

The Application of Pulsed Magnets for Investigations of Electrical Properties of Semiconductors and Manganites

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Introduction

The advance in electrical engineering and electronics has demanded the application of new materials in device design. Problems of the testing electrical properties of new materials are very topical due to their practical importance and scientific interest. Therefore measurements of electrical properties of semiconductors and other materials were developed in the same pace as electronics to guarantee the technical support of investigations in physics and technological sciences.

One of main electrical parameters is specific resistance. Two or four probe method or their modified analogues are used to test mentioned parameters due to simplicity of their realization. Two or four contacts are formed at the surface of investigated specimens and a voltage drop is measured [1]. This contact methods are destructive because they require electrical contacts at the surface of tested specimen. Moreover these methods are not applied for express measurements.

High frequency methods are used for non-contact and express measurements. Investigated specimen is placed into inductance coil or measuring capacitor and the change of an amplitude and phase of high frequency current can be registered. Resonators or waveguide line are applied in super high frequency range where a tested sample is placed at a measuring aperture of measuring device and a change of Quality-factor or an impedance is registered. The value of specific resistance is calculated according to standard curves. More complete information about properties of semiconductors can be provided by measurements of concentration and mobility of free charge carriers of semiconductors. These measurements require a high frequency generator and magnetic field source [2].

The physical properties of polycrystalline manganites, single crystals and thin films were widely investigated. The electrical and magnetic properties of thin films are of special interest due to their potential application for the development of spin-electronics devices, such as vertical tunnel junctions, nanostructured devices and magnetic field sensors. It is very important to guide technological processes during preparation of thin films in order to

obtain samples with special requirements. The electrical and magnetic properties of manganite films are closely related to their composition, crystallinity and epitaxy and characteristics strongly depend on the deposition conditions [3]. The films prepared for pulsed magnetic field applications have to satisfy several requirements like no magnetoresistance saturation at high magnetic fields, high-speed responsiveness, and sensitivity independence to the direction of magnetic field.

For material investigations magnetic field is used as one of most powerful facility. The resolution and sensitivity of measurements in material science experiments is closely limited by the maximal available magnetic field. As higher magnetic field is available as wider ranges of measurements can be achieved [4].

Electromagnets with iron core are classic and applicable in most scientific laboratories. The peak field is determined by the saturation of the material magnetisation and for most application it does not exceed 2 Tesla limit. For the magnetic field generation exceeded iron magnetisation limit solenoids without cores were designed. These magnets are powered with several megawatts power sources. Power consumption can be reduced by superconducting material application. Hybrid magnetic systems including superconducting and non-superconducting magnets in the same system can be used for the measurements in constant magnetic fields up to 40-45 T. But such facilities are unique and expensive.

Pulsed technology is a very attractive alternative way to generate high magnetic fields. Pulsed magnetic field application in measurement science has long time traditions. Basic apparatus for generating of pulsed magnetic fields are compact and not such expensive as constant magnetic field facilities.

Recently the interest for pulsed magnetic field facilities increased very much and compact non-destructive equipment is under constant development [5].

In present article compact high magnetic field facilities used for investigation of electrical properties of semiconductors and manganites are described and measurements of magnetoresistance, mobility and concentration of free charge carriers are offered.

Measurements of concentration and mobility of free charge carriers in CdHgTe and InSb samples

The application of magnetoplasma effects in semiconductors to measure electrical properties of semiconductors is known. If semiconductor specimen is placed in external magnetic field high frequency magnetoplasma waves can be excited and propagate along the direction of magnetic induction.

The propagation of magnetoplasma wave in semiconductor is determined by dispersion equation

$$\frac{k^2 c^2}{\omega^2} = \varepsilon'_\pm + i\varepsilon''_\pm = \varepsilon_l \left(1 - \frac{\omega_p}{\omega[(\omega \pm \omega_c) + iv]} \right), \quad (1)$$

where k – propagation constant; c – light velocity in vacuum; $\varepsilon'_\pm, \varepsilon''_\pm$ – the real and imaginary parts of the complex relative dielectric constant in the medium respectively; ω – exciting frequency; ε_l – lattice constant; $\omega_c = qB/m^*$ – frequency of cyclotron resonance; $\nu = 1/\tau$ – frequency of carriers collisions; $\omega_p = (e^2 n/m^* \varepsilon_0 \varepsilon_l)^{1/2}$ – plasma frequency. The subscripts (+) and (-) refer the ordinary and extraordinary waves.

