

SCIENTIFIC PROGRAM

ORAL SESSIONS

SECTION A

PROCESSING OF OXIDE FILMS AND MULTILAYERS

SESSION 1

THURSDAY, 28.09.2000, 9³⁰ - 12⁰⁰

Chairman Dr. Albert FIGUERAS

- O1A. **Georg WAHL** (invited), *Institut für Oberflächentechnik und Plasmatechnische Werkstoffentwicklung, Technische Universität Braunschweig, Germany*
INDUSTRIAL SCALE PROCESSING OF OXIDE LAYERS AND MULTILAYERS
- O2A. **Jean-Pierre SÉNATEUR** (invited), *LMGP, ENS de Physique de Grenoble, France*
APPLICATION OF PULSED-INJECTION MOCVD TO THE SYNTHESIS OF MULTILAYERS AND SUPERLATTICES AT NANOMETRIC SCALE
- O3A. **Johannes LINDNER**, *AIXTRON AG, Aachen, Germany*
INDUSTRIAL SCALE MOCVD PROCESSING OF ELECTROCERAMIC THIN FILMS
- O4A. **Catherine DUBOURDIEU**, *LMGP, ENS de Physique de Grenoble, France*
PULSED LIQUID-INJECTION MOCVD OF MANGANITE – BASED SUPERLATTICES
- O5A. **Arunas TEISERSKIS**, *Vilnius University, Lithuania*
LARGE AREA YBCO FILMS DEPOSITED BY PULSED INJECTION CVD

SESSION 2

THURSDAY, 28.09.2000, 14³⁰ - 16⁰⁵

Chairman Dr. Francois WEISS

- O6A. **Andrej KAUL** (invited), *Moscow State University, Russia*
EPITAXIAL PHASE STABILISATION PHENOMENA IN THIN FILM GROWTH
- O7A **Jose SANTISO**, *ICMAB-CSIC, Barcelona, Spain.*
COMPOSITION DEPENDENCE OF THIN FILM GROWTH CHARACTERISTICS IN COMPLEX OXIDES.
- O8A. **Mazhar BARI**, *Trinity College, Dublin 2, Ireland*
THIN FILMS OF Sr₂FeMoO₆ DEPOSITED BY PULSED LASER DEPOSITION
- O9A **Ahti NIILISK**, *University of Tartu, Estonia*
TiO₂ THIN FILMS BY ATOMIC_LAYER CHEMICAL VAPOUR DEPOSITION: GROWTH AT 365 °C

SECTION B

**CHARACTERISATION AND APPLICATION OF OXIDE FILMS AND
MULTILAYERS**

SESSION 3

FRIDAY, 29.09.2000, 9⁰⁰ – 10³⁵

Chairman Prof. Michael COEY

- O1B. **Josep FONTCUBERTA** (Invited), *Institut de Ciència de Materials de Barcelona, Spain*
Sr₂FeMoO₆ DOUBLE PEROVSKITES. ARE THEY ALTERNATIVE MATERIALS FOR SPIN DEVICES?
- O2B. **Oleg GORBENKO**, *Moscow State University, Russia*
NON-DESTRUCTIVE CHARACTERIZATION OF EPITAXIAL PEROVSKITE LAYERS BY RAMAN SPECTROSCOPY
- O3B. **Bonifacas VENGALIS**, *Semiconductor Physics Institute, Vilnius, Lithuania*
SPIN-POLARIZED QUASIPARTICLE INJECTION DEVICE BASED ON MOCVD-GROWN YBa₂Cu₃O₇/SrTiO₃/La_{1-x}Sr_xMnO₃ HETEROSTRUCTURES
- O4B. **Karol FRÖHLICH**, *Slovak Academy of Sciences, Bratislava, Slovak Republic*
NON-LINEAR CURRENT-VOLTAGE CHARACTERISTICS OF La_{1-x}MnO₃ FILMS.

SESSION 4

FRIDAY, 29.09.2000, 10⁵⁵ – 12³⁵

Chairman Dr. Karol FRÖHLICH

- O5B. **Piotr DŁUŻEWSKI**, *Polish Academy of Sciences, Warsaw, Poland*
TEM AND X-RAY CHARACTERIZATION OF MICROSTRUCTURE OF YBaCuO THIN FILMS DEPOSITED BY LASER ABLATION ON COLD SUBSTRATES
- O6B. **A. GALDIKAS**, *Kaunas University of Technology, Lithuania*
ION BEAM DEPTH PROFILING OF MULTILAYERS
- O7B. **Vladimir IGNATOVICH**, *FLNP JINR, Dubna, Moscow region, Russia*
MULTILAYERED SYSTEM WITH FORBIDDEN REFLECTIONS
- O8B. **Sigitas TAMULEVICIUS**, *Kaunas University of Technology, Lithuania*
OPTICAL MEASUREMENTS OF STRAIN AND STRESS IN THIN FILMS
- O9B. **Vladimir POKROPIVNY**, *Institute for Problems of Materials Science of NASU, Kiev, Ukraine*
NANOTUBULAR 2D CRYSTALS. SYNTHESIS AND PROMISING APPLICATIONS.

POSTER SESSION

THURSDAY, 28.09.2000, 16¹⁵ - 18⁰⁰

SECTION A

- P1A. **C. Jiménez^a, F. Weiss^a, J. P. Senateur^a, A. Abrutis^b, A. Teiserskis^b, O. Stadel^c, J. Schmidt^c, G. Wahl^c, M. Krellmann^d, D. Selbmann^d, N.V. Markov^e, S. V. Samoylenkov^e, O. Yu. Gorbenko^e, A. R. Kaul^e, F. Fillot^f, H. Guillon^f.**

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DEPOSITION OF YBCO BY MOCVD FOR COATED CONDUCTORS
FABRICATION

- P2A. **L.Fàbrega¹, M. Caussanel, R.Rubi¹, J.Fontcuberta¹, V.Trtík², F.Sánchez², C.Ferrater², M. V. García-Cuenca² and M.Varela²**

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FERROMAGNETIC AND SUPERCONDUCTING OXIDE HETEROSTRUCTURES
FOR SPIN INJECTION DEVICES

- P3A. **A.A.Bosak^{*1}, S.V.Samoilenkov¹, O.Yu.Gorbenko¹, A.N.Botev¹, A.R.Kaul¹, J.-P. Sénateur², C.Dubourdieu²**

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SELF-TUNING APPROACH TO THE MOCVD OF HIGH-QUALITY LEAD-
CONTAINING HETEROSTRUCTURES

- P4A. **B. Vengalis, K. Oginskas, V. Lisauskas, R. Butkute, A. Maneikis, L. Dapkus, V. Jasutis and N. Shiktorov**

Semiconductor Physics Institute, A.Goštauto 11, LT-2600 Vilnius, Lithuania,
GROWTH AND INVESTIGATION OF THE (LaNiO₃, RuO₂)/La_{1-x}Ca_xMnO₃
HETEROSTRUCTURES

- P5A. **V. Plausinaitienė^{1,2}, A. Abrutis¹, B. Vengalis², R. Butkute², J.P. Senateur³, Z. Saltyte¹, V. Kubilius¹, S.Pasko¹, L.Dapkus²**

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MOCVD GROWTH AND CHARACTERISATION OF La_{1-x}Sr_xMnO₃/SrTiO₃/
La_{1-x}Sr_xMnO₃ HETEROSTRUCTURES

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OXIDE THIN FILM DEPOSITION ON EUTECTIC SUBSTRATES

P7A. P. B. Tavares

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THE EFFECTS OF TEMPERATURE ON BISRCACUO THIN FILMS USING AEROSOL-ASSISTED METALORGANIC CHEMICAL VAPOR DEPOSITION (MOCVD).

P8A. A. Teiserskis, A. Abrutis, Z. Saltyte

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MOCVD DEPOSITION OF YBCO FILMS ON BIAXIALLY TEXTURED Ag SUBSTRATES

P9A. A. Rakauskas, J. Dudonis, V. Stankus

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DEPOSITION OF OXIDE THIN FILMS BY THE COMBINED CATHODIC ARC-MAGNETRON SPUTTER TECHNIQUE

P10A. V. Stankus*, A. Rakauskas, J. Dudonis

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SYNTHESIS OF PbTiO₃ IN SPUTTERED Pb/Ti LAYER BY SOLID STATE REACTIONS

P11A. S. Rajesh, K. Perumal

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EFFECT OF DEPOSITION PARAMETERS ON THE OPTOELECTRICAL PROPERTIES AND MICROSTRUCTURE OF THE SPIN COATED METAL OXIDE THIN FILMS

SECTION B

- P1B. **M. Rosina^a, K. Fröhlich^a, C. Dubourdieu^b, F. Weiss^b, M. Jergel^c**
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^cInstitute of Physics, SAS, Dúbravská cesta 9, 842 28 Bratislava, Slovakia.
INVESTIGATION OF THE QUALITY OF SURFACES AND INTERFACES OF SINGLE LAYERS, BILAYERS AND MULTILAYERS BASED ON $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3$ BY AFM AND X-RAY REFLECTIVITY METHOD
- P2B. **M.Pripko^a, K. Fröhlich^a, M. Maryško^b**
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MAGNETO-TRANSPORT PROPERTIES OF OXYGEN POST-ANNEALED LaMnO_3 THIN FILMS
- P3B. **A. Kalvane, M. Antonova, M. Livinsh, A. Spule, L. Shebanovs, A. Sternberg**
Institute of Solid State Physics, University of Latvia, 8 Kengaraga str., Riga, LV 1063, Latvia
STRUCTURE AND PROPERTIES OF HIGH PIEZOELECTRIC COUPLING $\text{Pb}(\text{B}', \text{B}'')\text{O}_3 - \text{PbTiO}_3$ BINARY SYSTEMS
- P4B. **A.Jukna¹, J.Paršeliūnas¹, S.Balevičius¹, O.Kiprijanovič¹, V.Lisauskas¹, A.Abrutis², V.Plaušinitienė², A.Teišerskis²**
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ELECTRIC PROPERTIES OF THIN SUPER-CONDUCTING FILM AERIAL
- P5B. **P. Dłużewski, A. Szczepańska, J. Pelka, W. Paszkowicz, A. Wawro, L.T. Baczewski**
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X-RAY AND TEM STRUCTURAL STUDY OF Co/Gd MULTILAYERS WITH EXOTIC MAGNETIC PROPERTIES
- P6B. **G.-J.Babonas¹, L.Leonyuk², R.Szymczak³, A.Reza¹, V.Maltsev², M.Baran³, L.Dapkus¹, V.Jasutis¹**
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SURFACE LAYERS ON $(\text{M}_2\text{Cu}_2\text{O}_3)_m(\text{CuO}_2)_n$ SUPERCONDUCTORS
- P7B. **F. Anisimovas, J. Liberis, B. Vengalis**
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MICROWAVE NOISE IN $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ THIN FILMS

P8B. V. Karpus¹, A. Rėza¹, G.-J. Babonas¹, A. Suchodolskis¹, W. Assmus², R. Sterzel², V. Kazlauskienė³, J. Miškinis³, and A. Miniotas⁴

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SURFACE OXIDE LAYERS ON *i*-ZnMg(Y,Ho) QUASICRYSTALS

P9B. O.Kiprijanovič, S.Balevičius, V.Pyragas, E.E.Tornau, A.Jukna, B.Vengalis, F.Anisimovas

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EFFECT OF NANOSECOND MAGNETIC AND ELECTRIC PULSES ON RESISTANCE OF $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ THIN FILMS

P10B. K. Bormanis, M. Kalnberga, M. Livinsh, A. Patmalnieks, and A. Sternberg

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MICROSCOPIC STUDIES OF THE SURFACE OF FERROELECTRIC AND HIGH TEMPERATURE SUPERCONDUCTOR LAYERS

P11B. V.Vaicikauskas, R.Antanavicius, R.Januskevicius, J.Bremer*, O.Hunderi*

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OPTICAL CONSTANTS OF ITO DETERMINED BY ELLIPSOMETRY OF SURFACE PLASMONS

P12B. A.Galdikas, S.Kačiulis, A.Mironas, D.Senulienė, V.Strazdienė and A.Šetkus

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SENSITIVITY TUNING BY ADDITIONAL LAYERS IN TiN OXIDE THIN FILM BASED GAS SENSORS

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TRANSMISSION ELECTRON MICROSCOPY STUDY OF OXIDE HETEROSTRUCTURES

APPLICATION OF PULSED-INJECTION MOCVD TO THE SYNTHESIS OF MULTILAYERS AND SUPERLATTICES AT NANOMETRIC SCALE

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A new technique has been developed in LMGP and in Vilnius University for the generation of active gases for Metal Organic Chemical Vapor Deposition of thin layers. The principle is a sequential, computer driven injection of micro amounts (few milligrams) of a solution of organic precursors into an evaporator, where a flash volatilization occurs. An important particularity of this new vapor generator is that only one injection source is needed for the synthesis of compounds containing several chemical elements : for conventional CVD processing, one source is needed for each element. This system offers a considerably higher versatility than PVD single sources techniques: the synthesis of a PVD single source for multi components materials is a complex operation (mixing, compaction, sintering..), while the injection source is only a liquid "cocktail" of precursors. An other fundamental difference with conventional MOCVD source is that the thickness of the layer is only determined by the number of drops injected ("digital growth"), for fixed injection and growth parameters (injection frequency, aperture time, pressure, substrate temperature, gas flows).

