T_m

value. The *MR* is highest at $T<T_m$ also for films grown at lowest T_{sub} (f650), see Fig. 5a. We suggested to analyse transport in *GB*s regions with reduced magnetic order using modified Mott's hopping model. In such a case the *MR* in ferromagnetic state suppose to scale the Brillouin function B, and we added *LFMR* term: $MR = \frac{\Delta\rho}{\rho} = A_{MR}\mathrm{B}\left(\frac{gJ\mu_B}{k_B T}\right) + LFMR$, while in paramagnetic state *MR* was analyzed as $MR = \frac{\Delta\rho}{\rho} = A_{MR}\mathrm{B}^2\left(\frac{gJ\mu_B}{k_B T}\right)$. Here spin moment *J* and A_{MR} are fitting parameters. At *T* between T_m and T_C which differ ≈ 20 K in our case, the experimental results can be fitted by sum of both contributions $MR = A_{MR}[f\mathrm{B} + (1-f)\mathrm{B}^2]$, where *f* is a fitting parameter of ferromagnetic phase fraction in the film.

The experimental *MR vs. B* dependencies (points) and fitting curves at various *T* are presented in Fig. 5b. It was found that parameter *J* at low temperatures is small and increases exponentially with *T*. Close to T_m the *J* is highest for the film with lowest T_m and highest *MR*. *J* values in the vicinity of T_m indicate that the *GB* in polycrystalline film behaves like a superparamagnet of magnetically aligned clusters with extension of typically 10 manganese ions.

Study of *MRA* in high magnetic field showed that it does not depend on *x* and crystallite size and is negligible (<3%).

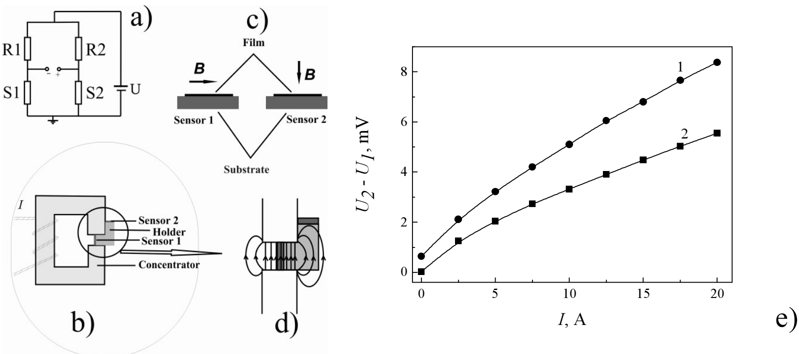


Fig. 6. Design of contactless current sensor: (a) electrical connections, (b) construction, (c) sensors position in respect of magnetic field, (d) magnetic field distribution in a gap, sensor signal at temperature 285 K (1) and 300 K (2), supply voltage 3.5V (e)

The obtained results of investigations of *MR* and *MRA* of *LSMO* films where used to upgrade *CMR*-*B*-scalar sensors and for the development of new contactless current sensor (Fig. 6). The sensor is made from flux concentrator and two manganites sensors. The first *CMR* sensor (sensitive to *B*) is placed in a gap so that its plane is parallel to the magnetic field direction. The second *CMR* sensor (insensitive to *B*) is placed inside the concentrator at sufficient distance from the gap where *B* is much smaller (see Fig. 6c).

Signals of both sensors at two temperatures are presented in Fig. 6e. The signal change is about 7.73 mV at 285 K and 5.53 mV at 300 K while "zero" temperature shift is about 0.62 mV in this *T* range, i.e. about 8% of sensor's signal at 285 K. The sufficient non-linearity of characteristics is observed in 0-2.5 A range, but it is not important for sensor operation when current to be controled is more than 7 A. For better sensitivity of the sensors electrical circuit with microcontroller can be used.

