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OTRZYMYWANIE KOMPOZYTU WŁÓKNO WĘGLOWE-MIEDŹ W PROCESIE ZGRZEWANIA OBJĘTOŚCIOWEGO

Kompozyty wzmacniane włóknami należy uznać za najbardziej atrakcyjną odmianę materiałów o programowanych właściwościach. Techniki wytwarzania tych materiałów stwarzają duże możliwości doskonalenia ich własności mechanicznych, cieplnych oraz elektrycznych. Własności te zależą od rodzaju osnowy, włókna, stopnia wypełnienia włóknami oraz od sposobu ich rozmieszczenia i mikrostruktury połączeń osnowa-włókno.

Manipulując tymi czynnikami można uzyskać zmianę takich własności materiałów jak: wytrzymałość na zrywanie, oporność właściwa, przewodnictwo cieplne, twardość czy zmianę temperaturowego współczynnika rozszerzalności liniowej.

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OTRZYMYWANIE KOMPOZYTU WŁÓKNO WĘGLOWE-MIEDŹ TECHNIKĄ ODLEWANIA FOLII

Przedstawiono związek właściwości: mechanicznych, elektrycznych i cieplnych kompozytów włókno węglowe-metal, w zależności od mikrostruktury kompozytów i techniki wytwarzania.

Szczególna uwaga została poświęcona doświadczeniom własnym autorów pracy, obejmującym wytwarzanie kompozytu włókno węglowe-miedź z zastosowaniem techniki odlewania folii. Z przedstawionych badań własnych wynika, że metoda foliowa może być przydatna w produkcji kompozytów włókno węglowe-miedź, ponieważ daje możliwości stosowania włókien ciągłych. Pozwala to na wytwarzanie kompozytów o ukierunkowanym ułożeniu włókien. Otrzymane kompozyty mogą być stosowane m. in. jako elektrody do zgrzewania oporowego, jako kompozytowe styki elektryczne i podkładki dylatacyjne do półprzewodnikowych elementów mocy.

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FORMOWANIE SIĘ WARSTW PRZEJŚCIOWYCH NA GRANICY WŁÓKNO WĘGLOWE-MIEDŹ W ZALEŻNOŚCI OD DODAWANYCH PIERWIASTKÓW AKTYWNYCH

Kompozyty z miedzianą matrycą mogą być stosowane w przypadku, gdy wymagane jest wysokie przewodnictwo cieplne i elektryczne oraz odporność w szerokim zakresie temperatur. Włókna węglowe charakteryzują się zarówno dobrym przewodnictwem jak i właściwościami mechanicznymi. Wynika stąd, że kompozyt włókno węglowe-miedź powinien posiadać optymalne z tego punktu widzenia właściwości.

Największym problemem przy otrzymywaniu kompozytów włókno węglowe-miedź jest pokonanie braku zwilżalności włókien przez czystą miedź (graniczny kąt zwilżania = 160^{o}). Problem ten można rozwiązać dwoma sposobami: pokrywając włókna metalem, który jest dobrze zwilżalny przez miedź lub domieszkując miedź pierwiastkami aktywnymi, które tworząc z węglem związki powinny obniżać wartość naprężeń na granicy włókno węglowe-miedź. Wynika stąd jasno, że idealnym rozwiązaniem byłoby pokrywanie włókien miedzią z pierwiastkami aktywnymi, co wpływałoby odpowiednio na formowanie się warstwy przejściowej, a jednocześnie nie zmieniało właściwości miedzianej matrycy. Ilość domieszkowanych pierwiastków aktywnych w miedzi może być nie za wysoka, a reakcje (ewentualne) powinny być moderowane termodynamicznie.

W pracy przedstawiono wyniki z badań warstw pośrednich włókno węglowe-miedź w kompozytach: matryca miedziana (czysta lub domieszkowana pierwiastkami aktywnymi) - włókno węglowe (czyste lub pokrywane) otrzymywanych metodą zgrzewania objętościowego.

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EXTENDED 1993 FICHE WORKSHOP ON INSTABILITIES IN CRYSTAL GROWTH Zurich, Swiss, 10-12/03.1993

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A NEW APPROACH TO CONSTITUTIONAL SUPERSATURATION AND GROWTH STABILITY IN THE HIGH TEMPERATURE VAPOUR GROWTH

Constitutional supersaturation criterion deduced from Mullins-Sekerka growth stability theory is applied to high temperature vapour growth system.

Experimental transition from unstable to stable growth conditions and mathematical formulation of the novel constitutional supersaturation criterion is presented. The origin of the flat faces at the growth interface and consequence of the criterion on the practical design of physical vapour transport growth system is discussed. Influence of the growth rate, pressure of the inert gas, furnace temperature profile and surface kinetics mechanism on the growth stability is presented. Experimental and theoretical results are compared to results of other authors.

Materiał przedstawiono na sesji referatów.
Pełny tekst opublikowano w materiałach konferencyjnych Extended 1993 Fiche Workshop on Instabilities in Crystal Growth. Laboratorium fur Festkorperphysik ETH, Zurich.

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SINGLE CRYSTALS WITH ANISOTROPIC PROPERTIES GROWN BY CZOCHRALSKI METHOD

It has been known that during crystal growth by the Czochralski method the flat interface attends the best quality of crystals. The flat interface executes uniform gradient of temperature along the crystal/melt interface and then for polycomponents and doped crystals uniform distribution of components is fullfiled.

Most crystals grown by the Czochralski method have the structure in which a/b/c axis are not differed strongly. Sometimes to avoid defects a little convex crystal/melt interface is available, however, facets formation and cracking can be appeared.

In the case of $ABCO_4$ compounds, where A=Ca.Sr: B=Y, rare elements and C=Al, Ga, lattice parameters are a=b=0.36-0.38 nm and c=3a. The compounds crystallize in the tetragonal structure of K_2NiF_4 - type structure. These crystals show strong anisotropic properties: structural, thermal, magnetic and it reflects in nature of crystal growth.

Several authors have reported that the crystal growth is unstable and some crystals are easy opaque and cracked. On the basis of our investigations the reason of this behaviour is discussed. Crystals of good structure quality are grown if the growth surface of crystals is strongly convex and formed by crystallographic (101) type planes.