A wide variety of single layers and multilayers have already been grown, both at LMGP and in Vilnius University, using this new CVD source (from simple Y, Ti, Ce, Al or Ta oxides to more complex compounds like $\text{YBa}_2\text{Cu}_3\text{O}_7$, $\text{PrBa}_2\text{Cu}_3\text{O}_7$ or SrTiO_3 , BaTiO_3 or $(\text{La,Sr})\text{MnO}_3$). In the field of high temperature superconductors, high quality $\text{YBa}_2\text{Cu}_3\text{O}_7$ epitaxial layers have been recently obtained in Vilnius University on 3" LaAlO_3 single crystal substrate ($T_c=92.1$ K, $\Delta T_c=0.15$ K, $J_c(77\text{K})=4-6$ MA/cm² over the whole surface).

Using two injection sources working sequentially, and injecting different solutions, it is possible to obtain complex stackings of layers. After a brief description of the injection technique, the presentation will be focused on the synthesis of amorphous, polycrystalline or epitaxial multilayers like , amorphous $\text{Ta}_2\text{O}_5/\text{SiO}_2$ multilayers or $\text{SrTiO}_3/\text{BaTiO}_3$, $\text{YBa}_2\text{Cu}_3\text{O}_7/\text{PrBa}_2\text{Cu}_3\text{O}_7$ and $(\text{La,Sr})\text{MnO}_3/\text{SrTiO}_3$ superlattices with various modulation length. The structural and transport properties of some of these superlattices will be presented.

INDUSTRIAL SCALE MOCVD PROCESSING OF ELECTROCERAMIC THIN FILMS

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Deposition methods like chemical solution deposition or sputtering methods will not meet the requirements of future ULSI memory devices, where feature sizes decrease rapidly and 3-dimensional structures become necessary. Hence, the unanimous opinion of experts in this field see MOCVD as strongly required for higher density devices. CVD combines advantages, such as easy composition control, uniform large area deposition and the coverage of non-planar shapes (fig.1).

Computational modelling. Computational modelling of MOCVD of electroceramic thin films is useful to not only understand basic mechanisms of the growth process, but also to optimise the performance of the processing equipment used to reproducibly grow uniform layers of e.g. BST on large surface area substrates.

The modelling approach to predict MOCVD BST growth involves the computation of Navier-Stokes flow coupled with heat transfer and chemical species mass transfer. In spite of the limited knowledge about the detailed chemical mechanisms involved in MOCVD of BST homogeneous and heterogeneous chemical models have been included into pure transport models to describe the growth process realistically.

Three dimensional reactor design tools and extensive computational modeling of three-dimensional flow, heat transfer and species mass transfer were conducted in order to optimize the uniformity of precursor distribution in the process chamber and of the layer deposition on large area substrates, as illustrated by fig.2.

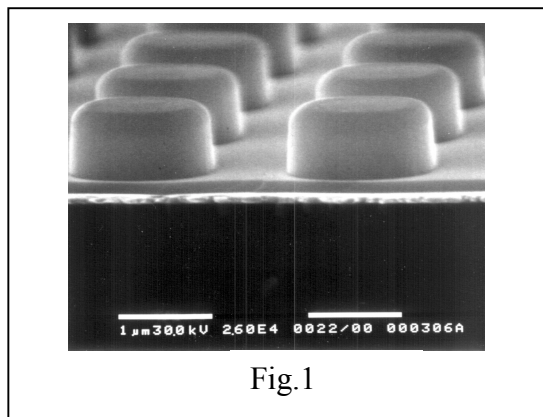


Fig.1

Fig.1 Step coverage of a SBT thin deposited on trenches and stacks.

Fig.2 Simulated MO precursor concentration in a cross-sectional plane of the central part of the process chamber, located between the showerhead

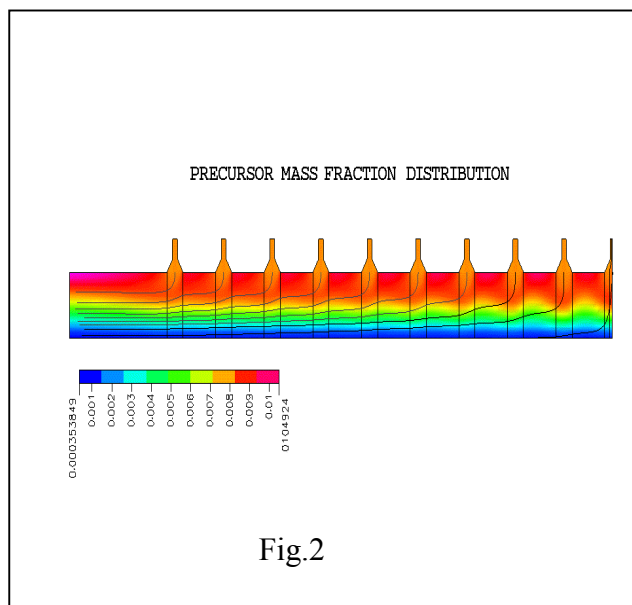


Fig.2

Outlook. MOCVD has become an important tool for a wide range of thin film material deposition already. The introduction of new materials such as ceramic BST, SBT and PZT into standardized silicon production process flows results in challenging tasks for semiconductor equipment companies. Intensive research and development work has to be carried out further in order to meet all the demands required by large scale production processes with respect to long term stability, reproducibility and cost. Once accomplished this hurdle, MOCVD will become one of the major entry keys into the coming multi-media era.

PULSED LIQUID-INJECTION MOCVD OF MANGANITE-BASED SUPERLATTICES

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Pulsed liquid-injection MOCVD is used at LMGP to grow oxide thin films and heterostructures. Briefly, the precursor solution is injected by a microvalve into an evaporator, where it is "flash" evaporated. The pulse width and injection frequency of the microvalve are controlled by computer. This delivery scheme allows one to control precisely the amount of each element in the gas phase. Heterostructures are grown by using sequentially two or more of these microvalves.

In a first part, results obtained on single manganite layers deposited on various substrates will be presented. The effect of substrate on the lattice strain will be shown.

In a second part, the synthesis and properties of $(\text{La}_{0.67}\text{Sr}_{0.33}\text{MnO}_3/\text{SrTiO}_3)_{15}$ superlattices (LSMO/STO) will be presented. The superlattices have been grown on different substrates (LaAlO_3 , SrTiO_3 , $\text{SrTiO}_3:\text{Nb}$, MgO). From $\theta/2\theta$ scans, the LSMO and STO layers are oriented mainly with the c-axis perpendicular to the substrate plane. The satellite peaks characteristic of a superlattice structure are clearly visible on the X-ray diffraction patterns. The period of the superlattices is calculated from the position of the satellites peaks.

We have prepared different set of samples where one of the constituent thickness was kept constant, whereas the other one was changed by varying the number of injected droplets. The corresponding period of the stacking depends linearly on the number of injected droplets, which shows that the individual layer thicknesses can be adjusted quite precisely. In our deposition conditions, the average growth rates are ~ 0.13 nm/injection for LSMO and 0.042 nm/injection for STO, with no significant difference as regard to the substrate used. The mean lattice parameter (in the [001] direction) and thus the strain state of the superlattices strongly depends on the relative thicknesses of the LSMO and STO layers and also on the substrate used. Defects present in the superlattices have been studied by electron diffraction patterns, transmission electron microscopy images and pole figures. Magnetization experiments have been performed in order to probe the oxygenation of the multilayers. Finally, the limits of the liquid-injection MOCVD process will be discussed with examples of multilayers grown down to very small periods ($(8 \text{ u.c.} / 8 \text{ u.c.})_{15}$, $(8 \text{ u.c.} / 3 \text{ u.c.})_{15}$ and $(3 \text{ u.c.} / 3 \text{ u.c.})_{15}$).

LARGE AREA YBCO FILMS DEPOSITED BY PULSED INJECTION CVD

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YBCO film growth process was optimized in a built new pulsed injection CVD reactor assigned for large area deposition. Y, Ba, Cu 2,2,6,6-tetramethyl-3,5-heptandionates dissolved in monoglyme were used as precursor materials. The deposition was carried out at 825°C and partial oxygen pressure 2 Torr (total pressure 5 Torr). Homogeneity of the critical temperature T_c and critical current density J_c was first verified by deposition of YBCO film (~0.4 μm) on 13 small (5×5 mm²) LaAlO₃ (100) substrates distributed on 3 inches substrate holder. Several YBCO films of various thickness (0.2-0.7 μm) were deposited on 3 inches and 2 inches LaAlO₃ substrates and the distribution of critical current density was established by AC susceptibility measurements (resolution 5 mm). Films with thickness d~0.2-0.3 μm prepared on 3 inches substrate showed $T_c \sim 92$ K, $\Delta T_c \sim 0.2$ K and exhibited high J_c values of 4-6 MA/cm² over an area of 65-70 mm in diameter. Typical value of the measured low-field surface resistance R_s was 0.35 m Ω (8.5 GHz and 77 K). Thicker large area films had lower J_c values.

EPITAXIAL PHASE STABILISATION PHENOMENA IN THIN OXIDE FILM GROWTH

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An analysis of authors experience in high temperature MOCVD of thin films of multicomponent oxide compounds in respect of epitaxial phase stabilization phenomena will be given in the paper. The phase relations of complex oxide systems in thin epitaxial films may differ from those known for bulk materials. In this study, the formation of nonequilibrium-in-bulk phases and the appearance of new phase relations has been observed in various thin film oxide systems including off-stoichiometric epitaxial films of high T_c superconductors (001) $\text{RBa}_2\text{Cu}_3\text{O}_{7-\delta}$, magnetoresistive materials, and rare earth nickelates of perovskite structure. It was shown, that the formation of coherent interfaces, which are inherent in epitaxial film growth, enables stabilization of otherwise non-equilibrium phases. The effect is due to contribution of coherent film/substrate interface lowering free energy of the system respectively to polycrystalline state.

The thermodynamic and kinetic respects of these phenomena, their relation to Dankov-Van der Merve principle, as well as the role of interface mismatch and volumetric factors are discussed. It is concluded that thin film growth techniques provide a powerful tool for synthesis of unstable-in-bulk oxide compounds in an epitaxial state thus making them possible components of epitaxial heterostructures.

COMPOSITION DEPENDENCE OF THIN FILM GROWTH CHARACTERISTICS IN COMPLEX OXIDES.