6. Strong electrical field effects in polycrystalline LSMO films

This chapter is intended for studies of manganite properties in high electrical field. It was obtained that polycrystalline manganites exhibit negative electroresistance (*ER*) phenomenon at applied high electrical fields. The *ER* is defined by the formula $ER=100\%\cdot[R(E)-R(0)]/R(0)$, where $R(E)$ and $R(0)$ are resistance at high and low electrical fields, respectively.

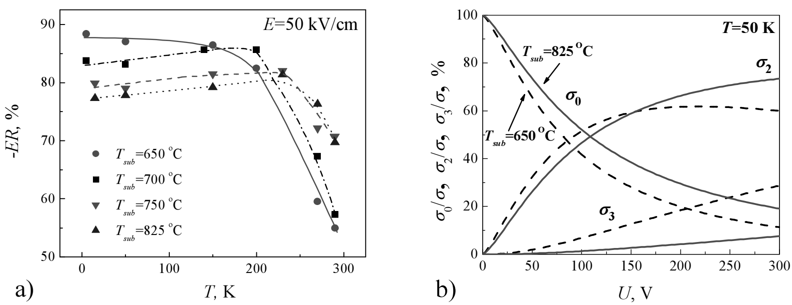
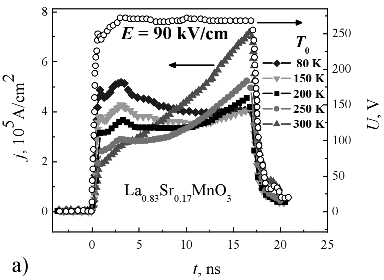


Fig. 7. Electroresistance *vs*. temperature at different substrate temperatures (a), relative σ_0, σ_2, σ_3 dependences on voltage (b)

ER dependences on *T* were investigated for films with different *x* and grown at different T_{sub}. It was found that with increase of *x*, the *ER* value in all temperature range decreases. Fig. 7a. presents the *ER* dependence on temperature for films deposited at different T_{sub}. At low temperatures the *ER* value increases with lowering T_{sub} while at higher temperatures ($T>T_m$) it decreases. It was found that *ER* starts at critical electrical field E_c. The investigations showed that with decrease of T_{sub} a number of *GB*s increases and thus the higher external voltage is needed to obtain the same value of *ER* (resistance of crystallites is many times smaller than *GB*s).

Carrier transport mechanisms were analyzed using formula $\sigma = \sigma_0 + \sigma_1 U^{\alpha}$, where σ_0 is specific conductance at low voltage, σ_1 and α are parameters related to carriers transport mechanism and non-linearity of current-voltage characteristic. The obtained α value was in the range between 1.3 and 2.5 predicted for multi-step inelastic tunneling through 2 or 3 localized states in *GB*s, respectively. For more detailed analysis we used formula $\sigma(U) = \sigma_0 + \sigma_2 U^{4/3} + \sigma_3 U^{5/2} + ...,$ where σ_0 cpecific conductance at low voltage, σ_2, σ_3 represent specific coefficients for terms related with tunnelling through 2 and 3 states in *GB*s, respectively. Contribution of different parts of σ to *ER* effect for different films is presented in Fig. 7b.

The influence of the high magnetic field on the electroresistance (*ER*) was studied at electrical field strengths up to 80 kV/cm in the temperature range from 100 K to 300 K. It was found that depending on the temperature and magnetic field value the *ER* can be enhanced or suppressed by the applied magnetic field. These results are explained by assuming that the *ER* appears due to inelastic multi-step tunneling through grain boundaries and by taking into account the redistribution of voltage across crystalline grains and grain boundary regions.