For extraordinary wave the real and imaginary parts of complex relative dielectric constant can be written as

If the semiconductor has one type of free charge carriers and an isotropic effective mass, constant relaxation time the real and imaginary parts of the complex relative dielectric constant for extraordinary wave can be expressed as following:

$$\varepsilon' = \varepsilon_l \left(1 + \frac{\omega_p^2}{\omega_c \omega} \right) = \varepsilon_l \left(1 + \frac{en}{\varepsilon_0 \varepsilon_l \omega B} \right),$$

$$\varepsilon'' = \frac{\varepsilon_l \nu \omega_p^2}{\omega \omega_c^2} = \frac{nm^* \nu}{\varepsilon_0 \omega B^2}. \quad (2)$$

These equations are applied for lossless conditions, when magnetoplasma waves can propagate in a semiconductor

$$\omega \pm \omega_c \gg \nu; \omega_c \gg \omega; \omega_c \tau \equiv \mu B \gg 1. \quad (3)$$

The wave length of magnetoplasma wave is determined by equation

$$\lambda = \frac{2\pi}{k'} = \frac{c}{\omega \sqrt{\varepsilon'}} = \frac{c}{\omega} \left(\frac{\varepsilon_0 \omega B}{en} \right)^{1/2}. \quad (4)$$

The amplitude of magnetoplasma wave can be expressed as

$$A = A_0 e^{-k''d}, \quad k'' = \frac{\omega \varepsilon''}{2c \sqrt{\varepsilon'}}, \quad (5)$$

where $\varepsilon', \varepsilon'', k', k''$ – real, imaginary parts of relative dielectric constant and a wave propagating constant

respectively. Thus by observation of geometrical resonance of magnetoplasma waves in a semiconductor specimen with known thickness d it is possible to determine the concentration and mobility of free charge carriers of semiconductors [6]. Experimentally resonance curves of magnetoplasma waves can be observed with high frequency Rayleigh interferometer which structure is shown in Fig. 1.

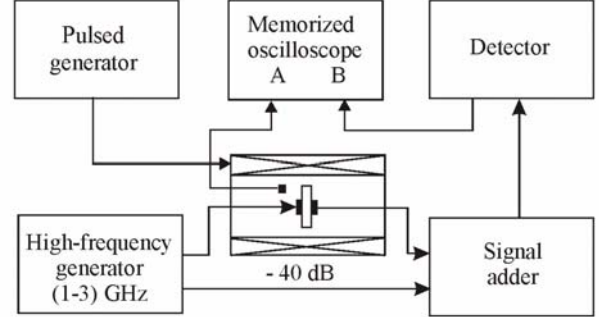


Fig. 1. Structure of magnetoplasma interferometer with pulsed magnetic field source

Tested semiconductor sample is placed in the centre of pulsed magnet. HF signal from the generator connected with exiting antenna excites magnetoplasma wave in the sample. Propagated through the semiconductor sample magnetoplasma wave is registered by receiving antenna. Received signal interferes with a reference signal. Obtained interferogram is registered by memorized oscilloscope. Magnetic field is controlled with inductive probe. Semiconductor samples were investigated at room and liquid nitrogen temperatures. Some experimental results are put in Table 1.

Table 1 Obtained values of concentration and mobility of free charge carriers in CdHgTe and InSb

Material	Concentration, 10^{24} m^{-3}		Mobility, $\text{m}^2 \text{ V}^{-1} \text{ s}^{-1}$	
	passport	obtained	passport	obtained
CdHgTe	0,12	0,12	23	21
CdHgTe	1,2	1,25	17	16
CdHgTe	13,0	12,5	0,19	0,17
CdHgTe	105	110	0,12	0,11
InSb	1,5	1,4	23	21
InSb	0,2	0,21	70	65

High frequency magnetoplasma interferometer with pulsed magnetic field source up to 40 T provides non-contact measurements of concentration in $(10^{20} \text{ to } 10^{24}) \text{ m}^{-3}$ range and mobility in $(0,5 \text{ to } 100) \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1}$ range respectively with relative error $\pm 15 \%$ for concentration and $\pm 25 \%$ for mobility.