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Thin films of a complex oxide system like Y-Ba-Cu-O have been deposited by off-axis Pulsed laser deposition. The films have been prepared in a special geometry so they reproduce the inhomogeneous distribution of the species in the plasma plume. In this way the films show a cation composition gradient in one direction onto the film surface. In these samples it was possible to explore the variations on different features of the films, like morphology of the surface, domain orientation, microstructure, oxidation, as a function of cation composition only, along an extended composition range on the Y-Ba-Cu-O phase diagram. The influence of other important factors like fluctuations in the deposition parameters (substrate temperature, oxygen pressure, laser energy), or in substrate characteristics (surface state, miscut angle) that very often obscure the comparison between the characteristics of the films, have been neglected by this method.

In the particular case of Y-Ba-Cu-O system, it has been observed the existence of a critical Cu/Ba ratio 1.6 that separates two regions in the phase diagram with clearly different film growth characteristics. For $\text{Cu/Ba} > 1.6$ the films show a flat and very well oriented *c*-axis YBCO matrix with an increasing amount of CuO precipitates in the surface, typical for Cu-rich YBCO films. At compositions $\text{Cu/Ba} < 1.6$ a dramatic change occurs and the films become progressively rough and misoriented. This change in microstructure also affects the final oxidation state of the films. The optimal conditions for YBCO deposition (smooth and free-of-precipitates surface morphology, and highly textured structure) lie in a very narrow range of compositions $1.65 > \text{Cu/Ba} > 1.55$. The shift, towards a Cu-rich composition, of the critical ratio $\text{Cu/Ba} = 1.6$ from the expected value $\text{Cu/Ba} = 1.5$, corresponding to the stoichiometric $\text{YBa}_2\text{Cu}_3\text{O}_7$ phase, could be only explained in terms of an improvement of the crystalline quality of the YBCO material through the formation of supplementary Cu-O planes. These supplementary Cu-O planes would help the typical spiral-mediated growth to develop, and would prevent the antiphase boundaries, which are originated at substrate steps, to progress towards the film surface.

THIN FILMS OF $\text{Sr}_2\text{FeMoO}_6$ DEPOSITED BY PULSED LASER DEPOSITION

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We have fabricated films of the double perovskite $\text{Sr}_2\text{FeMoO}_6$ (SFMO) by pulsed laser deposition on SrTiO_3 and MgO substrates. X-ray diffraction shows that all films are well textured along the (00l) direction. We have studied magnetic and transport properties of films deposited at different substrate temperatures (850-950° C). Films deposited on MgO have poor magnetic properties and nearly insulator behaviour whereas films deposited on SrTiO_3 have properties similar to those of the bulk material.

We have also analysed the film surface morphology by AFM. Films deposited on SrTiO_3 are smooth with roughness of ~2 nm. Films deposited on MgO are smooth but have L-shaped holes, probably resulting from island growth mode.

**TiO₂ THIN FILMS BY ATOMIC_LAYER CHEMICAL VAPOUR DEPOSITION:
GROWTH AT 365 °C**

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Atomic-layer chemical vapor deposition (ALCVD) is a pulse technique devised for growing thin films in gas transport reactors. Distinctive features of the technique arise from the division of the overall CVD reaction into partial surface reactions ensuring the self-limited growth with the steps of the order of a monolayer. The oxide growth can be made well controllable and stable even in the case of the ALCVD development by the submonolayer steps.

Here we apply ALCVD in a newly designed reactor to the growing of titanium dioxide thin films from TiCl₄ and H₂O at 365 °C on quartz glass and GaAs(100) substrates. The reactor has an optical access capability allowing us to *in situ* monitor the growth by incremental dielectric reflection. We obtain additional information from the scanning force microscopy, electron probe microanalysis, reflection high-energy electron diffraction, and UV-VIS spectrophotometry measurements performed *ex situ* on a series of the films of different thickness.

The nonhomogeneous film growth as a function of time is revealed. The change from the rise of anatase particles inside and on the top of the amorphous base layer of TiO₂ into the polycrystalline overlayer growth is characterized in detail.

Sr₂FeMoO₆ DOUBLE PEROVSKITES. ARE THEY ALTERNATIVE MATERIALS FOR SPIN DEVICES?

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Double perovskites of Sr₂FeMoO₆-type are ferromagnetic metals having a Curie temperature substantially higher (about 420K) than that of the celebrated manganese perovskites (about 360K). It has been suggested that these oxides are half-metallic ferromagnets, and thus a full spin polarization of the itinerant carriers should be expected. Accordingly, they appear to be promising candidates to be used in the spin devices. However, the synthesis of these materials both in bulk and thin film form appears to be challenging and consequently the understanding of their properties is still in its preliminary steps. In this paper we shall review the basic properties of these novel materials and the progress that has been achieved in the synthesis and growth of bulk and thin films.

NON-DESTRUCTIVE CHARACTERIZATION OF EPITAXIAL PEROVSKITE LAYERS BY RAMAN SPECTROSCOPY

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Recently the growth of the epitaxial perovskite heterostructures including a variety of functional materials, like CMR manganites $R_{1-x}A_xMnO_3$ (where R - rare earth cation, A - alkaline earth cation), ferroelectrics ($PbZr_{1-x}Ti_xO_3$, $(Ba,Sr)TiO_3$), electrodes ($La_{1-x}Sr_xCoO_3$, $LaNiO_3$) and structurally related to the perovskites high T_c superconductors ($RBa_2Cu_3O_{7-x}$), was successfully demonstrated by the vapor deposition techniques. The structure and physical properties of such epitaxial layers in the heterostructures can differ significantly from those of bulk materials of the same nominal chemical composition mainly due to the contribution of the epitaxial lattice strain. On the other hand, the precise control of the composition necessary for high performance heterostructures is more difficult for the films than for bulk material. Thus, there is a great demand for the non-destructive characterization techniques for the epitaxial perovskite layers. We demonstrate that the Raman spectroscopy can be successfully applied for structure and composition characterization of the films.

Raman spectra measurements were performed with a triple monochromator system (Jobin-Yvon T64 000) in a subtractive arrangement and in a backscattering geometry of the incident laser light (514.5 nm, Ar⁺-ion laser) [1]. The attached OLYMPUS optical microscope allowed to focus laser beam on a selected area on a micrometer scale. For low temperature measurements samples were placed in a vacuum microchamber cooled with liquid helium.

CMR manganites were characterized with respect to the cation substitution in the perovskite structure, oxygen isotope exchange and variation of the Mn^{3+}/Mn^{4+} ratio. The effects were separated by analysis of the experimental band shifts of the $A_g(2)$ soft mode as a function of the tolerance factor t and a parameter $V^{1/3}t$, where V is the perovskite cell volume. This allows to structurally and chemically analyse mixed-valence manganites like $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}Mn^{16}O_3$, $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}Mn^{18}O_3$ ($x=0-1$), $(La_{0.25}Pr_{0.75})_{1-x}Ca_xMnO_3$ ($x=0.2-0.5$), $R_{1-x}Sr_xMnO_3$, $La_{1-x}MnO_3$. The mechanism of lattice strain in thin films of the compounds was tested; variations of the Jahn-Teller distortion and valence angle perturbations can in this case be separated by Raman spectrometry. We have found, that the selective chemical analysis of the perovskite layers by Raman spectroscopy often can be done with better precision than EDX analysis. In addition the Raman spectrometry is very sensitive to detect the secondary phases in the films like Mn_3O_4 even of the amount which can not be detected safely by XRD or EDX.

Another application of the Raman spectrometry consists in the observation of the phase transitions in the perovskite films sensitive to the epitaxial strain. In particular, the behavior of strained $NdNiO_3$ films on $LaAlO_3$ and $NdGaO_3$ was characterized. The first order metal-insulator transition in the materials was found to produce the drastic changes in the Raman spectra of the films. The band symmetry was determined with polarized measurements both for high temperature metal phase and low temperature insulating phase. The separation of the phases in the temperature range of the electrical resistivity hysteresis was found. The epitaxial lattice strain becomes vivid in the low temperature phase where A_g mode near 430 cm^{-1} (absent in the high temperature phase) is the most sensitive to the substrate variation.

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SPIN-POLARIZED QUASIPARTICLE INJECTION DEVICE BASED ON MOCVD-GROWN $\text{YBa}_2\text{Cu}_3\text{O}_7/\text{SrTiO}_3/\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ HETEROSTRUCTURES

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During the last few years, increasing attention was attributed to various metal oxide heterostructures containing high- T_c superconductors (HTS). Combination of HTS and colossal magnetoresistance (CMR) manganites such as $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ (LSMO) in the heterostructures provides great interest both for basic research and novel applications. CMR manganites exhibiting almost 100% of spin-polarized carriers in the ferromagnetic (FM) state offer promising possibility to inject spin-polarized quasiparticles into a superconductor in order to suppress superconductivity by breaking Cooper pairs.

A key issue for spin-polarized quasiparticle injection devices (SPQID) based on HTS/I/FM heterostructures is perfect crystalline structure of all constituent layers ensuring certain electrical and magnetic properties. In addition, high quality (flat and sharp) interfaces are highly appreciated. The HTS/I/FM heterostructures reported up to now have been prepared mainly by pulsed laser deposition. In this work, we demonstrate similar SPQID based on high quality YBCO/I/FM heterostructures deposited by cost-effective pulsed injection MOCVD technique offering wider possibilities for industrial applications.

The heterostructures composed of the $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ ($x=0.1-0.3$) underlayers, intermediate SrTiO_3 layers with thickness $d=0-20$ nm and $\text{YBa}_2\text{Cu}_3\text{O}_7$ cap layers were grown *in-situ* at 800-830°C under partial oxygen pressure of about 200 Pa on $\text{LaAlO}_3(100)$ substrates by using single source pulsed injection CVD method and metal β -dicetonates as source materials. The technological conditions were optimised in order to improve crystallinity and surface quality of the constituent layers. Wet etching was applied to form tape-like superconducting film for investigation of both longitudinal and transverse electrical transport in the heterostructures. Significant suppression of supercurrent has been demonstrated by injecting spin-polarized current from the underlying FM layer.

The authors acknowledge financial support from European Community (FP5, MULTIMETOX network, contract C5RT-CT-1999-05001).

NON-LINEAR CURRENT-VOLTAGE CHARACTERISTICS OF $\text{La}_{1-x}\text{MnO}_3$ FILMS.

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Manganite based heterostructures have attracted great deal of interest because of spin-dependent electronic transport and their potential application as low magnetic field sensor. Spin-dependent tunnelling effect in the manganite based heterostructures should be restricted to the interface between manganite film and adjacent isolant layer. Manganite layer itself should be of high quality, containing as low quantity of defects as possible.

To check quality of single $\text{La}_{1-x}\text{MnO}_3$ films we have performed measurement of differential conductance at different temperatures. The films were grown by liquid injection MOCVD method on polycrystalline Al_2O_3 and single crystalline Al_2O_3 and SrTiO_3 films. Transmission electron microscopy revealed that the film on the poly- Al_2O_3 substrate has polycrystalline structure with fine grained (25 nm) randomly oriented grain size. The film on the single- Al_2O_3 substrate consisted of densely packed epitaxial nuclei in polycrystalline matrix. The film on the single- SrTiO_3 substrate has shown the best preferential orientation. In all samples we have observed non-linear current-voltage characteristics in a wide temperature range. Shape of differential conductivity (dI/dV) suggests that both types of tunneling (elastic and inelastic) are present. We discuss a possible correlation between structure, tunnelling characteristics and magnetoresistivity in the $\text{La}_{1-x}\text{MnO}_3$ films.