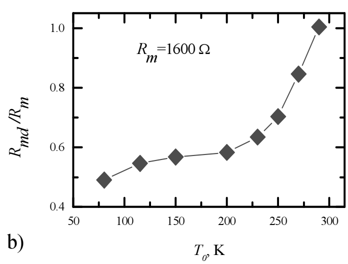


Fig. 10. Dynamics of current density through polycrystalline film measured at different initial temperatures T_0 (a), ratio of dynamic resistance maximum R_{md} corresponding to minimum of current pulse, and resistance maximum R_m obtained from ρ *vs* T dependence (b)

Influence of Joule heating on the resistance change in polycrystalline films at high electrical fields was studied. The high-current dynamics of *LSMO* film is presented in Fig. 10a. It was found that at low temperatures the dynamic resistance maximum R_{md}, which corresponds to minimum of the current in the pulse, is significantly lower than the resistance R_m obtained from the heating-free *R vs. T* dependence. Fig. 10b presents the ratio R_{md}/R_m as a function of the initial temperature T_0 of the film at which measurement was started. This ratio becomes equal to 1 at $T_0=T_m$ at which crystallites become paramagnetic (as well as the *GB*s).

It was shown that the *ER* effect of polycrystalline films and electrical bistability effect due to Joule heating in epitaxial films can be used for development of a fault current limiter against electromagnetic pulse. Fig. 11a presents schematic diagram of the limiter and its cross section along the $A–A'$ axis. Fig. 11b presents dynamics of limiter operation at 80 K: circular and triangular symbols correspond to input and output pulses, respectively. The solid line presents the simulation results. The inset shows the equivalent circuit of the limiter: R_E and R_P are resistances of epitaxial and polycrystalline films, respectively, and Z=50 Ω is the impedance of the stripline.

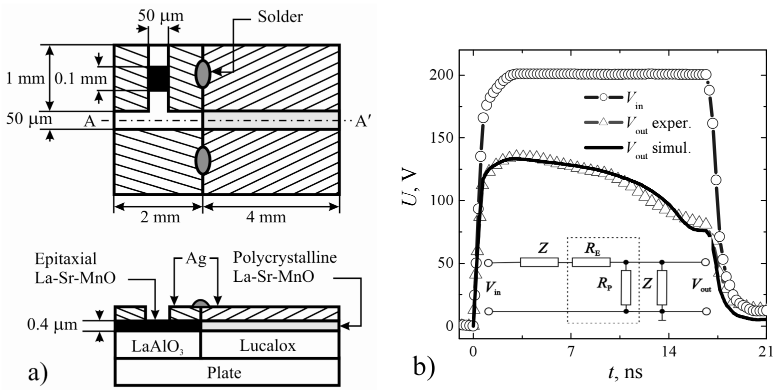


Fig. 11. Schematic diagram of the limiter and its cross-section along the A–A' axis (a), dynamics and electrical circuit of limiter at 80 K (b)

When fault current pulses with amplitudes greater than V_{th} are delivered to the input of the limiter, the resistance of the polycrystalline film connected in parallel to the transmission line decreases in subnanoseconds. It induces the attenuation of the line. Within a few ns, the current passing through the epitaxial film connected in series with the input of the limiter heats the film and causes its resistance to increase. This induces additional attenuation and decreases the current flow in the polycrystalline film, protecting it from high power dissipation. As a result, a 3.5 dB attenuation after 0.5 ns and 8 dB after 18 ns was obtained. Calculation of the temperature increase ΔT in the epitaxial film after each cycle was performed by assuming that the heating is homogeneous and that the cooling process is related only with the heat flow to the substrate.