Magnetoresistance measurements of BiSb and LaCaMnO samples by two electrode method

The magnetoresistance of semiconductor and manganites thin films have be investigated by two electrodes method in pulsed magnetic field up to 45 T.

Investigated sample was placed inside of pulsed coil and connected in series with standard resistor and stabilized direct current source. The value of magnetoresistance R_B can be determined as

$$R_B = \frac{U_B}{I} = \frac{U_{DC} - U_0}{U_0} R_0, \quad (6)$$

where U_{DC} – a voltage of direct current source, U_0 – a voltage drop measured by the oscilloscope, R_0 – resistance of standard resistor. The structure of experimental equipment is shown in Fig. 2.

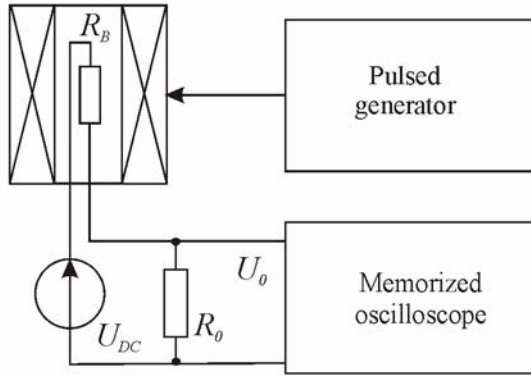


Fig. 2. Structure of experimental equipment for magnetoresistance measurements by two electrode method

Polycrystalline BiSb films were prepared on flat fibreglass substrates by thermal evaporation and investigated at temperature of 77 K. The magnetic field direction was parallel to the fibreglass substrate surface and the bias current passing through the sample. Films prepared by different thermal annealing techniques and having different grain structures were investigated. Longitudinal magnetoresistance of microcrystallites is determined by the diagonal components of the resistivity tensors ρ_{11} and ρ_{22} which show different behaviour in magnetic fields. Since the thickness of these films is approximately equal to the microcrystalline grain thickness, the resistivity ρ of these films can be simulated as a series of resistances like $\rho_{11}l_1^{-1}$, $\rho_{22}l_1^{-1}$, $\rho_{22}l_b^{-1}$ and thus

$$\rho = \frac{l_1[\rho_{11} + \rho_{22}]}{2l_1 + l_2} + \frac{l_2\rho_b}{2l_1 + l_2}, \quad (7)$$

where ρ_b – the resistivity of the grain boundary. If the grain size l_1 is larger than the grain boundary thickness l_2 resistivity can be expressed like

$$\rho = \frac{\rho_{11} + \rho_{22}}{2} + \frac{l_2\rho_b}{2l_1}. \quad (8)$$

Typical relationships of relative variations of magnetoresistance of BiSb films in the quantizing magnetic field region up to 50 T are shown in Fig. 3 [7].

A negative differential magnetoresistance of BiSb polycrystalline thin films has taken place. This is a result the affecting the energy band structure of microcrystallites by quantizing magnetic field. Moreover the shift of

magnetoresistance maximum can be applied to control the film composition.

