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SYNCHROTRON SECTION TOPOGRAPHY OF IMPLANTED LAYERS

Back-reflection and transmission synchrotron section topography was applied for examination of silicon implanted with high energy protons and α -particles. The experiments were performed using white beam X-ray radiation from the DORIS storage ring. The wave front was limited by 5 μm slit. A number of section patterns in different reflections were obtained.

The important aim of the present experiments was the direct localization of the destroyed layer and the evaluation of its thickness. In the case of crystals implanted with 1 and 1.6 MeV protons and 4.8 MeV α -particles the localization of the destroyed layer was possible as back-reflection geometry while for 9 MeV proton implantation only in transmission.

The Bragg-case section topographs revealed a characteristic pattern caused by the layer structure in the sample. A number of maxima due to shot-through layer and the deformation field in the vicinity of the destroyed layer was observed. This pattern was changed in different reflections but it was directly dependent on the energy of the implanted ions. The correlation of the experimental pattern and the theoretical one, obtained by numerical integration of Takagi-Taupin equation, was achieved.

In the case of samples implanted with the ions of lower energies the transmission section topographs exhibited slightly deformed Kato fringes and additional strong interference contracts coming from the implanted region. The samples bombarded with 9 MeV protons produced a very distinct moiré fringes due to the interference of the beams diffracted by two parts of the crystal separated by the destroyed layer.

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COMPATIBILITY OF $\text{YBa}_2\text{Cu}_3\text{O}_{7-\sigma}$ THIN FILMS AND ABCO_4 SUBSTRATES FOR DIFFERENT ORIENTATIONS

In the last year a great interest appeared in YBCO thin films preparation on different substrate materials. Preparation of epitaxial film is a very difficult problem. There are many requirements that must be fulfilled by the substrate materials. Main problems are lattice mismatch (misfit) and similarity of structure. From paper [1] it follows that difference in interatomic distances and angles of substrate and film is a more important problem than similarity of structure. In this work we present interatomic distances and angle relations between substrate materials belonging to ABCO_4 group (where A= Sr or Ca, B = rare earth element, C = Al or Ga) of different orientations and YBCO thin films.

There are many materials used as substrates for HTsC thin films. ABCO_4 group of compounds is characterized by small dielectric constants (necessary for the microwave applications of HTsC films), absence of twins and small misfit [2]. Three most interesting compounds CaNdAlO_4 , SrLaAlO_4 and SrLaGaO_4 were investigated. All these compounds are of pseudo - perovskite structure with space group $I4/mmm$. This structure is very similar to the structure of YBCO. SLG substrate has the lowest misfit (0.3%) and dielectric constant.

For preparation of thin films on substrates of this group of compound plane of $\langle 100 \rangle$ orientation is mainly used. Good quality films of $\langle 001 \rangle$ orientations are obtained [3]. In this case not only a-a misfit plays a role, but c-3b misfit is very important too.

Sometimes for preparation of thin films substrates of $\langle 001 \rangle$ and $\langle 110 \rangle$ orientations were used. On these substrates good films of YBCO with the same orientations were manufactured [3].

Different misfits for different YBCO faces have been analyzed. It has been found that the mismatching factor for (100) face is very similar to that for (001) face so there is a possibility of preparation of thin films on both orientations.

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GROWTH AND OPTICAL PROPERTIES OF Cr⁴⁺:YAG SINGLE CRYSTALS

Tunable solid state lasers working in the 1.2 - 1.6 μm range of great interest for medicine, optical communication, remote sensing and spectroscopic applications. One of the relatively new laser material of this type is tetravalent chromium ions (Cr⁴⁺) doped yttrium aluminium garnet crystal (YAG). Strong absorption in the 1 μm band offers a possibility of efficient pumping using powerful IR AsGa laser diodes and a construction of all solid state device. Cr⁴⁺:YAG single crystals were grown in ITME laboratory in Warsaw by Czochralski technique. In this paper details of the material technology are discussed. The effect of crystal growth on optical properties of crystals was studied by monitoring absorption spectra and argon or Nd³⁺:YAG laser excited emission spectra in the near infrared range. Further improvement in IR emission intensity has been obtained through crystal annealing in the oxygen containing atmosphere. Finally new results on Cr⁴⁺:YAG laser will also be presented.