General conclusions

1. It was obtained that $La_{1-x}Sr_xMnO_3$ films deposited on lucalox (99.9% Al_2O_3 + 0.1% MgO) substrates by *PI MOCVD* technique at T_{sub} =650–750 °C are polycrystalline, while at 825 °C are textured. The decrease of T_{sub} or thickness of the films results in decrease of dimensions of crystallites. The decrease of T_{sub}, thickness of the films or chemical composition x results in increase of the resistivity maximum ρ_m and the shift of T_m to lower temperatures.
2. Magnetoresistance value of thin polycrystalline $La_{1-x}Sr_xMnO_3$ films grown on lucalox substrate can be changed by changing chemical composition, substrate temperature and film thickness. The *MR* in ferromagnetic phase increases with decrease of T_{sub}, x or thickness. At high temperatures ($T>T_m$) the *MR* decreases due to increased crystallites influence.
3. Magnetoresistance anisotropy at low magnetic fields ($B<1$ T) is maximal close to T_m and increases with increase of x, dimensions of crystallites and film thickness at $T>T_m$. In high magnetic field the *MRA* does not depend on x and crystallite size and is negligible (<3%).
4. The *MR* phenomenon in thin polycrystalline *LSMO* films deposited on lucalox substrate can be explained using modified Mott's hopping model assuming that charge carriers transport mainly takes place in magnetically and structurally disordered grain boundaries (*GB*s). The *MR* dependence on magnetic field in these films in the temperature range between T_m and T_C can be explained using this model and taking into account both ferromagnetic and paramagnetic phases. Value of magnetic polaron J obtained as a fitting parameter increases exponentially with T and in the vicinity of T_m indicates that magnetically aligned clusters include approximately 10 manganese ions.
5. The electroresistance in polycrystalline $La_{1-x}Sr_xMnO_3$ films depends on chemical composition, dimensions of crystallites and T_m. In the feromagnetic phase the *ER* is maximal for films having small crystallites which dimensions define higher amount of the disordered *GB* material in the films. At room temperature the *ER* is higher for films with large size

crystallites. The threshold electrical field strength is highest for films having smallest crystallites. Carrier transport mechanism in the films can be described by Glasman-Matveev model: for films with small size crystallites the predominant charge carriers transport mechanism is inelastic tunneling through 3 localized states in *GB*s while for larger crystallites the tunneling through 2 states takes place.

6. The T_m of polycrystalline *LSMO* films shifts to higher temperatures with increase of electrical field strength. At ultrastrong electrical fields (>70 kV/cm) Joule heating of the films is observed during several tens of nanoseconds. The heating is inhomogeneous at $T<T_m$ due to different heating effect on crystallites and grain boundaries.
7. At low temperatures the magnetic field is able to increase the *ER* phenomenon in films with small size crystallites. For films with larger crystallites only decrease of the *ER* with magnetic field is obtained.
8. The obtained results enable to suggest prototype of contactless electrical current sensor based on polycrystalline $La_{1-x}Sr_xMnO_3$ film operating at room temperature and intended for 10–30 A electrical current sensing. The fault current limiter consisting of polycrystalline and epitaxial $La_{1-x}Sr_xMnO_3$ film operating at liquid nitrogen temperatures and generating 3.5 dB attenuation after 0.5 ns and 8 dB after several ns also can be developed.

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About the author

Skirmantas Keršulis was born in Alytus, on the 17 of August 1981. The first degree obtained in Telecommunication Physic and Electronics, Faculty of Physics, Vilnius University, in 2003. In 2005 – Master of Science, Faculty of Physics, Vilnius University. In 2005–2009 – PhD student of Semiconductor Physics Institute. At present – Assistant in High Pulse Power Laboratory of the Semiconductor Physics Institute.