TEM AND X-RAY CHARACTERIZATION OF MICROSTRUCTURE OF YBaCuO THIN FILMS DEPOSITED BY LASER ABLATION ON COLD SUBSTRATES

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We present the results of transmission electron microscopy (TEM) and X-ray synchrotron radiation (SR) diffraction measurements of YBaCuO thin films obtained by laser ablation method. The films were obtained by pulsed 248nm KrF excimer laser deposition on cold (300K) glass substrate from stoichiometric YBaCuO target. The same laser with pulse energy fixed at below the ablation threshold was then used for additional treatment of the films. TEM observations were performed with the help of JEOL2000EX microscope operating at 200keV. The specimens were prepared by scraping the film onto Cu 1000 mesh grids. The SR diffraction measurements have been done at B2 beamline of HASYLAB. The diffraction patterns were recorded at $\lambda=0.12065\text{nm}$ and the incident beam grazing-angle was fixed at 0.5° . The beam width was 0.4 mm. The scintillation detector and Soller slits of 0.1° acceptance angle were used for output radiation recording. X-ray diffraction patterns for the as-deposited samples and the samples treated with 1 to 10 laser pulses revealed that crystallisation of the initially highly amorphized material started after the first 1-2 pulses. The created structure was different then that observed in superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$. The linewidth indicating the size of nanocrystallites producing the diffraction practically does not depend on the number of pulses applied. The TEM investigations of the films confirmed amorphous structure of the as-deposited samples and occurrence of nanocrystallites in the treated films. The size of nanocrystallites determined from TEM images and calculated from linewidth of X-ray curves shows some differences.

This work was partially supported by Polish Government (KBN) grants no. 2P03B09516, 2P03B14814 and TMR-Contract ERBFMGECT950059

ION BEAM DEPTH PROFILING OF MULTILAYERS

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Both, surface and bulk processes influence the depth profiles of multilayered structures obtained by ion beam profiling technique (AES, SIMS) and both of them depend on temperature of sample. The bulk processes such as thermal and radiation enhanced diffusion, ion mixing give the broadening of the initially sharp. The surface roughness development during ion bombardment result that the layers are not removed monolayer by monolayer and it gives significant changes in the registered spectra of surface composition. Mainly two processes are responsible for the surface roughness development: sputtering and surface diffusion.

In the presenting paper the kinetics of the surface composition during the depth profiling of multilayered structures is considered by the proposed two simplified phenomenological models. In order to emphasize the composition changes on the surface produced by surface roughness development and ion mixing separately, the calculated results from the two different models are compared. The first one includes the processes of sputtering and surface migration of atoms, and second one includes the processes of the mixing between layers and sequential removal of surface monolayers (layer by layer). The calculated results are compared with experimental ones, which give a good qualitative agreement.

The depth profiling at elevated temperatures may contribute to the both increase or decrease of the amplitude of oscillations of the surface concentration, depending on prevailing process at given conditions. Temperature enhanced surface atom diffusion gives the increase in amplitude of surface concentration oscillations and ion mixing, generally, gives the opposite result. However, considering the last one process the influence of heat of mixing (negative or positive for given system) must be taken into account.

It is shown that both processes the surface topography development and ion mixing taking place during ion beam depth profiling of multilayered structures result in broadening of initially sharp interfaces between layers and asymmetric shape of the peaks of the oscillations of the surface concentration. Both processes affect the amplitude of the oscillations of the surface concentration and give the same effect that the peaks of higher mass component show asymmetry in the form of enhanced trailing tails on the back profile side and at the same time the peaks of the lower mass component have a more abrupt back side. The increase in the sputtering rate: 1) decreases the amplitude of the oscillations of the surface concentration as a result of the increase of the surface roughening and 2) increases of the amplitude as a result of the decrease of the mixing time. The process of surface atom migration result in the increase of the amplitude of the oscillations of the surface concentration. The depth profiling at elevated substrate temperatures increases the amplitude of the oscillations of the surface concentration as a result of the process of surface atom migration (surface smoothing) and decreases it if the process of ion mixing is prevailing. As the processes of sputtering, ion mixing, surface atom migration depend on ion energy, angle of ion incidence, ion flux and temperature the main conclusion made from the qualitative analysis is that the effects induced by the surface and bulk processes are quite similar and the interpretation of experimental results without theoretical analysis is not so obvious in many cases.

MULTILAYERED SYSTEM WITH FORBIDDEN REFLECTIONS

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We consider a multilayer system, which gives low reflectivity for second order Bragg diffraction. We use an analytical method, which permits to design any type of multilayer system.

To get low reflection at some order of diffraction it is necessary to prepare the period structure in such a way that it makes the desired order of diffraction to become forbidden. Then the reflectivity in this order is determined only by imaginary part of refraction index or the potential. With the analytical method we can predict minimal reflectivity at the given order of the Bragg diffraction, and help to choose materials for multilayer construction.

OPTICAL MEASUREMENTS OF STRAIN AND STRESS IN THIN FILMS

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Physical principles of the conventional method for the internal stress measuring - the cantilever technique – and electronic speckle interferometry are discussed and related to the stress control in thin film – semiconductor substrate system. Different types of the laser interferometers (Michelson interferometer for the in – situ measurements, prism interferometer for ex-situ measurements) were created and applied in thin film analysis. Original hardware and software for the electronic speckle interferometer was created and applied for the analysis of diffusive reflecting samples. The He-Ne laser interferometer installed in the vacuum chamber, system of registration of fast variation of the interference fringes (system of the photodiodes) and camera based registration of the whole system of interference fringes (to restore surface profile variation) were used to monitor strain variation in the silicon based cantilever. Thermal strain component contribution to the residual stress, relation between the residual stress and deposition conditions were revealed for the plasma deposited amorphous $\text{Ge}_x\text{C}_{1-x}$ films. Correlation between the growth mode, residual stress level and sign was found for the CdS, ZnS thin polycrystalline films on GaAs. It is shown that changes from three- dimensional to two - dimensional growth results in the change from tensile to the compressive residual stress. Atomic force microscopy, Electron Spectroscopy for Chemical Analysis were used to investigate mode of growth and composition of thin films.

NANOTUBULAR 2D CRYSTALS. SYNTHESIS AND PROMISING APPLICATIONS

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Review of recent research in field of nanotubes (NTs) arrays and crystals would be presented. Ways for synthesis of aligned carbon quasi-1D NTs arrays and techniques for fabrication of 2D crystals of C-NTs would be reviewed. Possible methods to fabricate 2D nanotubular crystalline composites on base of boron nitride BN-NTs [1,2], oxide MoO₃, superconductors such as YBCCO, LuNiBC etc. would be proposed. Quantum properties, proximity effect, ordering effect and other properties of nanotubular crystals would be shortly considered. Novel predicted promising applications of the nanotubular crystals would be outlined, namely: the high-T_c superconductor on base of superconducting nanotubular composite with predicted record T_c [3,4], the sieve and zeolite- like catalyst on base of polymerized fulborenes, the photonic crystals, the hydrogen-storage materials, etc.

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DEPOSITION OF YBCO BY MOCVD FOR COATED CONDUCTORS FABRICATION

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The fabrication of coated conductors involves several aspects: substrates choice, optimisation of buffer layers structure, and development of a reel-to-reel process for the YBCO deposition. In this work, we have used textured nickel-based substrates, and we have envisaged two different routes for the buffer layer deposition: a) CeO₂ films deposited in reducing atmosphere by MOCVD to avoid nickel oxidation, and b) textured NiO layers grown by rapid thermal oxidation of textured Ni tapes. These buffer layers were obtained in a small scale system, which has been used as a base to develop a large scale reactor. This system is adapted to a fast and continuous process with long time stability and to a reel-to-reel route. Main results concerning the different aspects of this study will be presented.

The MOCVD techniques and results presented here are based on a close collaboration of several European MOCVD research groups.

The authors acknowledge the financial support of EU (Brite-EuRam Project BRPR-CT98-0676)

**FERROMAGNETIC AND SUPERCONDUCTING OXIDE HETEROSTRUCTURES
FOR SPIN INJECTION DEVICES**

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We report on the growth by pulsed laser ablation of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}/\text{SrTiO}_3/\text{La}_{1/3}\text{Sr}_{2/3}\text{MnO}_3$ heterostructures in cross-strip geometry, intended to be used for spin polarized quasiparticle injection experiments. We shall present the developments on the growth of these complex heterostructures and experimental issues regarding their characterization. It will be shown that the current injected from the ferromagnet produces a suppression of the critical current of the superconductor

SELF-TUNING APPROACH TO THE MOCVD OF HIGH-QUALITY LEAD-CONTAINING HETEROSTRUCTURES

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High volatility of PbO is evidently the reason why there are not so much examples of the vapour deposition of lead-containing complex oxides. At the other hand, the volatility of lead oxide provides very important advantage: under certain conditions the film adopts exactly as much lead oxide from the gas phase, as much it is necessary for a stoichiometric formula, while the excessive lead oxide evaporates. Thus, a new approach to the growth of complex oxide films free of any secondary phases using the volatile component was developed. Pb-precursor used for the deposition by MOCVD was Pb(thd)₂.

The important feature of our experimental MOCVD setup is the separate continuous-type evaporator for lead precursor. The other precursors were supplied with the flash evaporator from a glass fiber band [1]. Mn(thd)₃, La(thd)₃ and Ti(i-PrO)₂(thd)₂ were used as precursors. The film growth was performed at 680-750°C and p(O₂)=2 mbar. The saturation ratio of PbO (α_{PbO}) equal to the ratio of PbO vapour pressure to the equilibrium pressure over condensed PbO at the given temperature was chosen as a characteristic parameter. This parameter depends on temperatures of both substrate and evaporator and on gas flow; the experimental α_{PbO} value derived from the mass loss of lead precursor and respective process parameters was regulated within 1-100%.

The self-tuning approach was successfully used for the MOCVD growth of single phase La_{1-x}Pb_xMnO₃ (x=0.1-0.6) and PbTiO₃ films on perovskite substrates (LaAlO₃, NdGaO₃, SrTiO₃) and on MgO at the high deposition rate up to ~1µm/h. Lead distribution was checked by RBS. Being dependent only on La/Mn ratio at a sufficiently high PbO partial pressure, the cation composition is easily adjusted. In spite of relatively high deposition rate single-orientation films are always very smooth as it was shown by AFM: S_a = 1.2-1.5 nm. This method of surface smoothening was already successfully applied for the deposition of refractory oxides such as LaAlO₃ and MgAl₂O₄ [2]. High quality of the films was explained by the presence of quasi-liquid PbO surface layer activating the surface diffusion during the growth; noteworthy, the high smoothness of the interfaces is the key demand for the creation of high-capacity ferroelectric memory.

Both the metal-insulator and FM-PM transitions occur for the La_{1-x}Pb_xMnO₃ films above 300K and at room temperature the material is metallic and ferromagnetic. So, this material is a good candidate for a fabrication of tunnel magnetoresistive devices operating at room temperature. As well, high electrical conductivity makes these films interesting for the applications as thin film electrodes in various oxide circuits. The possibility of La_{1-x}Pb_xMnO₃ epitaxial growth under PbTiO₃ deposition conditions was shown making possible a growth of CMR/ferroelectric heterostructures in one deposition run. The layers obtained are suitable for the fabrication of low-field tunnel magnetoresistance devices, thin film capacitors and other elements of spin-polarised electronics.

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GROWTH AND INVESTIGATION OF THE (LaNiO₃, RuO₂)/La_{1-x}Ca_xMnO₃ HETEROSTRUCTURES

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Heterostructures containing ferromagnetic (FM), isolating (I) and highly conductive (HC) oxide films are very promising for various electronic applications. Usually there is a great need in high crystalline quality and perfect surface morphology of the constituent layers in such heterostructures. Furthermore, one needs to ensure certain electrical and magnetic properties of different layers.

In this work, we were focussing on preparation of the ferroelectric La_{1-x}Ca_xMnO₃ (LCMO), dielectric NdGaO₃ (NGO), highly conductive LaNiO₃ (LNO) and RuO₂ layers and the corresponding FM/I/HC, FM/I/FM/HC heterostructures. Systematic investigations of structural, electrical and optical properties of the grown films and multilayers were performed in order to find optimal technological conditions.