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CRYSTALLIZATION AND PROPERTIES OF DOPED SrLaAlO₄ AND SrLaGaO₄ CRYSTALS

SrLaAlO₄ (SLA) and SrLaGaO₄ (SLG) crystals of general formula ABCO₄ have been grown by the Czochralski method. The quality of SLA and SLG crystals depend strongly on axial gradient of temperature and growth and rotation rates.

High quality crystals were obtained at axial gradient of temperature near crystal-melt interface lower than 50°C/cm, growth rate 1-3 mm/h and the rotation rate 10 - 20 rpm [1].

Strong anisotropy in morphology of SLA and SLG single crystals, grown by the Czochralski method, is clearly visible.

On the basis of our considerations for ABCO₄ type of the tetragonal crystals we find that there can appear {001}, {101} and {110} faces for ionic type model [2].

To investigate facets formations the crystals were doped with Cr³⁺, Er³⁺, Pr³⁺, Ba²⁺. Greater size ion of chromium, which is substituted for Al³⁺ clearly induces faceting. There {110} faces appear easily and SLA crystals crack even, when the amount of Cr is below 0.3at.%. SLG single crystals are not so sensitive to the content of chromium ions. It was also found that if {110} face appears at the beginning of growth process the crystal changes its color on the plane {110} but it happens only on the shoulder part. The projection of {110} face has a great amount of oxygen positions which can be easily defected.

Pure and doped SLA and SLG crystals measured by EPR in the <110> direction show more intensive lines than in other directions which suggest that the amount of oxygen defects on the {110} plane is higher.

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CRYSTALLIZATION AND SYNTHESIS OF SrLaAlO₄, BaLaAlO₄ AND THEIR SOLID SOLUTIONS

SrLaAlO₄ (SLA) crystallizes in tetragonal, I4/mmm, [1] and BaLaAlO₄ (BLA) in orthorhombic, P2₁2₁2₁, [2] structures. The compounds melt congruently and single crystals have been grown by the Czochralski method.

The influence of cation radii of Sr²⁺ (0.145nm) and Ba²⁺ (0.161nm) with coordination number 9 on the stability of crystal structure is investigated.

The results on single crystal growth of pure SLA and BLA and on one phase and solid solutions synthesis are presented.

SLA crystal of tetragonal structure is strongly anisotropic and the growing crystal is easy bounded by slowly grown {001}, {101} and {110} faces. The effect is not so clearly seen in the orthorhombic BLA crystals.

The synthesis of solid solutions is performed in the temperature range from 1350°C to 1450°C, in air. The total synthesis is found at 1450°C. Multiphase system is observed from 5 to 90 mol% BLA in SLA. In range from 50 to 70% BLA the cubic phase of perovskite structure with lattice parameter equal to 0.3788 nm is found. The well developed cubes of sizes about 5mm are observed on surface of polycrystalline pellets.

The defined chemical formula of crystals is LaAlO_3 doped with Sr and Ba. It should be pointed out that in this case the orthorhombic phase is not found. The orthorhombic phase has not been separated neither by solid-state reaction nor by the crystallization by the floating zone method. The melting temperature is about 2000°C . It can be concluded that the cubic phase is always formed at the gas-solid state interface.

The solubility of Ba in the tetragonal structure is lower than 5at.%.

The properties of crystals are discussed on the basis of X-ray, SEM and EPR measurements.

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SI-GaAs CRYSTAL GROWTH IN MODULATED THERMAL FIELD

The aim of this work was to find technological (thermal) conditions allowing to obtain SI-GaAs crystals of large and stable diameter of 3" and 4", weight 4.5 kg and 7.5 kg with good yield and process repeatability.

Crystals have been grown by HPLEC method on the high pressure puller GALAXIE MARK_IV. Multiheater graphite systems for 6 " and 8 " quartz and pBN crucibles have been constructed and optimized (for 5 kg and 8 kg of GaAs charge).

In these systems the following characteristics were obtained:

- axial temperature gradients above the melt as a function of ambient gas pressure for Ar and N_2 ,
- axial temperature gradients above the melt versus crucible position,
- temperature gradients inside the B_2O_3 encapsulant layer as a function of am-

bient gas pressure for Ar and N₂.

The process parameters for the Mark IV computer program have been optimized for "in situ" synthesis and HPLEC crystal growth process.

SI-GaAs crystals with 3" and 4" diameter have been obtained and the influence of the growth conditions on the crystal properties, especially on the structural quality, has been observed.