Single crystals of $ABCO_4$ have been reported by the Polish team in 1991 as substrate for high Tc superconductors from standpoint of lattice matching and their dielectric properties. These compounds are also considered as new lasers crystals.

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State Committee for Scientific Research.

Pełny tekst został zaprezentowany w formie wykładu.

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THICK-FILM ELECTRODYNAMIC PRESSURE SENSOR

Thick film technology has been applied to simplify the existing design of the pressure sensor. A special nickel paste has been elaborated to produce a porous metallic layer on the ceramic membrane instead of metallization previously made of tungsten and manganese. To include the nickel layer to the rest of the circuitry a silver metallization has been applied replacing former molybdenum and manganese metallization. The new metallizations require firing at lower temperatures (570°C and 850°C, respectively) in air, whereas hard metallization firing had been carried at 1250°C in cover gas.

Materiał zaprezentowano w formie posteru.
Pełny tekst wystąpienia opublikowano w: Thick and Thin Films Sensors. Wrocław, IET, 1993.

II POZNAŃSKIE KONWERSATORIUM ANALITYCZNE, Nowoczesne metody przygotowania próbek i oznaczania śladowych ilości pierwiastków, Poznań, Polska, 27-28/04. 1993

W dniach 27 i 28 kwietnia 1993 w Politechnice Poznańskiej odbyło się II Poznańskie Konwersatorium Analityczne, w którym uczestniczyło ponad 100 osób reprezentujących kilkadziesiąt różnych instytucji. Konwersatorium połączone było z pokazami nowoczesnej aparatury analitycznej i sprzętu laboratoryjnego.

W ponad 20 referatach i komunikatach zaprezentowano zarówno wyniki prac własnych, jak i najnowsze osiągnięcia techniczne i metodyczne w dziedzinie analizy śladowej.

Przedstawiono szerokie spektrum zarówno wykorzystywanych metod analitycznych jak i analizowanych materiałów.

Wiele uwagi poświęcono nowoczesnym metodom przygotowywania próbek. Jest to istotny etap oznaczania zawartości pierwiastków śladowych w próbkach naturalnych. Prezentowano metody roztwarzania z zastosowaniem specjalnych urządzeń (mikrofalowych) oraz mineralizację ciśnieniową w tzw. bombach ciśnieniowych. Szereg wystąpień poświęcono wykorzystaniu metody Atomowej Spektrometrii Absorpcyjnej (ASA) zarówno ze wzbudzeniem płomieniowym jak i z atomizacją elektrotermiczną w kuwecie grafitowej. Jest to jedna z głównych metod stosowana do oznaczania śladowych iości wielu pierwiastków.

Z najnowocześniejszych metod zaprezentowano oznaczanie śladowych zawartości pierwiastków metodami PIXE (Proton Induced X-ray Emission) i PIGE (Proton Induced Gamma-ray Emission) z wykorzystaniem uruchomionego w 1992 roku w Krakowie akceleratora cząstek naładowanych VAN DE GRAAFF'A. Duże zainteresowanie oraz szeroki czynny udział specjalistów analityków z różnych ośrodków naukowych, instytutów i przemysłu wskazują na celowość organizowania takiej wymiany doświadczeń w przyszłości.

II POZNAŃSKIE KONWERSATORIUM ANALITYCZNE,
Nowoczesne metody przygotowania próbek i
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Poznań, Polska, 27-28/04. 1993

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O PROBLEMACH ZWIĄZANYCH Z ANALIZĄ SKANDU

W Instytucie Technologii Materiałów Elektronicznych podjęto próby oczyszczania skandu i opracowania metod oznaczania zanieczyszczeń w skandzie.

Rodzaj zanieczyszczeń, ich stężenie i rozkład jest ściśle uzależniony od technologii otrzymywania skandu. Skand występuje najczęściej w postaci krystalicznej, składa się zasadniczo z trzech frakcji: blaszki, żużla i proszku. Każda z tych frakcji różni się w sposób istotny ilością zanieczyszczeń a więc czystością. Około 10% ogólnej masy stanowią blaszki i to jest frakcja najczystsza. Najbrudniejszy jest żużel, a jego jest najwięcej; ze względu na tę niejednorodność skand jest materiałem trudnym analitycznie. Wymaga stosowania różnych, wzajemnie uzupełniających się technik analitycznych.

Pierwszym etapem analizy skandu jest analiza jakościowa i półilościowa. Wykonuje się ją metodami spektrometrii masowej i spektrografii emisyjnej. Spektrometria mas (SSMS), jako metoda wielopierwiastkowa, pozwala w jednym akcie analitycznym, oznaczyć prawie wszystkie zanieczyszczenia pierwiastkowe. Jest to jednak wzbudzenie iskrowe co powoduje, że metoda ta obarczona jest dużym błedem. Bardzo ważną zaletą metody SSMS jest wysoka wykrywalność: od ppm do ppb. Ta cecha kwalifikuje ją przede wszystkim do analizy bardzo czystych materiałów. Gdy suma zanieczyszczeń wynosi maksymalnie 100 ppm można ją traktować jako metodę ilościową.

Przy wyższych stężeniach jest to metoda półilościowa. W takich przypadkach stosowano metodę Absorpcyjnej Spektrometrii Atomowej (AAS). Zadaniem analityka jest podanie informacji o skuteczności czyszczenia w poszczególnych

etapach procesu technologicznego. Bardzo dobrze nadaje się do tego metoda Emisyjnej Analizy Spektralnej (AES). Kompleksowe potraktowanie problemu analitycznego umożliwiło przy pomocy wzajemnie uzupełniających się technik analitycznych, takich jak: SSMS, AAS i AES ukierunkowanie procesu czyszczenia skandu podawaniem informacji o każdym cząstkowym etapie technologii.

Tekst wystąpienia opublikowano w materiałach konferencyjnych:
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śladowych zawartości pierwiastków", Poznań, 1993.

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ELECTRON SPIN RESONANCE IN ALKALI METALS AND Fe INTERCALATED FULLERIDES

Materials based on alkali metals intercalated fullerences exhibit pronounce ESR effects. The spin resonance lines which may be observed below and above superconducting transition are interpreted as originating form carriers localized on fullerene molecules and from carriers spread over the neighboring alkali metals cations. The experiments performed on samples with strongly distorted crystalline structure showed that localized on molecule states are responsible for superconductivity.