MAGNETINIO IR ELEKTRINIO LAUKO EFEKTAI POLIKRISTALINIUOSE $La_{1-x}Sr_xMnO_3$ SLUOKSNIUOSE

Tiriamoji problema ir darbo aktualumas. Pastaruoju metu suaktyvėjęs susidomėjimas polikristaliniais manganitų sluoksniais yra nulemtas juose aptiktais tik tokioms nehomogeniškoms sistemoms būdingais efektais – silpno ir stipraus magnetinio lauko magnetovarža ir stipraus elektrinio lauko elektrovarža, kurie galėtų būti pritaikyti, kuriant naujus elektronikos prietaisus. Šiam tikslui reikia sugebėti valdyti sluoksnių parametrus ir naudoti pramoninę šių prietaisų gamybos technologiją. Šiuo požiūriu labai perspektyvi yra *MOCVD* auginimo technologija, o pigus polikristalinis polikoro padėklas, turintis mažą dielektrinę skvarbą ir sudarytas iš didelio kiekio išorientuotų mažų kristalitų, leidžia kryptingai keisti elektrovaržos ir magnetovaržos efektų dydį. Todėl šių sluoksnių tyrimai yra labai aktualūs. Elektros krūvininkų pernašos mechanizmai tokioje sistemoje yra labai sudėtingi, nes kristalituose juos nulemia feromagnetinių ir paramagnetinių sričių sąveika, o tarpkristalitinėse srityse – tuneliavimo procesai struktūriškai ir magnetiškai netvarkioje aplinkoje. Tačiau detalūs šių mechanizmų tyrimai $La_{1-x}Sr_xMnO_3$ plonuose sluoksniuose, užaugintuose ant polikoro padėklo, nebuvo atlikti. Jų išaiškinimas yra labai svarbus, norint šiuos sluoksnius pritaikyti įvairiuose spintronikos prietaisuose.

Darbo tikslas ir uždaviniai. Šio darbo tikslas buvo ištirti polikristalinių $La_{1-x}Sr_xMnO_3$ sluoksnių elektrines savybes magnetiniame ir elektriniame lauke. Šiam tikslui pasiekti buvo suformuluoti tokie uždaviniai: 1. Ištirti plonųjų polikristalinių $La_{1-x}Sr_xMnO_3$ sluoksnių, užaugintų *PI MOCVD* būdu ant polikoro padėklo, elektrines savybes plačiame magnetinių ir elektrinių laukų bei temperatūros ruože. 2. Nustatyti, kaip šios savybės priklauso nuo sluoksnio auginimo temperatūros, legiravimo laipsnio ir sluoksnio storio. 3. Įvertinti mechanizmus, lemiančius šių manganitų elektrines savybes. 4. Išsiaiškinti sluoksnių praktinio pritaikymo galimybes, kuriant magnetinio lauko jutiklius bei sparčiuosius apsaugos nuo elektromagnetinio lauko įtaisus.

Tyrimų metodika. Darbe panaudoti eksperimentiniai metodai, paremti silpnų pastovių magnetinių, stiprių impulsinių magnetinių bei stiprių impulsinių elektrinių laukų generavimo bei matavimo principais.

Mokslinis naujumas. Rengiant disertaciją, buvo gauti šie kietojo kūno fizikos mokslui nauji rezultatai:

1. Ištirta, jog silpnų magnetinių laukų atveju *MR* efektas žemose temperatūrose yra stipresnis, esant žemesnei sluoksnio auginimo temperatūrai (mažiems kristalitams). Nustatyta, kad esant aukštoms temperatūroms ($T>T_m$), *MR* efektas didesnis aukštesnėje temperatūroje augintų sluoksnių atveju. Parodyta, kad $La_{0,83}Sr_{0,17}MnO_3$ sluoksniams silpname magnetiname lauke iki 200 mT sukinio poliarizuotas tuneliavimas nėra vyraujantis krūvio pernašos mechanizmas. Nustatyta, kad silpno lauko magneto varža *LFMR* yra mažesnė negu 15 %.
2. Parodyta, kad magnetovaržos efektą stipriuose magnetiniuose laukuose, polikristaliniuose $La_{0,83}Sr_{0,17}MnO_3$ sluoksniuose, užaugintuose ant polikoro padėklo, galima paaiškinti nagrinėjant krūvio pernašą magnetiškai ir struktūriškai netvarkiose tarpkristalitinėse srityse. Pasiūlyta polikristalinių sluoksnių magnetovaržą ir jos ypatumus temperatūrų ruože tarp T_m ir T_C paaiškinti remiantis modifikuotu Mott‘o šuolinio laidumo modeliu, įskaitant abiejų – feromagnetinės ir paramagnetinės – fazių dalių įnašą.
3. Nustatyta, kad $La_{1-x}Sr_xMnO_3$ polikristalinių sluoksnių atveju *ER* efektas mažėja didinant auginimo temperatūrą (kristalitų dydį) ir sluoksnių legiravimo laipsnį. Įvertinta tarpkristalitinių sričių (*GB*) įtaka *ER* reiškiniui. Nustatyta, kad smulkiakristalitiniuose sluoksniuose, pasižyminčiuose didesne tarpkristalitinių sričių struktūrine ir magnetine netvarka, vyraujantis krūvio pernašos mechanizmas – neelastinis tuneliavimas per tris lokalizuotas būsenas, tuo tarpu sluoksniuose su didesniais kristalitais vyksta neelastinis tuneliavimas per dvi lokalizuotas būsenas.
4. Įvertintas šilimo poveikis polikristaliniams $La_{0,83}Sr_{0,17}MnO_3$ maganitų sluoksniams, atliekant matavimus stipriame kelių dešimčių nanosekundžių trukmės impulsiniame elektriniame lauke. Parodytas skirtingas šilimo pobūdis kristalinėms (*K*) ir tarpkristalitinėms (*GB*) sritims.
5. Parodyta, jog magnetinis laukas gali tiek sumažinti, tiek ir sustiprinti elektrovaržos efektą. Nustatyta, jog esant žemoms temperatūroms, *ER* efektas yra stipriausias esant 4–5 T magnetiniam laukui. Toliau didinant magnetinio lauko stiprį stebimas tik *ER* efekto mažėjimas.