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PHYSICAL PROPERTIES OF CARBON DOPED SI-GaAs CRYSTALS

In this work we investigated the growth of semi-insulating GaAs crystals with control of carbon concentration. All crystals used in this study were grown by LP LEC or HP LEC techniques. The crystals were pulled from slightly As rich starting melt containing 7N Ga and 6N5 As situated in pBN or SiO₂ crucible.

The carbon was incorporated into the melt during synthesis or monocrystallization in the range $4 \cdot 10^{14} \div 2 \cdot 10^{16} \text{ cm}^{-3}$.

Electrical parameters of the crystals were measured by the Van der Pauw technique. Carbon concentration was evaluated from local vibrational mode infrared absorption for C_{As} with FTIR spectrophotometer at 77K, deep donor EL2 concentration by NIR absorption.

When the carbon concentration is low ($N_C < 0.5 \cdot 10^{15} \text{ cm}^{-3}$), many crystals become n-type with resistivity $\rho = 10^4 \Omega\text{cm}$. This value of carbon concentration was considered a critical one for obtaining high resistivity material, which does not have to be semiinsulating. If carbon concentration $N_C \geq 5 \cdot 10^{15} \text{ cm}^{-3}$ all obtained GaAs crystals were semi-insulating ($\rho = 10^7 \Omega\text{cm}$). The mobility as well as the resistivity is the parameter which decides about the properties of semi-insulating material.

For carbon concentration $N_c < 4 \cdot 10^{15} \text{cm}^{-3}$ the carrier mobility varied from 1000 to 6000 cm^2/Vs .

With $N_c = (4 \div 10) \cdot 10^{15} \text{cm}^{-3}$ most of the crystals have shown the mobility $\mu = (4000 \div 6000) \text{cm}^2/\text{Vs}$. When the carbon concentration was high, small decrease of the mobility was observed.

The influence of carbon concentration on thermal stability of SI-GaAs wafers and threshold voltage of FET transistors was also investigated.

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INFLUENCE OF TECHNOLOGICAL PROCESS ON SEMI-INSULATING BEHAVIOR OF UNDOPED InP CRYSTALS

Today, iron doping is the only reliable technique to make InP crystals semi-insulating with resistivities $\rho > 10^7 \Omega\text{cm}$. SI-InP crystals are of technological interest because they are used as a substrate for various promising electronic devices. Because of high Fe out-diffusion into the epitaxial layers undoped SI crystals should be more convenient for these applications. On SIMC-8 we presented the results concerning undoped semi-insulating crystals [1]. This material has been obtained by annealing of undoped bulk crystals with carrier concentration $n < 10^{15} \text{cm}^{-3}$. In conclusion we have stated that semi-insulating properties are not connected with residual Fe atoms, but with other factors.

In this work we have investigated the influence of the technological conditions on semi-insulating properties of undoped crystals. Special attention was paid to stoichiometry of InP melt and its influence on electrical parameters of the crystals. The crystals were grown from the melt with controlled nonstoichiometry. All crystals have been annealed at $900 \div 950 \text{ }^\circ\text{C}$ for $8 \div 75 \text{ h}$ under phosphorus pressure $1 \div 15 \text{ atm}$. The crystals were characterized by Van der Pauw technique, sheet resistance measurements, and NIR absorption for evolution of Fe concentration.

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GROWTH MORPHOLOGY OF TETRAGONAL $ABCO_4$ COMPOUNDS: THEORY AND OBSERVATIONS ON CZOCHRALSKI GROWN CRYSTALS

Tetragonal $ABCO_4$ compounds where A = Ba, Sr, Ca; B = rare earth elements and C = Ga or Al, are very suitable substrates for epitaxial growth of thin films of high-Tc superconductors. They are chemically stable and have very low dielectric constants. Moreover their lattices match very well with those of superconductors. In order to know the influence of the crystal structure on the crystal morphology, $CaYAlO_4$ has been selected as model. According to the Hartman-Perdok theory the F forms are in order of decreasing d_{hkl} : {002}, {101}, {103}, {110}, {112}, {200}, {123}. Calculation of attachment energies in an electrostatic point charge model yields theoretical growth forms. Because the Al-O bond is partially covalent, growth forms have also been computed as a function of the charge of oxygen, q_o . For the formal electric charges the growth form is planar following {001} with {101} as major and {110} as minor lateral form. At lower effective q_o theoretical growth forms are still tabular but the morphological importance of the lateral forms are