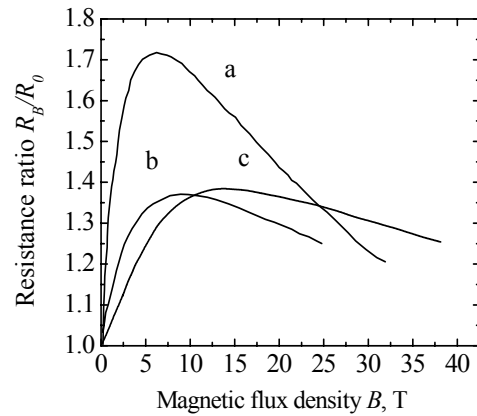


Fig. 3. Magnetoresistance of BiSb thin films in quantizing magnetic field B: a- calculated for annealed sample, b- calculated for unannealed film, c- experimental results for annealed film

The magnetoresistance of polycrystalline and epitaxial manganite thin films was studied in high pulsed magnetic fields up to 35 T [8]. A polycrystalline LaCaMnO films were grown on a lucalox substrate using a pulsed deposition technique. The thickness of the films was 400 nm. The films exhibited a non-textured single-phase polycrystalline structure with 50 nm size grains. Electrical contacts, spaced by 20 μ m gaps, were made by thermal deposition of Ag, using a Cr sub-layer. Tested samples were connected with output circuits with bifilar twisted wires and placed in central area of the pulsed coil connected with pulsed power supply. Discharging the capacitor bank through the pulsed coil a sinus shaped magnetic field pulse was generated. For transport measurements voltage from stabilized DC power supply was applied to in series connected manganite films and ballast resistors. The response of manganite films to magnetic pulse was registered with memorized oscilloscopes. Magnetoresistance of tested samples were measured at different temperatures and is shown in Fig. 4.

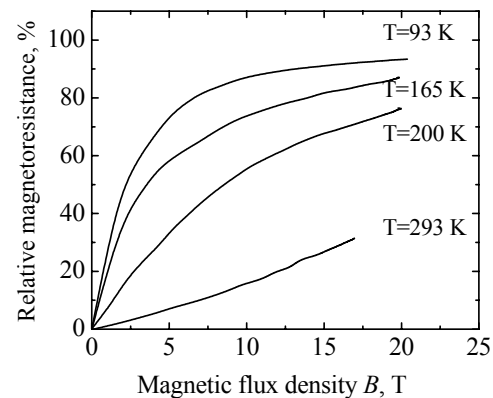


Fig. 4. Relative magnetoresistance of polycrystalline LaCaMnO films at different temperatures

With the change of the temperature the curves could have different character from non-linear at low temperature

to sub-linear or linear at temperatures close to room condition. Therefore such films can be used for magnetic field measurements. The typical results of output voltage responses to magnetic field of epitaxial and polycrystalline $\text{La}_{0.33}\text{Ca}_{0.67}\text{MnO}_3$ films are shown in Fig. 5.

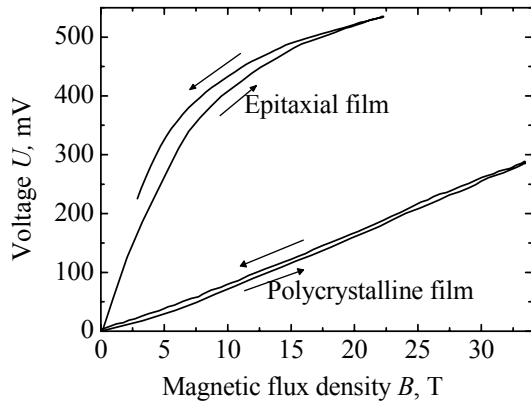


Fig. 5. Response of $\text{La}_{0.33}\text{Ca}_{0.67}\text{MnO}_3$ films to magnetic field pulse