In contrast to superconductivity, iron intercalated fullerities exhibit easily observed ferromagnetic behaviour.

In these materials, in contrast to alkali metals intercalated C_{60} where dopant ions are located in the interstitial positions, iron ions are bridging C_{60} molecules. The expected bonding mechanism introduces a longer range structural order, extending beyond pristine **fcc** C_{60} unit cell. The experiments indicate that for $x < 2$ the lattice can still be considered as a fcc distorted structure. The ESR measurements suggest interaction of localized Fe ions magnetic moments. The interaction leads to broadening of the observed resonance lines. Magnetization measurements of low iron concentration compound (x=0.5 and 1) exhibit the magnetic moment saturation in magnetic fields above 5 kOe. The saturation moments correspond to about 0.1 mB/f.u. at 40 K.

Materiał zaprezentowano na sesji posterów.
Pełny tekst wystąpienia opublikowany będzie w materiałach konferencyjnych.

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ESR INVESTIGATIONS OF Nd^{3+} CENTERS IN $SrLaAlO_4$

In this paper the results of ESR measurements on single crystal $SrLaAlO_4$ doped Nd^{3+} are reported. The ESR Nd^{3+} lines in the temperature range from 70K to 4K were observed. The values ΔH_{pp}=67mT, g=3.6667 0.05, ΔH_{pp}=176mT, g=1.5741 0.07 for magnetic field directions parallel and perpendicular to c-axis, respectively were obtained. The shape of measured lines were asymmetric.

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INVESTIGATION OF NEODYMIUM DOPED STRONTIUM LANTANUM GALLATE (SLGO) Pt. A. OPTICAL PROPERTIES

Czochralski-grown Nd-doped $SrLaGa_3O_7$(SLGO) crystals have been investigated for their optical properties. Absorbtion spectra in the range of 190 - 8000 nm and the luminescence spectra in the range of 200 - 800 nm were measured.

The crystals undergone laser pumping exhibit changes in the absorption spectra and these depend on the level and spectral distribution of irradiation energy. Differential changes of the absorption coefficient were investigated throughout the whole spectrum. Optical transitions corresponding to this effect have been indicated.

It was shown that changes in the absorption spectra have a significent influence on losing properties of Nd-doped SLGO.

Materiał zaprezentowano na sesji posterów.

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INVESTIGATION OF NEODYMIUM DOPED STRONTIUM LANTANUM GALLATE (SLGO)
Pt. B. LASING PROPERTIES IN FREE-RUNNING AND GIANT-PULSE GENERATION

Czochralski-grown Nd-doped $SrLaGa_3O_7$ (SLGO) crystals have been investigated for their lasing properties. It has been found that optical pumping leads to degradation of the optical parameters (longer absorbance, longer thresholds and lower efficiencies in laser action) and that thermal annealing of laser rods may limit these phenomena to a certain extent. A detailed study of lasing properties in a free-running mode indicated that optimization of laser head and especially of transmission of the output mirror is necessary for comparison of SLGO laser rods to *e.g.* Nd:YAG rods.

It was found that in the free-running mode laser thresholds are a few times larger for SLGO compared to YAG but the shape efficiency is lower only by several percents compared to a good quality YAG rods,and may be comparable to that of the average quality.

From the giant-pulse mode generation in the passive Q-switch modulation measurements it was evidenced that for SLGO laser rod the output energy is twice greater than in the corresponding YAG. This phenomenon can be utilised practically in laser pulsed systems which require high energies, *e.g.* surgery, stomatology, laser marking etc.

A continueous-work mode generation tests of SLGO laser rods have indicated. That further works on improvement of quality of this material is necessary for these applications.

Materiał zaprezentowano na sesji posterów.

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THE GROWTH AND CHARACTERIZATION OF $SrLaAlO_4$ SINGLE CRYSTALS

$SrLaAlO_4$ (SLAO) is one of the most promising materials for use as substrate for high temperature superconducting layers (HTSC) such as e.g. $YBa_2Cu_3O_{7-\delta}$ (YBCO). The material crystallizes in tetragonal structure of K_2NiF_4 and belongs to the group which can be described by the general formula $ABCO_4$.

In the paper, the method of preparation of charge material as well as the conditions of single crystal growth are presented. The crystals were grown from melt by the Czochralski method.

The inductive heating and system with iridium crucible and afterheater were used.

Undoped and doped with neodymium up to 5 at% SLAO single crystals of [100] orientation were obtained.

The chemical composition and lattice parameters of crystals were investigated by Atomic Absorption Spectrometry (AAS), inducted coupled plasma, electro microprobe analysis, Leco-gas analysis and X-ray diffraction. The chemical contents of atoms of good quality crystals differed from the exact stoichiometry, however, the changes along the grown crystal were not observed (changes were in the range of error).

Crystals of good quality, polycrystalline (opaque) and the rest of melt were always one phase. Lattice constants and chemical compositions of investigated samples were changed.

These include transmission characteristics within the wavelength range of 200-2000 nm for undoped and Nd-doped crystals and polariscopic images. The ESR measurements were carried out in the temperature range 70K to 4K.

Recent investigations have shown that the charge of crystal, the ratio of atoms with different valency ($A^{2+}B^{3+}C^{3+}O^{2-}$) play an important role in creation of SLA single crystal.

The substrates cut out from $SrLaAlO_4$ crystals have been successfully used for growth of high quality YBCO epitaxial layers.

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Materiał zaprezentowano na sesji posterów.

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THE GROWTH OF UNDOPED AND Nd-DOPED $SrLaGa_3O_7$ SINGLE CRYSTALS

Undoped $SrLaGa_3O_7$ crystals can be used as substrates for high-temperature superconducting layers. It was found, howewer, that these crystal doped with neodymium may also exhibit interesting lasing properties.

The crystals were pulled from the iridium crucible by the Czochralski technique . A suitable method of preparation of charge materials was applied. In order to achieve flat crystal-melt interface and adequate temperature gradients it was necessary to use appropriate system with good thermal insulation and carefully control pulling and rotation rates.

Undoped and doped with Nd at 5at% and 10at% single crystals were obtained. Their stoichiometry and dopant's distribution were investigated by the X-ray microprobe. The optical transmission characteristics and ESR spectra were also measured.

The Nd-doped crystals were used for manufacturing of laser rods which proved their potential use in pulse-work generation.

Materiał zaprezentowano na sesji posterów.