changing. At $q = -1.6$ le1 $\{110\}$ is much more important than $\{101\}$, and at $q \leq -1.4$ le1 $\{110\}$ becomes the most dominant lateral form, while $\{101\}$ has been replaced by $\{103\}$ as minor lateral form. CdNdAlO_4 , SrLaAlO_4 and SrLaGaO_4 have been grown by the Czochralski technique. At the convex crystal-melt interface facets of $\{101\}$ occur while the cylindrical part shows $\{001\}$ and depending on the growth direction $\{100\}$ ($// <100>$) or $\{110\}$ ($// <110>$). During the growth flat crystal-melt interfaces must be avoided. They produce inhomogeneities in colour caused by oxygen deficiency in the case of $\{110\}$ interfaces. Cracking parallel to $\{001\}$ interface occurs due to the strongly anisotropic character of the crystal structure. A flat interface parallel to $\{100\}$ is never stable during the conditions of Czochralski growth.

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CZOCHRALSKI GROWTH OF $\beta\text{-BaB}_2\text{O}_4$ (BBO) CRYSTALS

Low temperature phase $\beta\text{-BaB}_2\text{O}_4$ crystals (BBO) belonging to trigonal structure exhibit excellent nonlinear optical properties. High nonlinearity, large birefringence, transmission range between 190-3500 nm and damage threshold as high as 10 GW/cm² measured with 100 ps pulses make this material very useful for optical parametric oscillation and higher harmonics generation. Temperature transition to centrosymmetric phase $\alpha\text{-BaB}_2\text{O}_4$ is in 925°C while the melt point is $1100 \pm 5^\circ\text{C}$ so BBO crystals have been grown by flux technique. Growth process is very slow and the obtained crystals possess flux inclusions.

It was found possible to obtain $\beta\text{-BaB}_2\text{O}_4$ crystals by Czochralski method. Growth process is carried on from supercooled melt below the melt point. As a starting material specially prepared $\beta\text{-BaB}_2\text{O}_4$ powder was used. Suitable growth conditions i.e. temperature gradient near solid-liquid interface, pulling and rotation rates were found. Obtained β -phase crystals were confirmed by X-ray diffraction. Special cut samples with phase matching angle 22° respect to optic axis C were prepared for

SHG test. Green light 530 nm after irradiation with Nd:YAG beam was observed. Real structure of BBO crystals by X-ray high resolution diffraction was researched.

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SINGLE CRYSTAL GROWTH AND SPECTROSCOPIC PROPERTIES OF LiNbO_3 DOPED WITH Er^{3+} , Tm^{3+} , Yb^{3+} AND Pr^{3+}

Single crystals of LiNbO_3 doped with Er^{3+} , $\text{Er}^{3+} + \text{Tm}^{3+}$, $\text{Er}^{3+} + \text{Tm}^{3+} + \text{MgO}$, Yb^{3+} and Pr^{3+} were grown by Czochralski method starting from the congruent melting composition.

Uniformly doped, good quality crystals were obtained at low temperature gradients and pulling rate 1.5-2 mm/h and rotation rate 8-20 rpm. The crystals were grown either in Y or in Z direction. Their dimensions were up to 80 mm in diameter and up to 90 mm in length.

The as-grown crystals were regular and free of macroscopic defects. They were pulled in a separate operation. Electrodiffusion was not observed during pulling which was confirmed by X-ray microprobe analysis.

The absorption spectra of RE doped LiNbO_3 single crystals were studied in spectral range 28000 cm^{-1} - 2000 cm^{-1} at temperatures from 300 K to 15 K. The interaction between RE ions resulting in changing of absorption spectra structure for particular terms related to different ions has been observed.

It seems that doping of LiNbO_3 with different rare earth ions may broaden its potential application as a laser host material especially in integrated optics.

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THE CZOCHRALSKI GROWTH OF $\text{SrLaGa}_3\text{O}_7$, Nd^{3+} AND Pr^{3+} DOPED SINGLE CRYSTALS AND THEIR SPECTROSCOPIC PROPERTIES

Single crystals of $\text{SrLaGa}_3\text{O}_7$, undoped and doped with 5 at. % and 10 at. % of neodymium and 0.5 and 1 at. % praseodymium were grown by the Czochralski technique.