For epitaxial film the ΔU measurements showed high sensitivity in the magnetic field range up to 7 T and lower sensitivity at higher fields. The obtained “memory” effect in these films implies that when magnetic field is decreasing, the resistance of the film remains lower (ΔU on R_0 higher) than at increasing field. This behaviour could be explained by colossal magnetoresistance (CMR) phenomenon in manganites caused by phase transition from paramagnetic to ferromagnetic state. It should be noted, that sensors used for measurements of rapidly changing in time magnetic field have to satisfy several requirements, the main of which are the following: no saturation at high magnetic fields, high-speed responsiveness, and sensitivity independence to the direction of magnetic field. Therefore, in spite of high sensitivity, the dependence of output voltage on magnetic field of epitaxial films exhibiting “memory” effect and saturation at relatively small magnetic fields is disadvantage for pulsed magnetic field measurements. The considerably smaller sensitivity of resistance to the magnetic field is observed for polycrystalline samples. The magnetic “memory” effects in this case are negligible and voltage ΔU measured across the resistor R_0 increases almost linearly up to 33 T. Sensors based on polycrystalline manganite films are able to record absolute values of magnetic field, independent on the field orientation relative to the sensor’s substrate plane and bias current direction [9]. The obtained results can be explained by taking into consideration polycrystalline structure of $\text{La}_{0.33}\text{Ca}_{0.67}\text{MnO}_3$ film, which consists of grain boundaries (GB) between crystallites surrounded by mesoscopic regions. Investigation of electrical properties of semiconductors and manganites were done using pulsed magnetic field generator. Generally pulsed magnetic field source is one of most important facility which limits ranges of measurements and errors of measuring data. Below pulsed magnetic field generator construction is described.

Pulsed magnetic field generator

Pulsed magnetic fields commonly used in material science measurements are generated by the capacitor bank discharge through inductive coil. The view of pulsed magnetic field generator developed in Vilnius High Magnetic Field Centre is shown in Fig. 6.



Fig. 6. Pulsed magnetic field generator up to 50 T with control and data registration devices

Pulsed generator consists of the control unit, high voltage power supply, capacitor bank of 5mF, thyristor switch, pulsed inductor and memorized oscilloscope. The capacitor bank is able to store 62,5 kJ electric energy. Discharging the capacitor bank through the inductive coil electric energy is transformed into magnetic energy and a sinus shaped magnetic field pulse with maximal amplitude up to 50 T and (1-2) ms duration can be generated.

High magnetic field pulses were generated using single turn inductors, flux concentrators, helix inductors, pulsed coils and multi-section inductors. Wire wound coils were chosen as most acceptable to construct pulsed magnets with long pulse duration and good field homogeneity.

Magnetic flux density in any inside point of the solenoid was evaluated numerically using following equations:

$$\begin{cases} B_z(\rho, \theta) = B \left[1 + E_2 \left(\frac{\rho}{r} \right)^2 P_2(u) + E_4 \left(\frac{\rho}{r} \right)^4 P_4(u) + \dots \right], \\ B_r(\rho, \theta) = B \left[0 + E_2 \left(\frac{\rho}{r} \right)^2 P_2'(u) + E_4 \left(\frac{\rho}{r} \right)^4 P_4'(u) + \dots \right], \end{cases} \quad (9)$$

where $B_z(\rho, \theta)$, $B_r(\rho, \theta)$ – axial and radial components of magnetic field in the point with spherical coordinates r, θ ; $P_n(u)$, $P_n'(u)$ – Legendre polynomial and its derivative, respectively. $u = \cos \theta$, E_n – coefficients for partial field derivative in point $z = 0$ determined by Taylor’s formula:

$$E_{2n} = \frac{1}{B_0} \frac{1}{(2n)!} \left. \frac{\partial^{2n} B_z(z, 0)}{\partial z^{2n}} \right|_{z=0}, \quad (10)$$

$$B_0 = \mu_0 \frac{NI}{r} \left[\frac{F(\alpha, \beta)}{2\beta(\alpha - 1)} \right], \quad (11)$$

$$F(\alpha, \beta) = \beta \ln \frac{\alpha + \sqrt{\alpha^2 + \beta^2}}{1 + \sqrt{1 + \beta^2}} \quad (12)$$

here μ_0 is magnetic constant, N is the quantity of turns, I is a current, r_1 , r_2 , l are internal, external radius and the length of a solenoid, respectively, $\alpha = r_1/r_2$, $\beta = l/2r_1$ are relative sizes.