Thin LCMO films (d=100÷400 nm) were grown *in-situ* at 750°C under partial oxygen pressure of about 12-15 Pa using reactive dc magnetron sputtering system and disk-shaped ceramic La_{0.67}Ca_{0.33}MnO₃ target. Both lattice-matched NdGaO₃ and sapphire (buffered preliminary by conductive LNO or RuO₂ layers) were used as substrates. The LNO and RuO₂ films were sputtered in Ar-O₂ mixture (p_{O2}=5-10 Pa) at 650°C and 500-700°C, respectively.

Reflected high-energy electron diffraction and Θ -2 Θ X-ray diffraction revealed epitaxial growth of the prepared films and heterostructures. Perfect surface morphology of different layers was seen in SEM micrographs. Colossal magnetoresistivity of the LCMO layers and nonlinear current versus voltage dependencies have been indicated by passing electrical current both perpendicular and longitudinal to the LCMO film surface.

**MOCVD GROWTH AND CHARACTERISATION OF
La_{1-x}Sr_xMnO₃/SrTiO₃/La_{1-x}Sr_xMnO₃ HETEROSTRUCTURES**

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Metal oxide heterostructures consisting of La_{1-x}Sr_xMnO₃ ($x \approx 0.3$) layer, thin (5-30 nm) SrTiO₃ interlayer and top La_{1-x}Sr_xMnO₃ ($x \approx 0.3$) layer were prepared by single source pulsed injection MOCVD method. Heterostructures were grown on LaAlO₃ (100) substrates at 825°C under oxygen pressure of about 200 Pa. Metal 2,2,6,6-tetramethylheptane-3,5-dionates were used as precursor materials. The vapor phase was generated by injecting of microdoses of organic solution, containing a mixture of precursors in monoglyme. The technological conditions were optimised in order to improve crystallinity and surface quality of the constituent layers. Longitudinal electrical transport properties of the top and bottom layers in the FM/I/FM heterostructures was investigated. The influence of the thickness of SrTiO₃ layer to properties of the top La_{1-x}Sr_xMnO₃ layer was established. Paralelly SrTiO₃/La_{1-x}Sr_xMnO₃ bilayers on LaAlO₃ substrates were in-situ deposited and studied.

The authors acknowledge partial financial support from European Community (FP5, MULTIMETOX network, contract C5RT-CT-1999-05001).

OXIDE THIN FILM DEPOSITION ON EUTECTIC SUBSTRATES

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Thin films of different oxide materials have been deposited on special substrates prepared from eutectic composition in the Ca-stabilised Zirconia (CaSZ)/CaZrO₃(CZO) and Mg-stabilised Zirconia (MgSZ)-MgO systems. The substrates were cut from crystalline fibers grown by the LFZ technique. The substrates consist of monolithic single crystalline phases with different relative orientations, and well-defined sharp interfaces. Depending on the fiber preparation conditions both substrate phases can show different arrangements: from a randomly distributed lamellar pattern to a well oriented parallel lamellae in the case of CSZ-CZO, or parallel independent fibers of single crystalline MgO embedded in a MgSZ crystalline matrix. All of these phases are good candidates for oxide film deposition from the point of view of chemical inertness and lattice matching.

YBa₂Cu₃O₇ (YBCO) and La_{1-x}Ca_xMnO₃ (LCMO) films deposited on these substrates have shown a selective oriented growth depending on the substrate orientations. They reproduce the same pattern as the substrate, with different arrangements of two well-defined domain orientations. By choosing the proper substrate cut we have been able to obtain *i*) a parallel strip pattern of biepitaxial YBCO/CSZ-CZO, which shows a clear anisotropy in its transport properties, *ii*) or an array pattern of polycrystalline YBCO islands in a *c*-axis oriented YBCO film on MgSZ-MgO substrates, *iii*) or a strip pattern of epitaxial LCMO separated by polycrystalline, and misoriented, LCMO strips. These unique film structures have demonstrated interesting physical properties, as it is case of *i*) which showed a remarkable anisotropy in the electrical transport properties (longitudinal and transverse to the strips) in the normal and superconducting state, induced by the presence of weak links between the strips.

THE EFFECTS OF TEMPERATURE ON BISRCACUO THIN FILMS USING AEROSOL-ASSISTED METALORGANIC CHEMICAL VAPOR DEPOSITION (MOCVD).

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The aerosol-assisted MOCVD deposition was used to produce BiSrCaCuO thin films on MgO(100) and LaAlO₃ (100) substrates. This deposition device could easily deposit films on large area substrates at high rates. The effects of the deposition temperature, from 700 to 790°C, and in situ high temperature annealings are discussed.

X-ray diffraction (Bragg-Bentano, rocking curves and $\theta/2\theta$ scans), SEM/EDS and resistivity measurements were performed to characterise the films. Nominal compositions of the films are closer to 2224 stoichiometry. The Bi-2212, Bi-2201 and CuO phases were detected. Intergrowths between 2201/2212 and 2212/2223 phases were also found. The higher Bi-2212 phase content with good *c*-axis orientation are obtained in the narrow temperature window of 760–770°C for films deposited on LaAlO₃ substrate. Films of the Bi-2212 phase in LaAlO₃ substrates, at 760°C, present a $[110]_{\text{LaAlO}_3} \parallel [100]_{2212}$ in plane orientation. Films on MgO substrate present up to three in plane orientations $[110]_{\text{MgO}} \parallel [100]_{2212}$, $[100]_{\text{MgO}} \parallel [100]_{2212}$ and $[230]_{\text{MgO}} \parallel [100]_{2212}$.

In the early growth stages a competition between Frank van der Merwe (2D or terrace) and Volmer-Weber (3D or island) nucleation was found. As the film thickens the second dominates. The number of grains resolved on the film surface by high resolution SEM decreases as the deposition temperature increases, the nucleation activation energy being 5.9 ± 0.3 eV for the deposition on LaAlO₃ substrates and 2.8 ± 0.3 eV on MgO. The main difference is discussed in terms of CuO phase interference nucleated on MgO substrates.

After oxygen annealing up to 60 hours, the films show zero resistivity at 75 K. This relatively low value is due to the 2212/2223 intergrowth. The annealing time and annealing temperature effects on epitaxy and resistivity will also be discussed.

Results of thin film deposition of magnetoresistance manganites were already presented [1]. Development of superconductor/manganite multilayers by aerosol-MOCVD for devices like polarised currents is scheduled.

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MOCVD DEPOSITION OF YBCO FILMS ON BIAXIALY TEXTURED Ag SUBSTRATES

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Pulsed Injection MOCVD technique was optimised for the deposition of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films on $\{110\}\langle 112 \rangle$ textured Ag ribbons, prepared at D.P.M.C University of Geneva. All deposited thick YBCO films had about the same transition temperature (90-92 K) and (001) texture, but its in-plane orientation and consequently critical current density was dependent on substrate texturing quality. The best films (thickness 700-800 nm) exhibited superconducting transition with T_c (onset) = 92 K and T_c (end) = 90 K and critical current density about 10^5 A/cm^2 at 77 K. Thin (~200 nm) films on Ag substrates were not continuous and not superconducting.

This work was partially supported by the European Union through contract BRPR-CT-97-0556.

DEPOSITION OF OXIDE THIN FILMS BY THE COMBINED CATHODIC ARC- MAGNETRON SPUTTER TECHNIQUE

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Of late years, the multicomponent, and multilayer oxide thin films are intensively investigated. The new effective methods are searched for making these films. The combined cathodic arc- magnetron sputter method, which begin to use recently, can be ascribed to such methods. This method was used for making various metal alloys [1] and superlattice coatings of metal nitrides [2]. In our opinion, the cathodic arc- magnetron sputter method can be applied for reactive deposition of multicomponent oxide films too. The usage of the cathodic arc- magnetron sputter method allows us to extend the control possibilities for making films with more various properties. It is possible, because the cathodic arc deposition distinguish oneself by: (i) high evaporation efficiency which does not depend on the sputter coefficient of the target materials and low effect of reactive gas on the evaporation rate, (ii) high ionisation of evaporated particles (up to 100 %), (iii) high kinetic energy of emitted ions (10-100 eV).

The deposition process of various layers, such as, TiO_2 , PbTiO_3 , Ti-TiO_2 , TiN-TiO_2 was investigated in this work. The combined cathodic arc- magnetron sputter deposition was performed in standard arc deposition system BY-1B upgraded by fitting in the magnetron MAG-5 and the rotate substrate holder. The titanium, titanium oxide and titanium nitride were deposited using cathodic arc deposition and lead oxide was deposited using magnetron sputtering, because titanium has low sputtering coefficient. All these layers were deposited on Si(111) and glass substrates changing various processes parameters: cathodic arc current, magnetron power, substrate temperature and oxygen pressure. The background pressure was 0.004 Pa. The partial oxygen pressure was 0.27 Pa. The distance arc cathode- substrate and magnetron cathode-substrate was 21 and 7 cm respectively. The deposition rate of thin films were measured by weighing the films (to within 0.5 mg) deposited on aluminum foils before and after deposition. The dependence of the deposition rate TiO_2 on arc current and the dependence of the deposition rate PbO on magnetron power were measured. Changing the cathodic arc current from 110A to 160A the deposition rate of TiO_2 changes from 2 nm/s to 2.6 nm/s. Changing the magnetron power from 60W to 300W the deposition rate of PbO changes from 0.15 nm/s to 0.52nm/s. Are seen, that the deposition rate of TiO_2 is ten times bigger than PbO , therefore the inert gas Ar partial pressure was create to 0.04Pa for made the stoichiometric PbTiO_3 . In that case the deposition rate of PbO increase. The deposition rate of TiO_2 was decreased increasing the angle between substrate and particles flux. The two-layer structures Ti-TiO_2 , TiN-TiO_2 and multicomponent PbTiO_3 films were made. The optical properties and microstructure of these films were investigated.

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SYNTHESIS OF PbTiO₃ IN SPUTTERED Pb/Ti LAYER BY SOLID STATE REACTIONS

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Complex metal oxides (for example $A_xB_yO_z$ here A and B are conformable metals) thin films have been started to research widely of late years. The majority of complex metal oxides have a lot of specific, often unique properties. Perovskite dielectrics of metal oxides have a single class. It has a wide spectrum of the different properties. They are ferroelectric, piezoelectric, pyroelectric, dielectric non-linearity, electrooptic and other. PbTiO₃ thin films have had recently attracted considerable attention. Because of the unique physical properties of the material, and especially of thin film, give them a high potential for future applications such as ferroelectric random access memory (FRAM), pyroelectric infrared detectors, optical waveguide modulators and other. PbTiO₃ has relatively low dielectric constant and large remnant polarization, which are preferable properties in application to metal-ferroelectric-semiconductors (MFS) field-effect transistor (FET).

A variety of methods for thin film growth, including metal organic chemical vapor, metal organic chemical-liquid, pulsed laser deposition, sol-gel, RF sputtering, reactive magnetron sputtering deposition successfully developed to prepare epitaxial, preferred oriented and polycrystalline PbTiO₃. However the majority of them are difficult to realize in practice and too expensive to use for commercial realization.

One of methods to prepare $A_xB_yO_z$ thin films may be solid state reactions between metallic layer and oxygen. Whereas the solid phase reactions for massive ceramic synthesis are well known, the reactions in thin films correspond to different character, because of different activation energy of metastable structures in thin films, surface energy influences and etc. Therefore, it is important to research the processes, which occurs in solid phase reactions in thin films.

The formation of perovskite PbTiO₃ thin films by annealing in atmosphere substrate\Ti\Pb two layers structure (1 μ m) was investigated in this work. The titanium and lead layers were deposited using DC magnetron sputtering. The weighs of separate layers were chosen to reach the stoichiometry in PbTiO₃ thin film formation. The layers were deposited on Si(111), Si(100) and glass substrates. The deposited thin films were annealed in atmosphere at different temperatures (200-700°C) and times (20-180min).