A heating system with good thermal insulation and iridium crucible and after heater was applied in order to achieve appropriate radial and axial temperature gradients in the crystallization chamber. Low temperature gradients and flat crystal-melt interface can provide good optical quality crystals free of internal stresses.

All single crystals were grown in [001] direction. The pulling rate was 1.5-3mm/h and the rotation rate 50 rpm. The composition and the dopant's distribution in the crystals were checked by X-ray microprobe. The crystals up to 25 mm in diameter and up to 80 mm in length were of good quality and the dopant's distribution was uniform. The doped crystals were investigated for their spectral and lasing properties. Absorption spectra in the range of 180-8000 nm and luminescence spectra in the range of 300-3000 nm were measured, then laser rods were cut and their generation properties were evaluated.

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C-BN & DIAMOND CRYSTALLIZATION UNDER REDUCED PRESSURE. CONFERENCE, Jabłonna, Poland 27-29/06.1995

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STUDY OF CARBON NITRIDE FORMATION BY ION IMPLANTATION

Recent theoretical calculations of the properties of new materials indicate, that β - C_3N_4 carbon nitride could be a very interesting compound for numerous applications. The main interest in the synthesis of this material is its theoretical hardness which is expected to be higher than that of diamond.

The growing interest resulted in numerous attempts to synthesize carbon nitride. Physical vapor deposition (PVD) or chemical vapor deposition (CVD) methods were used mainly. It seems however, that the ion implantation may be an alternate and promising way to synthesize β - C_3N_4 since it provides high impurity concentration and high stress level during the process which should favourise the formation of covalent bonds. The aim of this paper is to present some preliminary results dealing with the implementation of ion implantation in the carbon nitride synthesis.

In the experiments the retention of nitrogen atoms implanted into carbon were determined by means of Rutherford Backscattering Spectrometry (RBS) and the formation of diamond-like carbon (DLC) layer with high nitrogen content, by decomposition of hydrocarbon vapour by nitrogen beam, was shown.

In the first experiment the samples from commercial graphite implanted with increasing doses of molecular nitrogen were used. The nitrogen implantations were performed at room temperature with 100 keV N_2^+ ions up to doses varying from 0.5×10^{17} at.N/cm² to 10×10^{17} at.N/cm². The DLC-like film was formed on the surface of silicium wafers and its composition was measured by means of RBS technique.

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OPTO-ELECTRONIC SCANNING DEVICE FOR THE OBSERVATION OF SCATTERING OF YAG LASER LIGHT IN SEMICONDUCTOR MATERIALS

Infrared laser light scattering is a powerful tool for investigation of inhomogeneities in the bulk semiconductor materials. A device for the observation of infrared scattering in semiconductor wafers is designed and manufactured. For sample illumination the diode pumped Nd:YAG laser emitting monomode 1,06 μm beam is used. The laser beam waist inside the semiconductor samples does not exceed 50 μm . The scattered centers inside the sample are observed perpendicularly to the direction of illuminating beam using microscope with infrared CCD camera. To obtain 2D image of scanned plane the sample is moved horizontally by the scanning stage driven by computer. Controlled changing of the scanning plane enables the investigation of the sample in the third direction. The scanning and scattered image processing are controlled by computer. The device is tested on GaAs wafers.

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INTERNATIONAL CONFERENCE ON ATOMIC COLLISIONS IN SOLIDS, Linz, Austria 17-21/07.1995

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ION CHANNELING IN SPINEL SINGLE CRYSTALS

Spinel is a material which crystallizes with a structure similar to that of the mineral having this name. The characteristic feature of the spinel family of materials is the enormous variation in chemical composition which is reflected in the broad range of their physical properties. Recently, a very low sensitivity of the MgAl_2O_4 spinel to the ionizing radiation led to its application as a window material in nuclear fusion devices and as an inert matrix for reactor irradiation of higher actinides. The aim of the reported project was to perform an investigation of the channeling properties of MgAl_2O_4 single crystals. The crystal structure of spinel, though cubic, is quite complex. There are 56 atoms in a unit cell: Mg atoms are occupying a diamond cubic structure, one half of the unit cell octants is occupied by Al atoms in a tetrahedral arrangement, whereas O atoms are located in all octants forming complementary oriented tetrahedra.