The distribution of axial magnetic field is shown in Fig.7

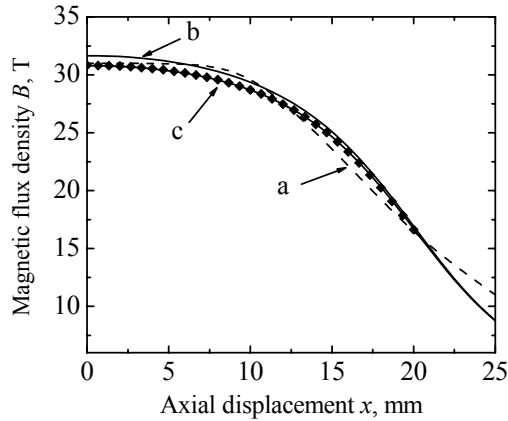


Fig. 7. Axial magnetic field measured by different ways: a – La-Ca-MnO sensor, b – pick up inductive coil, c – numerical simulation

Magnetic field measurements of pulsed solenoids were executed at room and liquid nitrogen temperatures. Computing results were verified by axial magnetic field measurements using pick-up inducting coil, current shunt and manganite sensor [10]. Strong coupling of mechanical and thermal loads takes place in the pulsed coil construction during discharge. The magnet is comparable to a high-pressure adiabatically heated vessel. Therefore the high mechanical strength of the materials is required to resist the Lorentz forces and the high conductivity is required to decrease Joule heating. Both parameters are most limited factors because the increase in magnetic field is accompanied by the quadratic increase in mechanical and thermal loads. To insure a long life operation of pulsed magnets a complex analysis of electrical, thermal and mechanical overloads was done [11]. Numerical analysis performed by the finite element method simulated transient behaviour of magnetic fluxes, induced heat and thermal fields in the pulsed generator. The non-linear thermal analysis was performed considering temperature dependent specific heat and conductivity. Numerical behaviour of magnetic fields was validated by the comparison with experimental measurements, while thermal analysis serves for prediction of the temperature dependent material properties. The measurements of mechanical properties of electrotechnical materials applied in pulsed magnet design were done to clear the influence of cyclic overloads on pulsed magnet reliability. Mechanical properties were measured experimentally and the influence of cyclic cooling and heating on material mechanical properties was investigated also. Composite materials with the high electric and mechanical characteristics were applied in

pulsed inductor design [12]. A metal-matrix Cu-Nb microcomposite rectangular cross-section conductor 1.70 mm x 0.80 mm having the high electric conductivity (65 % IACS) and the mechanical strength (UTS = 1.2 GPa) was wet impregnated, reinforced with Zylon and it put in external steel cylinder. The thickness of interlayer reinforcement was about 1 mm. The view of assembled construction is shown in Fig.8



Fig. 8. Pulsed magnet construction

Pulsed magnet construction were pre-cooled with liquid nitrogen and tested to insure the non-destructive operation. A maximal magnetic field of 50 T were achieved without construction disintegration.

Conclusions

The application of pulsed magnet in high frequency interferometer enabled to expand ranges of measurements of concentration and mobility of free charge carriers in semiconductors. InSb, CdHgTe samples were investigated at room and liquid nitrogen temperature. Magnetoresistance of epitaxial and polycrystalline films was measured by two electrodes method. The resistance response to high magnetic field pulses up to 50 T was studied for thin films of La-Ca-MnO, BiSb with different magnetic and electric properties. The obtained results showed that polycrystalline La-Ca-MnO films have several advantages in comparison with epitaxial ones for the development of pulsed magnetic field sensors. In spite of lower magnetoresistance sensitivity, polycrystalline films have no magnetoresistance saturation effects in high magnetic fields and can be successfully used to measure absolute value of the fields up to 35 T.

The compact system for high magnetic field generation up to 50 T was developed. Numerical analysis performed by the finite element method simulated transient behaviour of magnetic fluxes, induced heat and thermal fields in the pulsed generator was done. New constructing materials as polyphenylene-benzobisoxazole fibres, metal-matrix Cu-Nb microcomposite were applied in pulsed coil construction. Magnetic field measurements of pulsed solenoids were executed at room and liquid nitrogen temperatures using different methods. Axial magnetic field was estimated using analytical and numerical methods and acceptable agreement between calculated and measured results was achieved.