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THE GROWTH OF Er AND Fe-DOPED $LiNbO_3$ SINGLE CRYSTALS

This work presents some technological aspects connected with the growth and poling of $LiNbO_3$ doped with Er or Fe.

The crystals were grown from the melt by the Czochralski method. The Er contents were 0.1-0.3 at % and of Fe 0.17 at %, respectively. The obtained crystals were of Y or Z orientation and their dimensions were up to 82 mm in diameter and to 80 mm in length.

The optical transmission characteristics and the ESR spectra were measured and the influence of doping on the optical quality of the crystals was investigated. The Curie temperature was also measured for these crystals.

Both crystals were used for manufacturing of electropitc modulators, and Er-doped crystals were checked for their lasing properties. The influence of Er concentration on laser action was investigated.

Materiał zaprezentowano na sesji posterów.

PTWK: CONFERENCE AND GENERAL MEETING OF THE POLISH SOCIETY ON CRYSTAL GROWTH, Warszawa, Poland, 11-13/05.1993

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THE CZOCHRALSKI GROWTH OF BARIUM BORATE SINGLE CRYSTALS

Barium borate (BBO) is actually one of the best nonlinear optical material BBO single crystals are known in two modification: high temperature centrosymetric phase α-BaB_2O_4 and low temperature noncentrosymetric phase β-BaB_2O_4 with trigonal structure. Comparison of its properties with those of other nonlinear optical crystals shows that BBO is particularly useful in the generation of ultraviolet radiation of wavelength as short as 200 nm. It is also atractive material for SHG pumped by 1,06 μm neodymium laser radiation. Because BaB_2O_4 has a melting temperature of 1095°C and temperature transition is 925±5°C the β-phase crystals were obtained by flux method.

It was found that the β-BBO single crystals can be obtained by Czochralski method from undercooled melt below melting point.

Introductory results of obtaining β-BBO crystals by direct Czochralski method are presented.

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Materiał zaprezentowano na sesji posterów.

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Er:YAG AND CTH:YAG CRYSTAL GROWTH FOR LASER APPLICATION

In the paper the results on single crystal growth of the Er:YAG and Cr, Tm, Ho:YAG (CTH:YAG) are given. The crystals were grown from the melt by the Czochralski technique and the conditions of technological process are described.

The concentration of dopants was 1, 5.6, 0.36 and 33 at. % Cr, Tm, Ho and Er, respectively. The quality of obtained crystals is good enough for laser purposes. Output versus input energy characteristic as well as optical properties are presented.

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Materiał zaprezentowano na sesji posterów.

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PREPARATION AND PROPERTIES OF $SrLaGaO_4$ AND $LaGaO_3$ SINGLE PHASE POWDERS

Monophase powders of $SrLaGaO_4$ and $LaGaO_3$ were prepared by solid state reactions in air. Phase composition were investigated in ranges of temperature 900 - 1500°C and time 5 - 15 hours.

Quality of obtained powders were controlled by chemical, X-ray diffraction and X-ray microprobe analysis.

Conditions of synthesis of single phase compounds were found.

Results of investigations will be presented.

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PREPARATION $SrLaGaO_4$ AND $SrLaAlO_4$ DOPED BY Ba^{2+} IONS

We attempt to prepare single phase powders of $SrLaAlO_4$ and $SrLaGaO_4$ with addition Ba^{2+} ions by solid state reaction in air at 1500°C. Concentration of Ba^{2+} ions variaty in range 0.0-0.15 parts of one. We could't prepare single phase material with Ba in state of Sr. Some phase were formed in all samples with Ba. Preparation of materials were controlled by X-ray diffraction and X-ray micro-probe analysis.

Results of investigations will be presented.

This work was supported by State Committee for Scientific Research,
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the State Committee of Science and Technics for Scientific Research
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Materiał zaprezentowano na sesji posterów.

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ETCHING PROCESS OF $SrLaAlO_4$ CRYSTALS

The $SrLaAlO_4$ single crystals are used as a substrate for high temperature superconductor epitaxial layers with uniform physical properties. Good quality of epitaxial layers requires both crystallographic perfection and approriate physical properties of substrate material. The investigated crystals were grown by the Czochralski method at Institute of Electronic Materials Technology. The purpose of this work was to find an etching solution to investigate the defects in the crystals and to remove the destroyed layer after surface preparation (polishing, lapping and cutting). Surface microphotography has been done for different samples with high and low quality crystals in case of plane orientation (001) and (100). The polished and grinded surface of each sample were studying before and after etching. The investigations were taken by using optical, scanning electron microscopes and X-ray diffraction topography. The optical microscope photographs were obtained in case of reflected, transmitted light bright field and reflected light both for differential interference and polarizing contrast.

Etching experiments with various solutions, temperature and time were carried to obtain the best condition of etching process for different crystallographic plane.

In some of (100) samples the present X-ray investigations revealed thin lamellar volume defects. It was stated a different interplanar spacing in the defect region.

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SPECTROSCOPIC INVESTIGATIONS OF LASER MATERIALS EXCITED BY GaAlAs LASER-DIODES

Investigation of spectral properties of itrium-aluminium garnets ($Y_3Al_5O_{12}$) doped with rare-earth ions Pr^{3+}, Nd^{3+}, Eu^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} and single crystals of $SrLaGa_3O_7$ and $SrLaAlO_4$ doped with Nd^{3+} have been carried out. The crystals were grown by Czochralski - technique in the Institute of Electronic Materials Technology.

The absorbtion spectra of these crystals were measured in the wavelength range of 0,2 to 20 um, whereas the emission and excitation spectra in the range of 200 to 800 nm respectively. These investigations becames the basis for characterization of the above materials as active lasing media.

With the exception of Pr^{3+}:YAG and Eu^{3+}:YAG a strong absorption bands in the wavelength range of 780 to 840 nm have been recorded in all investigated crystals and this enabled the analysis of efficiency of selective pumping by GaAlAs laser-diodes.

In some of (100) samples the present X-ray investigations revealed thin lamellar volume defects. It was stated a different interplanar spacing in the defect region.

The work was supported by the
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Materiał zaprezentowano na sesji posterów.