Praktinė reikšmė. Tyrimų rezultatai buvo panaudoti prietaisų prototipų sukūrimui. Buvo pasiūlyti bekontakčio srovės jutiklio, veikiančio kambario temperatūros aplinkoje ir skirto matuoti stiprioms (10–30 A) srovėms bei saugiklio nuo elektromagnetinio impulso, sudaryto iš epitaksinio – polikristalinio sluoksnių sistemos, veikiančio skysto azoto temperatūroje ir turinčio 3,5 dB slopinimą pradiniu laiko momentu bei 8 dB impulso pabaigoje, prototipai. Rezultatai buvo panaudoti tobulinant stipraus impulsinio magnetinio lauko matuoklį. Tai leido sumažinti magnetinio lauko anizotropiją, esant (0–2 T) magnetinės indukcijos ruožui.

Ginamieji teiginiai

1. Silpno ir stipraus magnetinio lauko magnetovaržą polikristaliniuose $La_{1-x}Sr_xMnO_3$ sluoksniuose, užaugintuose ant polikoro padėklo, esant 650–750 °C temperatūroms, nulemia krūvio pernaša netvarkiose tarpkristalitinėse srityse, kurių savybes galima valdyti, keičiant kristalitų dydį. Jį sumažinus, sluoksnių magnetovarža feromagnetinėje fazėje padidėja, tuo tarpu paramagnetinėje fazėje dėl išaugusios kristalitų įtakos sumažėja. Jos ypatumus stipriuose magnetiniuose laukuose galima paaiškinti, remiantis modifikuotu Mott‘o šuolinio laidumo modeliu, įskaitant abiejų fazių įnašą temperatūrų ruože tarp T_m ir T_C.
2. Silpnuose magnetiniuose laukuose magnetovaržos anizotropija yra didžiausia T_m aplinkoje. Ji proporcinga magnetovaržai ir mažėja temperatūrose $T>T_m$, mažinant sluoksnio cheminę sudėtį x, sluoksnio storį ar auginimo temperatūrą. Tuo tarpu stipriuose magnetiniuose laukuose (B>3 T) anizotropijos efektas nepriklauso nuo x ir kristalitų dydžio ir yra nežymus (< 3 %).
3. $La_{1-x}Sr_xMnO_3$ polikristaliniuose sluoksniuose, užaugintuose ant polikoro padėklo ir pasižyminčiuose smulkiais kristalitais, vyrauja neelastinis tuneliavimas tarpkristalitinėse srityse per 3 lokalizuotas būsenas, tuo tarpu didesnių kristalitų sluoksniuose – neelastinis tuneliavimas per 2 lokalizuotas būsenas. Tai lemia didesnę elektrovaržą mažų kristalitų sluoksniuose temperatūrose $T<T_m$, tuo tarpu, kai $T>T_m$, elektrovarža sumažėja dėl padidėjusios kristalitų įtakos.
4. Silpno lauko magnetovaržos ir jos anizotropijos efektai polikristaliniuose $La_{1-x}Sr_xMnO_3$ (*LSMO*) sluoksniuose gali būti panaudoti bekontakčiuose srovės jutikliuose, skirtuose stiprių srovių (10–30 A) matavimui ir veikiančiuose kambario temperatūros aplinkoje. Elektrovaržos efektas šiuose sluoksniuose ir epitaksinių sluoksnių bistabilumas dėl *Joule* kaitimo gali būti panaudoti hibridiniuose apsaugos nuo staigaus elektromagnetinio impulso įtaisuose, suveikiančiuose per subnanosekundes ir atlaikančiuose perkrovas skysto azoto temperatūroje keletą dešimčių nanosekundžių.