Spinel single crystals of $\langle 110 \rangle$ orientation were polished with diamond paste down to $0.25 \mu\text{m}$ and subsequently heated at 1400°C in Ar/H_2 to anneal the polishing damage. The channeling experiments were performed using 2 - 2.9 MeV ^4He beam. The shape of the channeling spectra for principal directions of a spinel single crystal reflects clearly the structure of atomic planes and rows. Taking as a reference the relative height of steps in the random spectrum, characteristic for each atomic

species, one can deduce which kind of atoms is mostly exposed to the channeled beam. The channeling spectrum for the (111) plane shows similar features as the random one, thus indicating that no atomic component is shielded. In contrast, the increase of scattering intensity for Mg and the reduction for O can be observed in the (100) planar direction. Some shielding of Mg atoms was noted for the (110) plane. The $\langle 110 \rangle$ axial spectrum does not show any peculiarities which is in accordance with the channel structure. It is worth mentioning, that a largely enhanced scattering yield for Al and O was observed in the $\langle 111 \rangle$ axial channeling spectrum. This is apparently due to the location of a mixed Al-O row in the middle of that channel.

The basic work on radiation damage behavior in compound materials demands a separate analysis of lattice defects in each sublattice. We have demonstrated that ion channeling is a suitable technique for this purpose. By the judicious choice of the channeling direction the sensitivity to the atomic displacements in the chosen sublattice can be significantly enhanced.

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ICCE - 2ND INTERNATIONAL CONFERENCE ON COMPOSITES ENGINEERING, New Orleans, USA 21-24/08.1995

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METAL MATRIX COMPOSITES - METHODS OF JOINING

Metal matrix composites (MMCs) reinforced with short ceramic fibres (e.g. carbon or Al_2O_3 fibres) or with other metals (such as e. g., W) show numerous advantages since their properties can be programmed by modifying appropriately their composition and technology. A point of considerable importance is the possibility of joining the composites with metals or their alloys. The major problem here is to choose an appropriate joining technique, such that ensures the formation of a high quality joint, resistant to the service conditions, avoids the degradation of the composite microstructure, in particular of the interface layer between the matrix and the reinforcement, and, still, is not expensive [1]. The paper presents the results of experiments on joining the following composites: 6061Al-based materials containing 15vol.% of δ -alumina fibres, CuCr1 - based materials containing 20 vol% of carbon

fibres (C_f), CuZr1-based materials containing 20 vol% of carbon fibres and Cu-based materials with 10 vol% of dispersed tungsten powder [2,3]. The CuCr1- C_f and CuZr1- C_f composites were joined with austenitic steel, the 6061Al- Al_2O_3 composite - with the 6061Al alloy and the CuW composite - with copper of 99.99% purity. The material pairs were chosen so as to take into account their possible applications. Several different joining techniques were examined. This paper discusses the results obtained when using diffusion bonding and vacuum brazing. The aim of the present study was to select the optimum technique of joining. The measure of the joint quality was taken to be the uniformity of the distribution of the reinforcing phase within the interface layer.

The joining processes were conducted under optimum conditions, i.e. such which ensured the best mechanical properties of the joints. The degree of degradation of the structure was taken to be the coefficient of the relative content of the reinforcing material RCRM - X/B, where X is the percent content of the reinforcing phase in the composite after the joining process, and B is the percent content of this phase in the starting material.

In the CuZr1- C_f composite diffusion-bonded with austenitic steel, the content of the fibrous phase appeared to be markedly reduced compared with its content in the starting material. In the CuW brazed with copper, the composite was depleted of tungsten in the vicinity of the joint. In the other composites, the averaged RCRM distributions were uniform. The non-uniform distribution of the reinforcing phase observed in the joints can be explained in terms of the diffusion processes that proceed during the joining. In conclusion we can state that both the proposed techniques can be used for joining the investigated composites. It should however be remembered that the optimum techniques are those which do not degrade the composite microstructure, and this depends on both the technique employed and the chemical composition of the composites to be joined.