PTWK: CONFERENCE AND GENERAL MEETING OF THE POLISH SOCIETY ON CRYSTAL GROWTH, Warszawa, Poland, 11-13/05.1993

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THE GROWTH RATE AND MASS CHANGES OF QUARTZ CRYSTAL IN HYDROTHERMAL PROCESS

A technique of quartz crystal growth at a constant rate has two advantages. Firstly, a number of crystals having proper sizes is usually higher because we know how to calculate the required growth period and, secondly, we can get material with uniform distribution of Q factor, the principal quality quartz parameter. In fact, it is a difficult experimental problem to establish the appropriate hydrothermal conditions which would crystals to grow at a constant rate. From practical point of view, it is useful to begin research with rate measurement for processes where temperatures are kept constant.

This work presents a method of intentional microinclusion bands generation which was used to measure the growth rate of standard quartz bar. The simplified geometrical model for this crystal was introduced and the three-dimensional crystal expansion was described. As the results, the variations of growth rate, growth surface and crystal mass were determined as time dependence functions.

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Materiał zaprezentowano na sesji posterów.

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SINGLE CRYSTALS WITH K_2NiF_4 STRUCTURE FOR HTSc SUBSTRATES

Several single crystals of K_2NiF_4, structure with general formula (A'A")BO_4, where A' = alkaline earth element as Ca,Sr or Ba, A"=rare earth element or Y, and B=Al, Ti, Ga, Ta, Cr, Fe or Ni, are an attractive choice as the substrate material for the growth of high Tc thin films. Single crystals of $CaNdAlO_4$, $SrLaAlO_4$ and $SrLaGaO_4$, were obtained by Czochralski method.

Crystallization of these materials needs a special care when choosing a proper starting composition of the melt. It is due to the fact, that in this crystal structure large two - or three - valent ions (A' or A") can be placed at crystallographically equivalent positions and therefore local disturbance of stoichiometry can involve creation of defects (e.g. oxygen vacancies) in the crystal structure. For the same reason the crystallization process is very sensitive to growth conditions such a pulling rate or speed of rotation. Moreover tetragonal symmetry and layered structure of these crystals are responsible for the observed strong anisotropy of growth.

Similar crystals containing Fe or Ni ions instead of Al or Ga, which could be also interesting as HTSc substrates, cannot be grown by Czochralski method in the RF furnace due to their high electrical conductance of the melt. Crystallization of $SrLaFeO_4$ and La_2NiO_4 was performed by floating zone method with optical heating using starting materials in form of synthesized ceramic rods. Crystals obtained up to now were strongly defected (with internal strains and twins) and their quality is too poor for HTSc substrate application.

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Materiał zaprezentowano na sesji posterów.

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IMPROVEMENT OF PHYSICAL PROPERTIES SI-GaAs CRYSTALS BY HEAT TREATMENT

Thermally stable semi-insulating (SI) GaAs substrates are required for integrated high speed devices by ion implantation or epitaxy as well.

For these purposes big SI-GaAs crystals with high electrical parameters (ρ, μ), good homogeneity, low density of defects structure and 75..100 mm in diameter are necessary.

The most popular method for manufacturing of SI-GaAs is the Liquid Encapsulated Czochralski method (LEC).

High resistivity material is obtained by compensation of shallow donors and acceptors with deep acceptor (Cr) or EL2 deep center.

"As grown" crystals show high inhomogeneity and sometimes low electrical parameters and for this reason are not useful in application. Improvement of physical parameters and homogeneity is possible by post-growth single crystals annealing.

According to the thermal annealing process (temperature, cooling rate) it is possible to change some material parameters in the wide range (*e.g.* $\rho = 10^{-2}..10^{7}$ Ωcm).

In this work GaAs single crystals obtained under different technological process conditions have been annealed in temperature 800..1150^{o}C.

More than 100 GaAs crystals, grown by low-pressure (LP-LEC) or by high-pressure (HP-LEC) technique, undoped or Cr, V, In doped have been investigated.

The physical properties were assessed by measurement of the following parameters:

- resistivity, mobility and carrier concentration,
- EL2 centers concentration,

» carbon concentration,
» resistivity distribution along crystal diameter,
» EL2 centers mapping on the whole wafer,
» dislocation density EPD.

Influence of heating treatment on improvement of physical properties has been observed. It allowed to obtain high quality SI-GaAs single crystals.

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SEMI-INSULATING InP SINGLE CRYSTALS

Semi-insulating indium phosphide becomes technologically important as the material for integrated opto-electronics and high-speed microelectronic devices. For these applications, SI-InP wafers are used either as substrates for epitaxial layers or directly in ion-implantation technologies for manufacturing MISFET's, HEMT's and integrated circuits.

For the ion-implantation lightly Fe compensated or undoped SI-InP is necessary.

In this paper we present the results of our research concerning synthesis InP, crystal growth, doping and thermal treatment.

SI-InP crystals have been obtained by heavy iron doping or by thermal treatment of slightly Fe-doped or undoped material.

Undoped InP crystals after annealing in the range of temperature $700^{\circ}C..900^{\circ}C$ show different behaviour according to the carrier concentration and compensation ratio (CR).

Crystals with carrier concentration $n < 3 \times 10^{15} cm^{-3}$ and $CR < 0.5$, after heat treatment become semi-insulating with resistivity $> 10^{7} \Omega cm$.

In these crystals Fe atoms was not detected by EPR method.

The activation level of 0.89 eV observed in electrical measurements is probably, according to the literature data, connected with the presence of phosphorus antisite defect P_{In}.

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SEMI-INSULATING 100 mm GaAs SINGLE CRYSTALS - CRYSTAL GROWING AND PROPERTIES

Semi-insulating 100 mm in diameter, GaAs single crystals have been grown by the LEC method.

The crystal growth conditions have been investigated for the following techniques:

1. The Low-Pressure (LP-LEC) technique with application of "injection method" for GaAs synthesis.
   The combined synthesis and crystal growth process was carried out in Polish made puller GK-2 equipped by one resistance heater and optical control system of crystal diameter.
2. The High-Pressure (HP-LEC) method with application of "in situ" synthesis in French made installation type Galaxie Mark IV.
   This equipment is provided with the weigh control of crystal diameter system. Two heaters are used for modeling of thermal gradient in the crystallized GaAs liquid.

The parameters of single crystals after annealing were as follows:

$\rho > 10^7$ Ωcm,

$\mu > 4000$ cm^2/Vs,

EPD $< 10^5$ cm^2 (characteristic cell structure).