The reaction processes were researched by changing the annealing temperature and time. The structure changes, phase composition transition, surface roughness of as deposited and post annealed thin films were measured and analyzed by X-ray diffraction method and scanning electron microscopy. Reactions' kinetics and crystallites growth's regularities in annealed thin films were investigated too.

It was concluded that there is a possibility to get perovskite PbTiO₃ thin films through oxidation and solid state reactions by annealing the substrate/Ti/Pb system in atmosphere. The pure PbTiO₃ was formed at 700°C temperature and in 60 minutes as minimal time. The quality and surface roughness of formed thin films heavily depends on substrate type and cooling time.

EFFECT OF DEPOSITION PARAMETERS ON THE OPTOELECTRICAL PROPERTIES AND MICROSTRUCTURE OF THE SPIN COATED METAL OXIDE THIN FILMS

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Large area thin film solar cells have to be economically viable for terrestrial applications. This makes it necessary to exploit thin and thick film technique that satisfy the criteria of simplicity, cost effectiveness, large area uniform and controlled deposition, and that yield well defined structural, metallurgical and electro optical properties. The deposition techniques have been revived extensively by the scientists all over the world. Among the conventional methods employed for producing thin films, Spray pyrolysis technique is the simplest of all the technique. By modifying the spray technique, a new spin coating system is developed. The system is optimized for coating metal oxide thin films. The opto electrical properties are analyzed in accordance with the deposition parameters.

Transparent electrically conducting Tin Oxide films were deposited onto soda lime glass substrates by oxidizing SnCl_2 . The deposition temperature in the range of 350-500 degree celcius and 500 -2000 RPM of the substrates were analysed. Scanning electron microscope studies of this films indigate that the mean grain size vaies from .094-0.0213 micro meters. It is found that the film uniformity and mean grain size increases with increasing the deposition temperature and at high spin. This behavior is explained interms of RPM of substrate, surface mobility and grain growth rate.

INVESTIGATION OF THE QUALITY OF SURFACES AND INTERFACES OF SINGLE LAYERS, BILAYERS AND MULTILAYERS BASED ON $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3$ BY AFM AND X-RAY REFLECTIVITY METHOD

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Magnetic oxide materials possessing a high degree of spin polarisation have been found to exhibit enhanced spin-dependent transport properties. A colossal change of magnetoresistance (colossal magnetoresistance – CMR) in manganites is associated with transition from high temperature paramagnetic insulator to low temperature ferromagnetic metal. In epitaxial thin films of manganites CMR is observed only in relatively high magnetic field and in a short range of temperature in vicinity of Curie temperature. Enhanced magnetoresistance and broadened transition has been newly reported in superlattice structures of doped Mn-oxides and SrTiO_3 . It is supposed that electronic and magnetic properties of such structures could be improved by controlling quality of the surface and interfaces between the layers. We have deposited epitaxial $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3$ thin films, bilayers $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3/\text{SrTiO}_3$ and multilayers $[\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3/\text{SrTiO}_3]_{15}$ with different thickness by injection-MOCVD method. The same deposition conditions for growth all these structures have been used. In this paper we deal with the quality of surfaces and interfaces of deposited structures. The samples were studied by Atomic Force Microscopy and X-ray reflectivity. Surface smoothness of $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3$ single films has been studied and the results obtained for films deposited on different substrates (LaAlO_3 , SrTiO_3 , MgO) are discussed. Surface smoothness of SrTiO_3 has been studied on bilayers $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3/\text{SrTiO}_3$ growth on LaAlO_3 monocrystalline substrate. Interface sharpness was investigated on multilayers $[\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3/\text{SrTiO}_3]_{15}$ by X-ray reflectivity method.

MAGNETO-TRANSPORT PROPERTIES OF OXYGEN POST-ANNEALED LaMnO₃ THIN FILMS

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It is known, that properties of LaMnO₃ can be varied by doping with La-site vacancies and its chemical structure can be described by formula : La_{1-x}³⁺ □_xMn_{1-x-2y}³⁺Mn_{x+2y}⁴⁺O_{3+y}²⁻, (□-vacancy). We discuss the changes of properties of La_{1-x}MnO_{3-δ} with various oxygen content. To study their magnetic and electric properties, we have prepared a set of epitaxial La_{1-x}MnO_{3-δ} thin films. The films were deposited on SrTiO₃ substrates using metal organic chemical vapor deposition in one deposition run. After the growth they were post-annealed at different temperatures in O₂ atmosphere, to obtain various ratio of oxygen. The electric and magnetic properties were investigated by measuring of temperature dependence of resistance and magnetization. Annealing at high temperatures significantly changes their properties. The samples exhibit metal-insulator transition from 210 K to 300 K. Magnetization measurements versus temperature show a paramagnetic-ferromagnetic transition corresponding to metal-insulator transition. Presented results demonstrate, that magnetotransport properties of La_{1-x}MnO_{3-δ} can be varied in wide range not only by cation composition, but also by oxygen content.

STRUCTURE AND PROPERTIES OF HIGH PIEZOELECTRIC COUPLING Pb (B', B'') O₃ - PbTiO₃ BINARY SYSTEMS

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The Pb(B', B'')O₃ relaxor materials are being widely used. B' in the general formula stands for a low-valence cation like Mg⁺², Ni⁺², Sc⁺³, etc. B'' - for a high-valence cation as Nb⁺⁵, Ta⁺⁵ etc. Recently attention has been paid to relaxor - lead titanate (PT) binary systems since these material exhibit high values of electromechanical coupling and remnant polarization near the morphotropic phase boundary (MPB) between rhombohedral and tetragonal phases.

A number of binary ferroelectrics PSN - PT, PMN - PT and the PSN - PMN - PT ternary system have exclusive dielectric ($\epsilon > 30\ 000$) and electromechanical coupling parameters ($k_p > 52\%$, $k_{33} > 65\%$) near the MPB. Pb[$\{\text{Sc}_{1/2}\text{Nb}_{1/2}\}_{0,58}\text{Ti}_{0,42}$]O₃ (PSNT58/42) was used as the primary material for studies. To obtain ceramics the 99,9% grade oxides have been utilized in a three-stage calcining. The hot-pressed ceramics technology was used. To study the effects of annealing on the dielectric and piezoelectric properties the hot-pressed ceramics were annealed at 1000°C up to 100 h. The effect of hot-pressing parameters and polarization procedure on piezoelectric properties of the ceramics are also examined. As shown by X-ray diffraction the PSNT 52/42 is a single phase tetragonal perovskite. The density of hot-pressed ceramics is 99,9% of the calculated, the size of grains - 4÷7µ.

The value of dielectric permittivity ϵ at room temperature is exchanged from 1600 to 2100 during polarization, the maximum being 32 000 ($T_c = 273^\circ\text{C}$). The high value of remnant polarization ($P_r = 42\ \mu\text{C}/\text{cm}^2$) provide the main contribution to the high electromechanical coupling ($\sigma_p = 0.19$, $k_p = 70\%$, $k_{31} = 48\%$).

Solid solutions of the (1-x)Pb(Lu_{1/2}Nb_{1/2})O₃ - xPbTiO₃ (PLuNT) and (1-x)Pb(Er_{1/2}Nb_{1/2})O₃ - xPbTiO₃ (PErNT) binary systems have been synthesised for the first time. Hot-pressed (temperature 930°C to 1130°C, pressure 25MPa) ceramics series have been obtained in the interval $0 < x < 0.80$ to study the structural, dielectric and piezoelectric properties, especially in the MPB region.

The maximum values of the electromechanical coupling coefficients $k_p = 0.663$, $k_t = 0.481$, $k_{31} = 0.355$ of PLuNT(1-X)/X ceramics are obtained for compositions near the monoclinic / tetragonal MPB - PLuNT 59/41.

The PLuNT and PErNT systems, having the highest T_m ($> 350^\circ\text{T}$) among binary Pb(B', B'')O₃-PT perovskites is favourable for high temperature piezoelectric sensors and actuators, and may be of interest as promising basis for thin film and single crystal performance.

ELECTRIC PROPERTIES OF THIN SUPER-CONDUCTING FILM AERIAL

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Current biased high- T_c -super-conducting (HTS) film, being in a super-conducting state at $T < T_c$ (T_c -is a critical temperature of the superconductor), can be switched to the resistive state within several picoseconds by light of a short pulse laser [1]. Excited by the short electric pulse, the super-conducting thin film can radiate high frequency UWB signals [2]. Both results demonstrate that a powerful ground penetrating radar (GPR), operating in the frequency band of 0.2-3 GHz and containing both an UWB aerial (to receive and radiate pulse signals) and a high power pulse generator (to excite this aerial), can be designed using a current J_c ($J_c > 1 \text{ MA/cm}^2$ -is the critical current density) biased superconductor film, triggered by a pulse laser.

The J_c of the such aerial, determining the power of GPR's pulse generator, depends on super-conducting film thickness, homogeneity, number of defects, geometry etc. The purpose of this work was to investigate the $J_c(T)$ dependence of thin $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) HTS film aerials formed in various geometric shapes (annular-loop (Fig.1), horseshoe, micro-strip etc.) These shapes were formed using liquid lithography and chemical etching. The YBCO films with a thickness of $0.2 \mu\text{m}$ were MOCVD grown onto LaAlO_3 ($\sim 5 \times 5 \text{ mm}^2$) substrates.

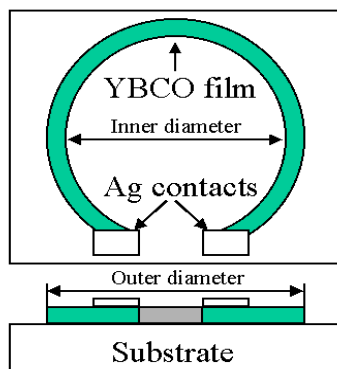


Fig.1 Schematic illustration of the super-conducting annular-loop aerial used for

The resistance vs. temperature dependence $R(T)$ of the best YBCO films demonstrated the $R(T > T_c) = -32.8 + 1.28 \cdot T_c(K)$ law at a temperature range of $300\text{K} > T > T_c$. The critical temperature was $T_c^{\text{off}}(R=0) = 92.7 \text{ K}$ and $\Delta T_c \leq 1\text{K}$. The current vs. voltage dependence ($I(U)$) of the super-conducting aerials was measured using a 7 ns duration pulse current, supported by a Hg-relay generator connected to a high frequency, 50Ω impedance transmission line loaded with a C8-13 sampling oscilloscope. The UWB radiation into free space of the pulse electric current exited super-conducting aerials was investigated in the frequency band of 0-5GHz using an Archimede double-spiral aerial and a real time C7-19 oscilloscope.

It was determined that the signal amplitude of the super-conducting annular-loop aerial (Fig.1) radiation does not depend on the dimensions of loop's inner diameter. We assumed that the Lorenz force induced by the non-homogeneous distribution of the magnetic field around the super-conducting loop causes a shift of the transport current towards the outer loop's boundary. We further assumed that the outer loop's boundary is equivalent to the aerial electrical length, which is responsible for the UWB radiation's central frequency. Most likely, the non-homogeneous distribution of the transport current causes a current "focussing" effect at the contacts of the super-conducting aerial, what, finally, affects a decrease of the critical current density of the superconductor. Thus even a pulse current of 7 ns intensively heats the contacts and, when $J^{7\text{ns}} > J_c^{7\text{ns}} \leq 1\text{MA/cm}^2$, this irreversibly damages the cathode region of the super-conducting aerial.

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X-RAY AND TEM STRUCTURAL STUDY OF Co/Gd MULTILAYERS WITH EXOTIC MAGNETIC PROPERTIES

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Transition metal – rare-earth TM/RE metallic multilayered systems (where TM is a 3d metal, like Fe, or Co and RE is a 4f metal – Gd, Dy, Tb, etc.) are interesting as model systems for studying the exchange interaction between TM and RE metals. Variety of magnetic properties from antiferromagnetic to the phenomena typical to ferrimagnets and ferromagnets observed in the TM/RE multilayers make them also promising technological materials, e.g. for magneto-optical devices.