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The system for high magnetic field generation up to 50 T was developed and applied for measurements of resistance, concentration and mobility of free charge carriers of semiconductors and manganites. High frequency interferometer had expanded ranges of measurements and specimens of InSb, CdHgTe samples were investigated at room and liquid nitrogen temperature. Magnetoresistance of epitaxial and polycrystalline films was measured by two electrodes method. The resistance response to high magnetic field pulses up to 50 T was studied for thin films of LaCaMnO, Bi, BiSb with different magnetic and electric properties. Polycrystalline La-Ca-MnO films have no saturation effects in high magnetic fields and can be used to measure absolute value of the fields up to 35 T. Axial magnetic field was estimated by analytical and numerical methods and verified experimentally. Numerical analysis of pulsed magnets was performed by the finite element method. New constructing materials were applied in pulsed magnet design and multilayer construction of metal-matrix Cu-Nb micro composite wire wounded Zylon-epoxy composite reinforced inductor was developed. Ill. 8, bibl. 12 (in English, summaries in English, Russian and Lithuanian).

Ю. Новицкий. Применение импульсных магнитов в исследованиях электрических свойств полупроводниковых материалов и манганитов // Электроника и электротехника. – Каунас: Технология, 2007. – № 6(78). – С. 29–34.

Созданный источник импульсного магнитного поля использовался для исследования электрических свойств полупроводниковых материалов и манганитов. Концентрация и подвижность свободных носителей заряда в InSb, CdHgTe полупроводниках определялась с помощью высокочастотного магнетоплазменного интерферометра в импульсных полях по 40 Т. Зависимость удельного сопротивления эпитаксиальных и поликристаллических LaCaMnO, Bi, BiSb образцов исследовалась по двухзондовой методике в импульсных полях порядка 35 Т. Установлено, что магнитосопротивление эпитаксиальных LaCaMnO образцов анизотропно, а поликристаллических – изотропно. В BiSb образцах наблюдается отрицательное изменение дифференциального сопротивления в сильных магнитных полях. Функционирование импульсных электромагнитов анализировалась путем моделирования процессов методом конечных элементов с последующей экспериментальной проверкой результатов. Для увеличения максимального значения генерируемого магнитного поля использовался микрокомпозиционный Cu-Nb проводник с многослойной изоляцией из Zylon волокна, пропитанной эпоксидным компаундом. Ил. 8, библи. 12 (на английском языке; рефераты на английском, русском и литовском яз.).

J. Novickij. Impulsinių magnetų taikymas puslaidininkinių medžiagų ir manganitų elektrinėms savybėms tirti // Elektronika ir elektrotechnika. – Kaunas: Technologija, 2007. – Nr. 6(78). – P. 29–34.

Impulsinio magnetinio lauko generatorius iki 50 T buvo panaudotas puslaidininkinių medžiagų bei manganitų elektrinėms savybėms tirti. Aukštadažnių magnetoplazminių interferometrų atlikti InSb, CdHgTe bandinių elektronų koncentracijos ir judrio matavimai kambario ir skystojo azoto temperatūroje impulsiniuose magnetiniuose laukuose iki 40 T. Taikant dvejų zondu metodiką, atlikti epitaksinių ir polikristalinių LaCaMnO sluoksnių bei Bi ir BiSb polikristalinių sluoksnių magnetovaržos tyrimai. Nustatyta, kad epitaksiniai manganitų sluoksniai pasižymi anizotropiniu neigiamos diferencialinės magnetovaržos ir „magnetinės atminties“ efektu. Polikristaliniuose sluoksniuose šie efektai yra izotropiniai. Bismuto ir jo junginių sluoksniuose neigiamos diferencialinės magnetovaržos reiškinys pasireiškia skystojo azoto temperatūroje magnetiniuose laukuose per 35 T. Baigtinių elementų metodu atlikta impulsinių magnetų lauko pasiskirstymo, mechaninių perkrovų bei deformacijų analizė. Siekiant padidinti generuojamo magnetinio lauko impulso maksimalią vertę, induktoriams gaminti panaudotas mikrokompozicinės medžiagos. Il. 8, bibl. 12 (anglų kalba; santraukos anglų, rusų ir lietuvių k.).