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EFFECTS OF CARBON ON THE ELECTRICAL PROPERTIES OF SEMI-INSULATING GaAs SINGLE CRYSTALS

Electrical properties of semi-insulating gallium arsenide are determined by the balance between the deep donor level EL2, shallow donors and shallow acceptors.

In current high-purity material, after full boule anneal, the EL2 concentration is uniform and reproducible at $(1.15..1.2)x10^{16}cm^{-3}$.

The only detectable impurities are carbon and sulphur. Thus the resistivity of the material can be controlled by the carbon concentration assessment.

In this work we have investigated the effects of carbon concentration in semi-insulating GaAs grown by the Low Pressure (LP-LEC) and High Pressure Liquid Encapsulated Czochralski (HP-LEC) techniques.

Comparison are made between measured electrical properties of LP-LEC and HP-LEC GaAs crystals undoped and intentionally carbon doped.

Starting from the pure materials (Ga, As, B_2O_3) semi-insulating GaAs is reproducible obtained with carbon concentration $(2..3)x10^{15}cm^{-3}$ independently of crystal growth method.

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Materiał zaprezentowano na sesji posterów.

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INVESTIGATION OF INFRARED ABSORPTION SPECTRA IN UNDOPED GaAs

It is well known that several native defects and impurity complexes occur in GaAs grown by liquid encapsulated method (LEC). Some of them like e.q. EL2 are very important topic as they control semi-insulating parameters of the material. Many of those defects show characteristic absorption in infrared region.

For this reason low temperature absorption measurements are very useful for investigation of these defects.

In this work low temperature absorption measurements in range 4000..500 cm^{-1} has been done. Several undoped GaAs crystals grown under different technological conditions have been investigated.

The influence of heat treatment of the crystals on the absorption spectra was also observed.

The comparison of measurement results with technological conditions of crystals growth allowed us to make some suggestion on the origin of some peaks.

A new infrared absorption peak at 1162 cm^{-1}, which seems to be related with carbon, has been observed. The correlation between carbon concentration and absorption coefficient of the peak at 582 cm^{-1} has been obtained.

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A NOVEL METHOD OF CRYSTAL GROWTH BY PHYSICAL VAPOUR TRANSPORT

Optimal thermal conditions for directional crystallization of CdTe from the vapour phase with no contact between crystal and ampoule wall are presented.

A novel method of in-situ "nucleation" and growth by physical vapour transport has been developed and applied to CdTe. The main feature of this method is the technique for formation of a perfect seed in the first stage of the crystal growth procedure. Large, high quality single crystals of CdTe up to $8cm^3$ both p-type and n-type have been grown using this method. The etch pit density on the {lll} crystal faces was found to be as low as $2 \times 10^3 cm^{-2}$.

The way of extension of this method on qrowth of larger crystals is proposed. The new approach to constitutional supersaturation criterion at high temperature vapour growth is presented.

Pełny tekst wygłoszono, na zaproszenie organizatorów, na sesji referatów.

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GROWTH OF TERNARY AND QUATERNARY ZnSe COMPOUNDS WITH TRANSITION METALS BY CHEMICAL VAPOUR TRANSPORT

Halogen transport method was applied to grow the crystals of solid solutions of ZnSe and transition metals at the temperature far below the melting point and phase transition temperature. The crystals of ZnSe, ZnFeSe, ZnNiSe of different concentrations were obtained. The technological parameters and the shape of the quartz reactor were chosen for growth a large bulk crystal by selfnucleation; the transparent quartz furnace enabled the control of nucleation by visual observation; the parameters of the crystal growth were determined. The crystal quality was estimated by X-ray diffraction method and observation in polarised light. The distribution of transition metals was determined by electron microprobe analysis and energy dispersive X-ray fluorescence analysis.

First quaternary ZnFeSnTe and ZnFeSeS were grown. The distribution of components was measured. The electrical measurements show that the crystals are of high resistivity; the results of transport phenomena measurements will be the subject of another presentation.

Materiał zaprezentowano na sesji posterów.

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EFFECT OF FLUID FLOW ON THE CONCENTRATIONAL NONUNIFORMITIES IN $LiNd_{1-x}Y_xF_4$

In this work we investigated the effect of growth conditions on structural and compositional nonuniformities of $LiNd_{1-x}Y_xF_4$ grown by Bridgman-Stockbarger technique. The Skaning Electron Microscopy study of the crystal was conducted. The Nd^{3+} in the crystal was studied in samples cut from the crystal and analyzed using ESR spectroscopy. The structure of convection vortices in various stags of crystal qrowth was investigated. The problem of the LiF-rich phase solidification in relation to thermal qrowth conditions and shape of the liquid/solid interface is discussed. The effect of the crucible construction on the shape of the solid/liquid interface is demonstrated.

Materiał zaprezentowano na sesji posterów.

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THE IDENTIFICATION OF DISLOCATION IN SILICON AND GALLIUM ARSENIDE CRYSTALS BY COMPARISON OF EXPERIMENTAL AND NUMERICALLY SIMULATED DOUBLE-CRYSTAL IMAGES

The important reason for attempts to obtain mostly realistic simulations of dislocation images in X-ray diffraction topography is a great importance of precise identification of dislocation in studying many crystalline materials. In the present paper we use the improved technique of simulation of dislocation images in the case of Bragg-case double-crystal topography of gallium arsenide and silicon crystals.

The main improvement of the present simulation was the calculation of integrated images taking into account the finite divergence of the incident beam in the real double-crystal experiments. It was realized by adding up to 100 images slightly differing in the assumed angle of incidence, weighted by appropriate rocking curve describing the angular distribution of radiation reflected by the monochromator. The numerical program calculates the gradient of deformation field in isotropic approximation including the relaxation at free surface.

The comparison of theoretical and experimental images were performed in the case high sensitive double-crystal arrangements with Cu $K_{\alpha 1}$ radiation for silicon and gallium-arsenide. Both experimental topographs and the simulations were obtained for asymmetrical reflections from different equivalent planes.

The present simulations were in much better agreement with the experimental images of dislocation than those assuming plane-wave incident wave. They

confirmed the preliminary identification of their type based on the Lang topography, and allowed the determination the sense of Burgers vector.