The objective of this work is the structural characterisation of the multilayer composed of 3d and 4f metals, obtained by MBE process. The growth of multilayers was carried out in MBE system under the vacuum better than $5 \cdot 10^{-10}$ Torr by alternate evaporation of Co and Gd elements using e-guns as deposition sources. The deposition rates were 0.14 Å/s and 0.6 Å/s for Co and Gd respectively. The number of bilayers was kept between 20 and 30. The structure of individual layers and interfaces was studied by transmission electron microscopy (TEM) and X-ray grazing-incidence methods: reflectivity, diffuse scattering and diffraction. The samples have been characterised by conventional X-ray as well as with synchrotron radiation. The ion beam milling cross-sectional method was applied for specimens preparation for TEM purposes.

The investigations showed the enhanced diffusion of Co into Gd leading to thickness reduction of pure Co layers. The obtained results concerning the interfacial roughness and the crystal structure of sublayers for metallic thin film systems composed of Co/Gd and with layer thickness in the range 1 - 5 nm are related with the observed magnetic properties.

This work was partially supported by Polish Government (KBN) grants no. 2P03B09516, 2P03B14814 and TMR-Contract ERBFMGECT950059. Financial support from NATO grant HTECHLG-971803 is also acknowledged.

SURFACE LAYERS ON $(M_2Cu_2O_3)_m(CuO_2)_n$ SUPERCONDUCTORS

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The $(M_2Cu_2O_3)_m(CuO_2)_n$ ($M=Ca, Sr, Bi, RE$) compounds are complex cuprates. The structure of single crystals (orthorhombic symmetry $a \approx 11.3 \text{ \AA}$, $b \approx 13.0 \text{ \AA}$, $c_1 \approx 3.9 \text{ \AA}$, $c_2 \approx 2.7 \text{ \AA}$) is composed of two incommensurate sublattices containing the ladder-type plane $M_2Cu_2O_3$ and the plane of CuO_2 ribbons, in which the CuO_4 -squares are sharing edges. The Ca-doped single crystals with $m/n=5/7$ were shown [1] to be superconducting at $T_c \sim 80 \text{ K}$. The superconducting single crystals were grown from Bi-containing melts at maximum temperature $T_{max} \leq 915^\circ\text{C}$. The film of the Bi-2212-phase was formed on the sample surface at the immersion of the crystal into the residual melt as well as from the gaseous phase in a closed void.

In the present work the surface films of Bi-2212 phase on $(M_2Cu_2O_3)_m(CuO_2)_n$ -type single crystals were studied by spectroscopic ellipsometry. The optical response of the intergrowth between $(M_2Cu_2O_3)_m(CuO_2)_n$ -type and Bi-2212 phases was analyzed by the model of complete surface film and in the effective media approximation. It was shown that the c -axis oriented Bi-2212-phase films on the surface were typically of thickness 20-70 nm and were formed on both (ac) and (bc) planes of the $(M_2Cu_2O_3)_m(CuO_2)_n$ -type single crystals. On the basis of the experimental data, the structural model of the intergrowth was discussed.

The results of optical investigations were correlated with the data of structural and magnetic measurements. The rocking curves indicated the presence of Bi-2212-type phase film on the surface of the $(M_2Cu_2O_3)_m(CuO_2)_n$ -type single crystals. The formation of dendrites, inclusions and regularly shaped thin platelets of the Bi-2212-phase was determined by SEM in the regime of back scattered electrons. The manifestation of superconductivity in the samples studied was indicated by the temperature dependence of magnetic susceptibility. After the surface layer was removed by ion etching, the magnetic susceptibility of the samples have shown the anisotropy which was typical for superconducting $(M_2Cu_2O_3)_m(CuO_2)_n$ -type single crystals.

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MICROWAVE NOISE IN $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ THIN FILMS

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Low frequency noise in colossal magnetoresistance (CMR) manganites has been reported by a number of authors [1]. In this work present for the first time clear experimental evidence of microwave noise in the $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ thin films.

The $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ thin films were deposited on MgO(100) substrates by using Nd^{3+} :YAG pulsed laser operating in a doubled frequency mode ($\lambda = 532$ nm) [2]. The duration, repetition rate and energy of laser pulses were 8 ns, 12.5 Hz and 50 mJ, respectively. The substrate temperature during film growth was set at 750°C.

The microwave noise measurements were performed at 10 GHz using a short time domain gated radiometer technique [3]. The samples mounted into a waveguide were matched to perform noise measurements. Experimental values of the equivalent noise temperature T_n determined from the measured noise power, P_n , corresponding to the bandwidth Δf ($P_n = kT_n\Delta f$, k is the Boltzmann constant). The equivalent noise temperature of current fluctuations was measured at 300 K and 80 K under external magnetic field with $B = 0 \div 0.8$ T by varying current in a wide range. Short pulses of dc bias voltage with the duration of about 4 μsec and repetition rate of 125 Hz were applied to avoid sample overheating.

The microwave noise temperature, T_n , increased smoothly with increasing current in the temperature range $T < T_c$, here T_c is the paramagnetic to ferromagnetic transition temperature in the manganite film. However, the current fluctuations were reduced substantially at $T > T_c$. The noise temperature depended strongly on temperature and magnetic field. The Joule heating of the sample and external magnetic field were found to suppress the microwave noise.

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SURFACE OXIDE LAYERS ON *i*-ZnMg(Y,Ho) QUASICRYSTALS

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Recent advances of a quasicrystal growth technology allow for preparation of high quality single-grain quasicrystalline samples with linear dimensions up to ~ 1 cm, and, therefore, enable for a qualitatively new stage of studies of their physical properties. The problem of the quasicrystal surface layers is important from both physical and practical point of views due to quasicrystal applications as specific coatings. Atomic composition of icosahedral *i*-ZnMgY and *i*-ZnMgHo quasicrystalline specimens grown by the Bridgman method is close to $Zn_6Mg_3(Y,Ho)$. The perfect long-range ordering of the quasiperiodic atomic arrangement is proved by sharp Bragg peaks of X-ray diffraction patterns.

The surface analysis of the quasicrystals performed by energy dispersive X-ray, Auger, XPS, and SIMS spectroscopic techniques shows the quasicrystals to be covered by surface layers, which contain rather high concentrations of oxygen and which are characterised by considerable changes of relative concentrations of host atoms. The evaluated layer width is of the order of $0.1 \mu\text{m}$.

The AES and SIMS analyses of the chemical composition of the surface layers show the ion-etched surfaces to be rather active chemically. Essential oxidation of the surfaces is taking place even within high-vacuum chamber ($8 \cdot 10^{-10}$ Torr), where "initial" atomic composition of surfaces restores in several hours. The XPS and SIMS spectra of both *i*-ZnMgY and *i*-ZnMgHo sample surfaces indicate oxygen to form chemical bonds with all three host atomic constituents of the quasicrystals.

Investigations of the optical response of the quasicrystals, performed by the spectroscopic ellipsometry technique in the FIR–VIS (0.5–5.5 eV) spectral range, reveal a formation of quasicrystal surface layers in atmospheric environment. This is evidently witnessed by temporal variation of ellipsometric parameters measured from freshly polished samples, as well as by dependence of the pseudodielectric function on the incidence angle. The atmospheric formation of surface layers is characterised by several time constants. Initial changes of surface state (with time constant of the order of several hours) are followed by shallow temporal variation with rather large time constant (of the order of months or years).

Theoretical analysis of experimental spectra of pseudodielectric function, which was based on solution of the nonlinear Drude equation, indicates, however, the effective surface layer width to be smaller than that estimated from the SIMS data. The discrepancy is probably due to the thin top surface layer, which has been removed from quasicrystals by preliminary plasma etching. This allows for assumption, that the *i*-ZnMg(Y,Ho) surface layers presumably consist of a top thinner layer, which essentially affects the optical response of quasicrystals, and a thicker one (of the order of $0.1 \mu\text{m}$), which gradually approaches bulk. This preliminary model can serve as a basis for theoretical decomposition of the PDF spectrum, which aims at extraction of the dielectric function of the quasicrystals, and, therefore, would enable for a determination of electronic energy spectrum and reconstruction of parameters of the quasiperiodic potential.

EFFECT OF NANOSECOND MAGNETIC AND ELECTRIC PULSES ON RESISTANCE OF $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ THIN FILMS

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Manganese oxide compounds with distorted perovskite structures, $\text{A}_{1-x}\text{B}_x\text{MnO}_3$ (A-trivalent rare earth ion, B-divalent alkali earth ion), have received great interest because of their colossal magnetoresistance (CMR). It was demonstrated that CMR of La-Ca-Mn-O thin films could be successfully used for strong (up to 40 T) pulses of 0.2 – 2 ms duration magnetic field measurements [1]. In order to design high-speed magnetic field sensors it is necessary to have data about the time of resistance change response to magnetic field action. For this reason we had investigated the effect of 40 ns duration 0.3 T amplitude magnetic field pulses on resistance of $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ thin films. The investigation was performed using samples having inter-electrode distance from 15 μm to 30 μm and small size of 80 nH inductance coil for pulsed magnetic field generation. The sample was biased by short (100 ns) electric pulse, which created the strong electric field inside the film. The ratio between magnetic field induced "noise" signal and signal of CMR response was varied with temperature from 2 to 5.

Thin films $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ used for the investigation were prepared by pulsed laser deposition technique. The samples having thickness from 0.1 to 0.2 μm were deposited on MgO substrates under oxygen pressure 20 - 25 Pa using ceramic target of $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ composition. During the deposition the substrate temperature was kept at 750 C. This enable to fabricate film with phase transition temperature (T_m) 140 K. After the deposition the oxygen's pressure was increased up to 1 atmosphere. This operation was realized keeping constant temperature of the substrate (750 C). The last stage of the sample preparation was 3 hrs duration slow reduce of substrate temperature from 750 C up to room temperature. The X-ray diffraction measurements demonstrated, that so prepared films were single-phased and of pseudocubic perovskite structure. CMR study was performed on co-planar shape samples having 0.75 mm width Ag electrodes. Electrical resistance of small gaps measured by two-probes method reveal that differences in thickness arising during laser ablation and 9% mismatch between lattice constant of a pseudocubic unit cell of $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ and that of substrate give different ratios $R(T_m)/R(300\text{ K})$. Explanation of these effects was presented in paper [2].

Experiments showed that fast CMR rise and decay time of electrical pulse corresponding to the resistance change response to magnetic field is of the same order as magnetic field pulse ones. This demonstrates that magnetic ordering and disordering appears during very short time (less then ns) that is typical for electron's spin relaxation process. Thus, received fast CMR response permit to use this phenomenon for measurements of short magnetic pulses and designing magnetic field controlled switches.

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MICROSCOPIC STUDIES OF THE SURFACE OF FERROELECTRIC AND HIGH TEMPERATURE SUPERCONDUCTOR LAYERS

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Ferroelectric layers are used in electrically tuneable capacities for lower voltages compared to bulk materials. Electrically controlled capacity is the current application of ferroelectric materials required to be usable in a wide range of temperature. The high temperature superconductor (HTSC) layers are used to comply with high current densities. The problem of increasing the critical current in HTSCs has promoted novel technologies and understanding of conductivity mechanisms.

Apart from thin films, the rather thick (50 – 150 μm) layers are of interest for electronic micro-device applications. Properties of the layers essentially depend on the microstructure forming at thermal treatment of the material. Crystal structure of the layer is determined by a number of factors, technological, physical, and chemical parameters included; it is also influenced by prehistory of the layer production.