Darbo apimtis. Disertaciją sudaro įvadas, šeši skyriai. Pirmame disertacijos skyriuje apžvelgiamos bendrosios manganitų savybės bei magnetovaržos ir elektrovaržos reiškinių prigimtis. Antrajame disertacijos skyriuje aprašoma sluoksnių auginimo technologija bei elektrinių kontaktų gamyba. Trečiajame skyriuje pateikiama matavimo metodikų analizė.

Ketvirtasis skyrius skirtas bandinių morfologijos, mikrostruktūros ir elektrinių savybių tyrimams. Penktajame skyriuje pateikiami gautieji rezultatai, atlikus tyrimus silpname ir stipriame magnetiniuose laukuose. Šeštasis skyrius skirtas rezultatų apžvalgai, gautai atlikus tyrimus stipriame elektriniame lauke. Darbo pabaigoje pateiktos bendrosios išvados, rezultatų apibendrinimas, autoriaus publikacijų disertacijos tema sąrašas ir cituojamos literatūros sąrašas.

Darbo apimtis yra 112 puslapių, tekste panaudotos 45 numeruotos formulės, 120 paveikslų ir 1 lentelė.

Bendrosios išvados

1. Buvo nustatyta, kad mangano oksidų $La_{1-x}Sr_xMnO_3$ sluoksniai, užauginti *PI MOCVD* būdu ant polikoro padėklo 650–750 °C temperatūrose yra polikristaliniai, o 825 °C padėklo temperatūroje pasižymi tekstūruota struktūra. Mažinant auginimo temperatūrą T_{pad} arba sluoksnių storį, kristalitų dydis sluoksniuose mažėja. Mažinant T_{pad}, sluoksnio storį arba cheminę sudėtį x, maksimalios varžos temperatūra T_m slenka į žemesnių temperatūrų sritį, o savitoji varža ρ_m išauga.
2. Plonųjų polikristalinių $La_{1-x}Sr_xMnO_3$ sluoksnių, užaugintų ant polikoro padėklo, magnetovaržos dydį galima keisti keičiant jų cheminę sudėtį x, padėklo temperatūrą T_{pad} auginimo metu bei sluoksnio storį. Magnetovaržos absoliutinė vertė feromagnetinėje fazėje didėja, mažinant T_{pad}, x bei sluoksnio storį. Temperatūrose $T>T_m$, *MR* dėl išaugusios kristalitų įtakos sumažėja.
3. Tyrimai silpnuose magnetiniuose laukuose ($B<1$ T) parodė, kad magnetovaržos anizotropija *MRA* yra didžiausia T_m aplinkoje ir didėja, didinant sluoksnių x, kristalitų dydį ir sluoksnio storį temperatūrose $T>T_m$. Stipriuose magnetiniuose laukuose ($B>3$ T) anizotropijos efektas nepriklauso nuo x bei kristalitų dydžio ir yra pakankamai mažas ($MRA<3$ %).
4. Polikristalinių sluoksnių magnetovaržą galima aprašyti panaudojant modifikuotą Mott‘o šuolinio laidumo modelį, kuriame tarpkristalitinės sritys nagrinėjamos kaip mezoskopinės sritys su mažesne magnetine tvarka nei kristalitai. Magnetovaržos priklausomybę nuo magnetinio lauko temperatūrų ruože tarp T_m ir T_C galima aprašyti įskaitant abiejų – feromagnetinės ir paramagnetinės – fazių dalių įnašą. Poliarono dydis temperatūrose $T<T_m$ didėja eksponentiškai su temperatūra ir apima maždaug 10 Mn jonų arti T_m.
5. Nustatyta, kad elektrovaržos dydis polikristaliniuose $La_{1-x}Sr_xMnO_3$ sluoksniuose priklauso nuo jų cheminės sudėties x, kristalitų skaičiaus bei dydžio, maksimalios varžos temperatūros T_m. Feromagnetinėje fazėje