The compound plane-wave images contained both systems of interference fringes and a characteristic black-white rosette seemed to correspond to the direct dilatation-orientation contrast. The contrast of the rosette was reversible with the change of the rocking curve slope and the sign of the Burgers vector. The effect of integration consist in averaging the interference fringes. It was more distinct in the case of lower absorbing silicon, where the interference fringes more strongly contribute to the image of dislocation.

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IMPROVEMENT OF CZOCHRALSKI METHOD FOR THE SILICON CRYSTAL GROWING

Main Czochralski method for Si crystal growing development trends are discussed in association with actual technical level of the crystal growing techniques.

Quality of the monocrystals is discussed for the monocrystals grown with influence of magnetic field. Such quality factors as dope homogenity and impurities segregation in composition with the structural properties are discussed.

Quality properties are discussed for monocrystals grown in Institute of Electronic Materials Technology as well as by other manufacturers.

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PHOTOLUMINESCENCE PROPERTIES OF POROUS SILICON PREPARED BY ELECTROCHEMICAL ETCHING OF Si EPITAXIAL LAYER

The photoluminescence (PL) properties of porous layers prepared by anodization of p/p^+ silicon epitaxial wafers are presented. The shift of the PL spectrum towards shorter wavelength due to the porosity increase and the experimental dependence of the PL maximum position on HF concentration during anodization are shown. Degradation of the PL intensity dependence on the storage time is described.

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VISIBLE LUMINESCENCE FROM POROUS SILICON

This work present results of investigation of the temperature dependence of visible luminescence porous silicon layers prepared by anodization in hydrofluoric acid. Luminescence spectra were measured in the temperature range between 40 K and 350 K. Room temperature reflectivity spectra have been also measured in vacuum ultraviolet radiation range from 4 eV to 12 eV.

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QUANTUM CONFINEMENT EFFECT AS A MECHANISM OF GENERATION OF VISIBLE LUMINESCENCE FROM POROUS SILICON

In this paper we present photoluminescence (PL), photoluminescence excitation (PLE) and decay times measurements on anodically etched boron-doped Si. To explain our resalts we assumed model in which the multi-barrier structure is formed by Si crystal (quantum well) surrounded by Si quantum wires, oriented perpendicular to the sample surface with diameters in the range of 2 to 12 nm (barrier region). The visible photoluminescence originates from radiative recombination between discrete energy levels in quantum well.

Materiał zaprezentowano na sesji posterów.

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X-RAY TOPOGRAPHIC INVESTIGATION OF CELLULAR STRUCTURE AND ITS RELATION TO ANOTHER DEFECTS IN VARIOUS TYPES OF GaAs SINGLE-CRYSTALS

The LEC-, GF- and Bridgman-grown GaAs single-crystals with different types of doping were characterized by complementary use of double-crystal, Lang and Berg-Barrett methods of X-ray diffraction topography. The samples were cut out from different parts of the crystals perpendicular and parallel to growth axis.

The most frequent occurrence of the cellular structure was observed in the case of not doped and low doped crystals grown by LEC method. It is otherwise the defect characteristic to the material of this kind delivered by various producers. The cellular structure was not developed in the samples cut from starting parts of the crystals. The cellular structure was observed occasionally in the case of heavy Si-doped GF crystals in the neighborhood of large precipitates or dendrites.

Using double-crystal method we found that the existence of cellular structure is connected with considerable deformation of the lattice. Usually no significant misorientation is observed between particular cells. It occurs between grains containing a number of cell, forming in extreme situations a mosaic structure. It was confirmed (also confronting with selective etching method) that most of dislocations was grouped in the cell walls. Some topographic evidences for the simultaneous existence of the precipitates were found.

We found a strong connection of the occurrence of the most dense, low-diameter cells and the lattice strain. On the other hand the areas without cells or with larger ones correspond to the region with smaller strain. That may suggest that

the cellular structure is formed in regions with greater thermal stress present in the growth process. The topographs of the longitudinally cut crystals did not reveal neither any elongation of cells nor any connection of the cells with the shape of the growth front.

Presently investigated LEC- and GF-grown, heavy indium doped crystal did not reveal the cellular structure, but the forms due to the "cellular growth". The last phenomenon is due to the constitutional supercooling, causing that some parts of the crystal grow faster and form long channels, built of {111} facets. In actual case it was confirmed that the cellular growth occur at higher dopant concentration when thermal gradient is low.

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ANISOTROPIC MAGNETIC SUSCEPTIBILITY OF NEODYMIUM SUBSTITUTED $SrLaAlO_4$ CRYSTALS

The uniaxial anisotropy of magnetics properties of single crystals $SrLa_{1-x}Nd_xAlO_4$ (with x=0,01 and 0,05) has been found from the measurements of temperature dependences of magnetic susceptibility. Results of measurements, with magnetic field along *a*- and *c*- axis, are compared with the similar data obtained for $CaNdAlO_4$ crystal. The succesful description of experimental data was done in frames of the crystal field approximation. The anisotropy of magnetic susceptibility appears due to crystal field acting on magnetic neodymium ions in a system without exchange interactions.

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EUROPEAN CONFERENCE PHYSICS OF MAGNETISM'93.
Strongly Correlated Electron Systems
Poznań, Poland, 21-24/06. 1993

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ANISOTROPIC EFFECT OF NEODYMIUM IN $CaNdAlO_4$ AND $SrLaAlO_4:Nd^{3+}$ CRYSTALS

In this paper the rsults of the electron spin resonance (ESR) measurements on single crystal $SrLaAlO_4$ doped with Nd^{3+} in range 1 to 5 at.% are presented.

The shape of measured lines is asymmetric and in case of undoped $CaNdAlO_4$ there is one very broaded anisotropic absorption ESR line.

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BI-DOPED GARNET FILMS INVESTIGATION BY MICROWAVE TECHNIQUES.

Fast recent development of epitaxial film technology has lead to the creation of Bi-substituted garnet films having high Faraday rotation, a small optical absorption and a small ferromagnetic resonance linewidth ΔH. This is very important for observing interaction between optical waves and magnetic excitations. Maximum diffraction efficiency per unit microwave power was obtained using Bi-doped films (interaction of guided light and magnetostatic waves was investigated).

For this work magnetic films $(Bi, Lu)_3Fe_5O_{12}$ were grown on (111)-oriented gadolinium gallium garnet substrates by liquid-phase epitaxy. Small ΔH (less than 1[Oe]) was measured.