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ECAART 4 - EUROPEAN CONFERENCE ON ACCELERATORS IN APPLIED RESEARCH AND TECHNOLOGY

Zurich, Switzerland 29/08-02/09.1995

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RADIATION DAMAGE IN SPINEL SINGLE CRYSTALS

The crystal structure of spinel, $MgAl_2O_4$, though cubic, is quite complex. There are 56 atoms in a unit cell. Basic work on radiation damage behaviour in compound materials demands a separate analysis of lattice defects in each sublattice. Spinel single crystals of $\langle 110 \rangle$ and $\langle 100 \rangle$ orientation were analyzed by means of the RBS/channeling technique using 2.0 - 2.9 MeV 4He beam prior and after ion implantation with Kr and Xe ions to the fluences ranging from 10^{14} to 10^{17} at/cm². The results of our study can be summarized as follows:

- spinel can be driven amorphous at room temperature by the implantation with approx. $2 \cdot 10^{16}$ Kr(Xe)/cm²,
- radiation damage behavior in aluminum and oxygen sublattices is closely similar; magnesium sublattice is much more stable with respect to ion bombardment, however, the precise analysis of Mg - sublattice is rather difficult because of the Al - Mg signal overlapping and the presence of 3 Mg isotopes.
- recovery stage of simple defects at a temperature close to 500°C was observed.

Materiał prezentowano na sesji posterów.

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**ECAART 4 - EUROPEAN CONFERENCE ON ACCELERATORS IN
APPLIED RESEARCH AND TECHNOLOGY
Zurich, Switzerland 29/08-02/09.1995**

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**THE EFFECT OF Cr IMPLANTATION ON THE MORPHOLOGY OF
Al:Si FILMS**

Annealing of Al-based metallization layers on SiO₂/Si substrates results in the formation of hillocks i.e. large outgrowths that may cause shortcuts in multilevel circuits. In this work we study the properties of Al: Si 1% and Al: Si 1%: Cu 4% films subjected to irradiation with Cr⁺ ions. The oxidized silicon wafers were coated with 1µm thick metallic layers by magnetron sputter deposition. Prior to sintering, typically at 450°C for 30 min, the films were implanted with chromium ions at an energy of 130 keV and to doses $4 \cdot 10^{15}$ - $9 \cdot 10^{15}$ cm⁻². Investigations of the processed surface using SEM, Alpha-step together with a procedure for digital evaluation of the surface morphology shows that chromium implantation leads to a significant reduction of the hillock population, in particular those of largest size. It should be stressed that the effect is observed for Al:Si and Al:Si:Cu alloys but not for pure Al. SIMS profiling gives evidence of measurable mobility of Cr atoms during the annealing. TEM investigations reveals in Cr⁺ implanted Al:Si layers an array of fine precipitates located within the grain volume. In addition to that, in Al:Si:Cu metallization there appear pellet-like crystallites in the bulk and regular crystallites 0.15µm in size, presumably CuAl₂, at the grain boundaries. Undoubtedly this latter phases are associated with copper however their precise identification by electron diffraction analysis is still in progress due to small dimensions of the objects. From the fact that the modification of grain-growth kinetics occurs only when Si is present in the film one may conclude that Cr atoms precipitate in a Si-rich phase (binary or ternary). Other effects of Cr implantation into Al-based films are reduction of lattice constant and increase in sheet resistance (by 20%). Formation of Cr containing precipitates seems beneficial in suppressing the overall diffusivity in the layer thus preventing the excessive vertical growth of the grains.

Materiał prezentowano na sesji posterów.

**V POLSKA KONFERENCJA CHEMII ANALITYCZNEJ:
ANALITYKA W SŁUŻBIE CZŁOWIEKA I ŚRODOWISKA
Gdańsk, Poland 3-9/09.1995**

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**SYSTEM JAKOŚCI W LABORATORIUM CHARAKTERYZACJI
MATERIAŁÓW WYSOKIEJ CZYSTOŚCI INSTYTUTU
TECHNOLOGII MATERIAŁÓW ELEKTRONICZNYCH**

Instytut Technologii Materiałów Elektronicznych specjalizuje się w technologii otrzymywania i w wykorzystaniu materiałów o strukturze monokrystalicznej. Wyroby opracowywane w ITME charakteryzują się wysoką czystością, doskonałą strukturą krystalograficzną i specyficznymi własnościami wymaganymi we współczesnej elektronice. Aby osiągnąć takie rezultaty problemy jakości w Instytucie są uwzględniane zarówno w pracach badawczych jak i wdrożeniowych.

Zgodnie z normą PN-EN 45001:1993 i przewodnikiem ISO/IEC Nr 25:1990 opracowano system zapewnienia jakości dla Laboratorium Charakteryzacji Materiałów Wysokiej Czystości zajmującego się pełną charakteryzacją materiałów, obejmującą zarówno analizę składu chemicznego jak i struktury. Zasady, jakimi kieruje się Laboratorium w swojej działalności zawarto w 22 rozdziałach Księgi Jakości. Sposób realizacji zasad polityki jakości opisano w 21 procedurach ogólnych.