elektrovarža yra didžiausia mažiausių kristalitų sluoksniuose, kuriuose yra didesnis netvarkių tarpkristalitinių sričių kiekis. Kambario temperatūroje didžiausia elektrovarža stebima didelių kristalitų atveju. Elektrovaržos slenkstinis elektrinis laukas yra didesnis mažų kristalitų sluoksniams. Krūvio pernašą stipriuose elektriniuose laukuose galima aprašyti Glazmano-Matvejevo modeliu. Sluoksniuose iš smulkių kristalitų vyrauja neelastinis tuneliavimas tarpkristalitinėse srityse per 3 lokalizuotas būsenas, tuo tarpu sluoksniuose iš didesnių kristalitų – neelastinis tuneliavimas per 2 lokalizuotas būsenas.

6. Rasta, kad didėjant elektrinio lauko stipriui, T_m slenkasi į aukštesnių temperatūrų sritį. Ultrastipriuose (>70 kV/cm) kelių dešimčių ns trukmės elektriniuose laukuose stebimas sluoksnio kaitimas dėl *Joule* šilumos. Žemiau T_C feromagnetiniai kristalitai ir paramagnetinės tarpkristalitinės sritys kaista nevienodai.
7. Atlikti eksperimentai parodė, kad tam tikromis sąlygomis (temperatūroje $T<T_m$ smulkiakristaliniuose sluoksniuose) elektrovaržos efektą galima sustiprinti magnetiniu lauku. Aukštose temperatūrose $T \geq T_m$ ir stambių kristalitų sluoksniuose visame matuojamų temperatūrų ruože magnetinis laukas sumažina elektrovaržos efektą.
8. Naudojant polikristalinius $La_{1-x}Sr_xMnO_3$ sluoksnius buvo sukurtas bekontaktis srovės jutiklis, veikiantis kambario temperatūros aplinkoje, skirtas stiprių srovių (10–30A) matavimui. Naudojant polikristalinius ir epitaksinius $La_{1-x}Sr_xMnO_3$ sluoksnius sukurtas hibridinis apsaugos nuo staigaus elektromagnetinio impulso įtaisas, suveikiantis per subnanosekundes ir atlaikantis perkrovas skysto azoto temperatūroje keletą dešimčių ns.

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MAGNETIC AND ELECTRICAL FIELD EFFECTS IN POLYCRYSTALLINE $La_{1-x}Sr_xMnO_3$ FILMS

Summary of Doctoral Dissertation
Physical Sciences, Physics (02P), Condensed Matter: electronic structure; electrical, magnetic and optical properties; superconductors; magnetic resonance; relaxation; spectroscopy (P260)

MAGNETINIO IR ELEKTRINIO LAUKO EFEKTAI POLIKRISTALINIUOSE $La_{1-x}Sr_xMnO_3$ SLUOKSNIUOSE

Daktaro disertacijos santrauka
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