The ferromagnetic resonance measurements were carried out at X-band spectrometer (Bruker ESP -300 equipped by continuous flow criostat Oxford Instrument ESP 900) . Magnetic anisotropy constants were determined as a temperature function.

The propagating MSW were excited and detected by microstrip lines (from 0,5 to 4 GHz frequency range).

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INTERFACE STRUCTURE, HALL EFFECT, MAGNETOSTRICTION AND MAGNETORESISTIVITY IN Fe/Zr LAYERS

Fe/Zr multilayers of the thickness ratio $d_{Fe}/d_{Zr}=0.5$ and 1, with the modulation wavelengths ($d=d_{Fe} + d_{Zr}$) varied in the range $2.5 \leq d \leq 80$ nm, were sequentially deposited by rf sputtering from Fe and Zr targets. The total thickness of the film stacks was 240 nm. The growth of amorphous phase in as-deposited and annealed samples was controll by resistivity, X-ray diffraction and electron conversion Mössbauer spectroscopy (CEMS). From CEMS measurements, in as-deposited samples, were identified three crystalline magnetic phases: bcc-Fe (H_1=33T) from iron sublayer and two interfacial phases high (H_2=29T) and low (H_3=12T) magnetic. The strain-modulated ferromagnetic resonance method SMFMR, which via magnetoelastic coupling is phase sensitive, support phase segregation obtained from CEMS spectroscopy. The spontaneous Hall coefficient R_S and magnetostriction λ_S, (both originated from spin-orbit interaction) are practically independent of the thickness of iron layer for $d_{Fe} \geq 10$ nm. For film with d_{Fe}=5 nm R_S and absolute value of λ_S increase three times, due to formation of the interfacial phases. Magnetoresistivity (at the room temperature) is extremely low ($\Delta R/R \approx 0.09\%$).

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SYNTHEZIS NEW SOLID SOLUTION IN Sr-Ba-La-Ga-O and Sr-Ba-La-Al-O SYSTEMS

In the last time there has been a big interest to materials for substrates of HTSc thin films. There are some classes of this materials representing the most interest: BCO_3 and $ABCO_4$, where A-Sr,Ca; B-Ln; C-Al, Ga. The most attractive properties have compounds $SrLaAlO_4$ and $SrLaGaO_4$. On substrates from crystals of these compounds thin $YBa_2Cu_3O_{7-d}$ films with good superconducting properties were produced. However, there is not satisfied matching in lattice constants between film and substrate, so to increase a(b) parameters Ba^{2+} ions have been chosen to replace Sr^{2+} ions. Barium has greater ionic radius equal to 0.134 nm than strontium 0.112 nm, so the ratio of Ba/La ions is greater than Sr/La, and a(b) lattice parameters can increase.

Compounds of $SrLaGaO_4$, $SrLaAlO_4$ and solid solutions $Sr_{1-x}Ba_xLaGaO_4$, $Sr_{1-x}Ba_xLaAlO_4$, where x=0.05, 0.10, 0.15 were investigated. As starting materials $SrCO_3$, BaO_2, La_2O_3, Ga_2O_3 and Al_2O_3 with purity not less then 99.98 were used. Compositions of initial chemicals was controlled by thermal analysis (derivatograph Q - 1500D) and chemical analysis. Compounds and solid solutions prepared by solid state reactions and stoichiometric mixtures were heated at temperature 1400°C in the air during 25h.

Synthesized samples were investigated by X-ray diffraction (difractometer DRON-4M, filtering CuKa irradiation) and X-ray microprobe analysis (SEM CamScan S4/DV with EDX Link AN85/S). SEM with microanalysis system

worked under the following conditions: accelerating voltage 15 kV, work distance 33 mm, absorbed current 0.5 nA, live time 100 s, calibration element - Co. The following compounds: $BaCO_3$, $SrCO_3$, $LaP0_4$, Al_2O_3, Ga_2O_3 were used as standards. Some diffraction patterns and results of microanalysis are presented. Some unidentified peaks on all diffraction patterns, where x O are found. Lattice constants for compounds of $SrLaGaO_4$, $SrLaAlO_4$ and solid solutions $Sr_{1-x}Ba_x$-$LaGaO_4$, $Sr_{1-x}Ba_xLaAlO_4$, where x=0.05 were measured by X-ray diffraction method. Results for compounds and solid solutions are presented in Table 1.

Table 1. Lattice constants data

Compound	Lattice constant, nm	
	a	c
$SrLaAlO_4$	0.37550(3)	1.26314(9)
$SrLaAlO_4$ [4]	0.3754	1.263
$Sr_{1-x}Ba_xLaAlO_4$	0.37556(3)	1.26316(9)
$SrLaGaO_4$	0.38409(4)	1.26804(8)
$SrLaGaO_4$ [4]	0.3843	1.2681
$Sr_{1-x}Ba_xLaGaO_4$	0.38414(4)	1.26807(9)

The differences between the lattice parameter values of comoounds $ABCO_4$ type and solid solutions, where x=0.05 are in the range of experimental errors. So, it can be concluded, that Sr^{2+} is not substituted by Ba^{2+} ions. Microanalysis results showed that the unidentified reflections in the diffraction patterns belong to phases $Sr_{2(1-x)}Ba_{2x}LaGaO_5$ and $Sr_{2(1-x)}Ba_{2x}LaAlO_5$ and x=0.33 for both 'solid solutions.

Then the syntheses of these solid solutions were made using the previously described experimental conditions assuming B^{2+} ions content equal to x=0.33 for both compounds. Diffraction patterns of these solid solutions are presented. The lattice constants were measured for $Sr_{1.33}Ba_{0.66}LaAlO_5$ compound and are equal to a=0.97453(4) nm and c=1.11017(8) nm. It should be noted that some compounds of A_2BCO_5 type are known and crystallize in tetragonal structure (space group I4/mcm).

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MODELING OF OUTPUT POWER IN SOLID STATE LASERS

Modeling of output power in solid state lasers, taking into account spatial field effects, is presented. Laser characteristics revealing the dependence of the optimal output mirror reflectivity, providing maximal power efficiency of the system, on the system parameters, geometry of the resonator as well as the position of the active medium in the resonator are obtained for conventional lasers as well as for lasers with gaussian mirror.

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