Ferroelectric and HTSC materials have been synthesized by solid state reaction from metal oxides and carbonates with two-stage calcination. 50–125 μm thick layers of superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) materials being obtained by Stokes sedimentation on ceramic (SrTiO_3 ; ZrO_2) and single crystal (MgO ; BaF_2 ; LiNbO_3) substrates and firing in air or oxygen following the modified MTG technology, performed in the gradient tube furnace.

The surface morphology and structure of bulk samples and layers were studied by electron microscope with respect to production regime (rate and temperature of crystallization) and physical properties.

The layers were found to have a textured polycrystalline structure consisting of several phases, the superconductor phase being represented as crystal platelets. The nonsuperconductor phase usually forms in the shape of rods. The YBCO layers deposited on single crystal substrates (MgO , BaF_2 , LiNbO_3) stick strongly to it and the crystal structure of the substrate unambiguously determines morphology of a dense and ordered layer. Practically single crystal layers with hardly distinguished grain boundaries and crystallographic (001) planes parallel to the substrate surface are obtained if cooling is started at the temperature of peritectic transition or slightly above it. Long grains (up to 100 μm) are formed on single crystal substrates. Morphology of the crystal platelets reveals a stepwise growth. Analysis of microstructure of the YBCO layers suggest existence of a rather narrow interval of slow cooling temperature of the MTG process must be started to obtain a homogeneous and oriented structure of the layers.

The different microstructure of superconducting (YBCO) and ferroelectric (BaTiO_3) grains was established. Coexistence of superconducting grains and non-superconducting configurations was observed in pure and YBCO composite layers. The more ferroelectric particles there are in the composite material, the more and bigger are formed the regions where crystalline needles or rods of the (211) phase prevail and the smaller are the structure elements. The microstructure of YBCO- BaTiO_3 composite layers is inhomogeneous with respect to grain size and orientation after the thermal treatment.

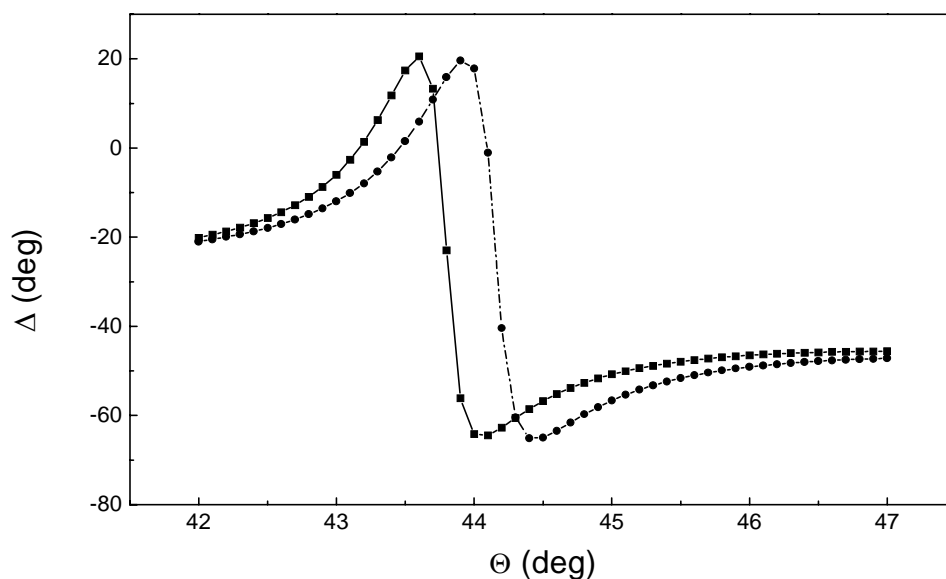
OPTICAL CONSTANTS OF ITO DETERMINED BY ELLIPSOMETRY OF SURFACE PLASMONS

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In experiments with surface plasmons (SP) one usually measures the angular dependence of the p-polarized intensity. We have used an ATR prism in the Kretschmann configuration and measured both amplitude and phase in a rotating polarizer-analyzer setup. The setup was used to determine the optical constants at 632.8 nm of a 20 nm thickness indium tin oxide (ITO) film. The numerical values $n = 1.91$, $k = 0.01$ for ITO were obtained by a best-fit procedure. It is argued from model calculations that in thin film cases the SP phase measurements give more precise n and k values than ellipsometry and conventional SP amplitude methods. Model calculations demonstrate that an ITO film of 1 nm thickness is capable of shifting the SP resonance at 44° about 0.5° . The change in the Δ parameter is almost 90° . It should be noticed that outside SP resonance this change is only half degree.



Besides a high submonolayer sensitivity, the SP phase method has some other advantages when compared with the amplitude one. This nondestructive method does not depend on laser stability and film interference effects. The optical system is compact, simple and cheap, and will be useful in both research and applications.

SENSITIVITY TUNING BY ADDITIONAL LAYERS IN TIN OXIDE THIN FILM BASED GAS SENSORS

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Semiconducting oxides are one of the most successful bases for gas sensors until now. High sensitivity, stability, reproducibility and compatibility with micromachining technology are the main features resulting in well-known advantages for this type of the sensors. On the other hand, the selectivity of these sensors is limited by the fundamental restrictions arising from the sensitivity mechanism. It was proved, that it is impossible to develop an individual sensor based on metal oxide suitable to detect only a particular gas. Recently, a method was proposed to improve the selectivity. The method was based on processing of the response signals of a sensor array by a special software. This method is well-known as an electronic nose. In this case, it is essential to collect a group of the sensors parameters of which is different as much as possible. Since the number of the sensors should be equal to about the number of gases in the smell that should be analysed, it is essential to develop technological methods for modification of the parameters of the sensors.

Present report deals with an original method based on deposition of additional layers on top and/or under the base thin film of tin oxide. Multi-layered gas sensors based on tin oxide are fabricated and investigated in this report. A dc-magnetron sputtering is employed for deposition of the layer. Electrical properties sensitive to gas-surface interaction are compared for various multi-layered structures. An influence of the modifying layers on the sensitivity to gas is analysed. Correlation between the sensing properties and the surface chemical composition is discussed based on the results of the XPS analysis.

It was demonstrated that the sensing properties of tin oxide thin films were significantly modified if ultra-thin metal layer was added. A change of the parameters was dependent on where the additional layer was situated. If the impurity metal layer was deposited on the top of tin oxide film, fine tuning of the sensing properties was possible by a change of an amount of deposited metal. A change of the surface properties depended on the metal used. An influence of different metals (Pt, Cu, Ag) was studied. From the XPS profiles, it was demonstrated that the edges of the layers in the multi-layered sensors could be distinguished only if Pt was used for the top or the bottom layer. If Cu and Ag were used, some complex structure with smooth edges of the layers was formed. The state of the impurities (metallic, oxide, hydroxide) were dependent on the metal and the place of the additional layer.

Based on the original method of the multi-layered structures, several families of tin oxide thin film based gas sensors were developed. The resistance response to similar gases (CO, H₂, NH₃, C₂H₅OH) were different in different families of the sensors. Moreover, the response signal could be modified to be different even within the same family. The modification method mostly influenced the kinetic of the sensor response to gas.

Based on the investigation of the properties of the tin oxide thin film gas sensors modified with the additional layers of impurities, some improvements of the model explaining gas sensitivity mechanism were suggested. New approach was discussed considering the role of the impurities in the gas-surface interaction and the conversion of the chemical interaction into electrical signal.

TRANSMISSION ELECTRON MICROSCOPY STUDY OF OXIDE HETEROSTRUCTURES

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High-resolution transmission electron microscopy (HRTEM) is a unique method used for analysis of crystal structure and morphology of thin epitaxial films. Unlike traditionally used X-ray diffraction, TEM is a local method. It gives direct image of atomic structure with ~0.1 nm resolution. "Cross-section" technique, i.e. analysis of a cut perpendicular to the film/substrate interface, is of primary importance. When samples with this geometry are studied, one can get information on details of interfaces, volume of the film and its surface, reveal misfit dislocations, inclusions, strain fields, determine domain boundaries structure.

This paper investigates thin epitaxial oxide heterostructures obtained by metal organic chemical vapor deposition technique (MOCVD). Cross sectional electron transparent areas of films were obtained by ion milling, with the film facing away from the ion gun. Electron microscopy was performed using a Philips CM30UT electron microscope equipped with the field emission gun operating at 300 kV, and a Link EDX element analysis system. Electron diffraction was performed with spot sizes between 4 and 20 nm; exposure times ranged from 0.1 to 5 s. Image analysis was made by standard programs Digital Micrograph 2.5 Demo and NIH Image 5.7.

The following heterostructures were investigated $\text{LnBa}_2\text{Cu}_3\text{O}_{7-y}/\text{CeO}_2/\text{R-Al}_2\text{O}_3$ (Ln=Y, Ho, Lu), $\text{HoBa}_2\text{Cu}_3\text{O}_{7-y}/\text{La}_{0.35}\text{Pr}_{0.35}\text{Ca}_{0.3}\text{MnO}_3/\text{LaAlO}_3$, $\text{HoBa}_2\text{Cu}_3\text{O}_{7-y}/\text{La}_{0.35}\text{Pr}_{0.35}\text{Ca}_{0.3}\text{MnO}_3/\text{SrTiO}_3$, $\text{CeO}_2/\text{ZrO}_2/\text{R-Al}_2\text{O}_3$, $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3/\text{PbTiO}_3/\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3/\text{MgO}$, $\text{NdNiO}_3/\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3/\text{LaAlO}_3$. The structure of interfaces, domain boundaries, inclusions and strain fields are discussed.

"DECOMPOSED" VORTEX LATTICE IN YBCO/PBCO MULTILAYERS

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The angular dependence of the longitudinal resistivity in superconducting (YBCO)_n/(PBCO)_m (n=4,8; m=12) multilayers was measured in magnetic fields up to 16 T, with a special focus on field directions close to the superconducting *ab* planes. The results were analysed in the framework of the 3D anisotropic scaling theory which predicts, for an angle θ between the field and the *ab* planes, $\rho(B,\theta) = \rho(B\varepsilon(\theta))$, where $\varepsilon(\theta) = (\sin^2\theta + \cos^2\theta/\Gamma^2)^{1/2}$ and Γ is the anisotropy parameter. Our results show that for the (YBCO)₄/(PBCO)₁₂ sample, a single Γ value of 200 leads to a perfect scaling of the $\rho(B,\theta)$ curves, over all the field and angle range explored. For the (YBCO)₈/(PBCO)₁₂ sample, the $\rho(B,\theta)$ curves do not scale with a single Γ value: for large angles and high magnetic fields a scaling with $\Gamma \approx 15$ is achieved, whereas for small magnetic fields a $\Gamma \approx 500$ is required [1]. These two regimes for Γ may be understood by attributing the $\Gamma \approx 15$ value to the intrinsic anisotropy of the YBCO layers and the $\Gamma \approx 500$ value to the effect of the insulating PBCO layers, which force the vortices to hit perpendicularly the interfaces between the YBCO and PBCO layers.

A case of particular interest arises for high magnetic fields and small angles, where a 3D scaling law is not capable of describing our data, whatever Γ we use. The failure of the 3D scaling law for this particular combination of fields and angles may result from the coexistence of two vortex lattices ("combined" or "decomposed" vortex lattice), as proposed by several authors [2]: one perpendicular to the layers and the other parallel to them. In this case, the total resistivity is due to the independent dissipation of the two lattices and can be approximated by: $\rho(B,\theta) \approx \rho(B\sin\theta) + \rho(B\cos\theta)$. For very small angles ($\theta \approx 0^\circ$), this expression leads to $\rho(B,\theta) \approx \rho(B\sin\theta) + \rho(B,0^\circ)$, in agreement with our results. The existence of this decomposed vortex lattice in the (*B*, θ) plane will be discussed.

[1] A. Casaca et al., Proceedings of LT22, Physica B (2000).

[2] See for instance: A. E. Koshelev, Phys. Rev. Lett. **83** (1999) 187; D. A. Huse, Phys. Rev. B **46** (1992) 8621.