Materiał prezentowano na sesji posterów.
Pełny tekst wydrukowany będzie w materiałach z konferencji.

**EPMS'95 - THE 5th INTERNATIONAL WORKSHOP ELECTRONIC
PROPERTIES OF METAL/NON-METAL MICROSYSTEMS
Polanica Zdrój, Poland 11-14/09.1995**

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DIGITAL ANALYSIS OF PHOTO-INDUCED CURRENT TRANSIENTS IN SEMI-INSULATING GaAs AND InP

Semi-Insulating (SI) GaAs and InP wafers are widely used as substrates for advanced high speed integrated circuits. They are also starting materials for manufacturing low dimensional electronic devices such as HEMTs and HBTs. So, the quality of these materials is of great importance in terms of device performance, yield and reproducibility.

The objective of this paper is to show how the deep-level defects, determining the electrical and optical properties of semi-insulating materials, can be characterised by measurements of photocurrent transients. The traps are filled with excess charge carriers generated by optical pulses and the measurements are performed while the light is off. A new digital method for recording and analysis of these transients is presented. The deep traps in high-resistivity bulk crystals of GaAs:Cr, undoped GaAs and InP:Fe were studied and the characteristics of thermal emission rate of charge carriers as a function of temperature were obtained. From these data the activation energy and apparent capture cross-sections for deep traps were calculated. The studies were completed by the measurements of ESR spectra for the same samples so the traps could be identified with the specific point defects such as EL2 in SI GaAs and Fe²⁺ in SI InP.

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EPMS'95 - THE 5th INTERNATIONAL WORKSHOP ELECTRONIC PROPERTIES OF METAL/NON-METAL MICROSYSTEMS Polanica Zdrój, Poland 11-14/09.1995

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ELECTRICALLY ACTIVE DEFECTS IN Ni-CONTAMINATED CZ-SI WITH OXYGEN PRECIPITATES

Silicon single crystals (Cz-Si), grown by the Czochralski method, contain a high concentration of dissolved oxygen atoms and much lower concentration of other non-purposely introduced contaminants, e.g. transition metals. During the IC fabrication the silicon wafer is oxidised and this system in the region adjacent to the oxide is affected by a high stress. Under these conditions the system is also exposed to a high temperature heat treatment resulting in a generation of various extended and point defects. So, the knowledge about the behaviour of these defects is of great importance for development of future advanced ULSI technologies.

The object of this work is to find how the defect structure of n-type Cz-Si containing oxygen precipitates (OP) changes due to the heat treatment carried out at high temperatures (HT) and high hydrostatic pressures (HP). The samples with the net donor concentration of $\sim 1 \times 10^{15} \text{ cm}^{-3}$ and the interstitial oxygen concentration $c_o \leq 9 \times 10^{17} \text{ cm}^{-3}$ were prepared in three ways : a) without preannealing (defect free), b) by preannealing at 720 -1000 K for 192 hours in order to create the nucleation centers for oxygen precipitation, c) by multiple-step process - the first annealing as in the case b) and the second at 1270 K for 20 hours. The high-pressure annealing was performed for a period up to 5 hours at temperatures up to 1550 K and hydrostatic pressures up to 1.35 GPa. Ni atoms were introduced by wrapping the samples into Ni foil during HP-HT treatment.

The changes in the sample defect structure were monitored by Deep Level Transient Spectroscopy (DLTS) as well as by chemical selective etching and X-ray methods. Four electron traps labelled as T1 ($E_a = 0.20 \text{ eV}$, $\sigma_a = 2 \times 10^{-14} \text{ cm}^2$), T2 ($E_a = 0.45 \text{ eV}$, $\sigma_a = 7 \times 10^{-14} \text{ cm}^2$), T3 ($E_a = 0.62 \text{ eV}$, $\sigma_a = 2 \times 10^{-12} \text{ cm}^2$) and T4 ($E_a = 0.48 \text{ eV}$, $\sigma_a = 7 \times 10^{-14} \text{ cm}^2$) were detected. It was found that the traps concentrations are strongly dependant on the preannealing temperature controlling the oxygen precipitation process and on the hydrostatic pressure applied to the samples, which determines the stress at the boundary between oxygen precipitate and the matrix of silicon atoms.

Materiał prezentowano na sesji posterów.
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**EPMS'95 - THE 5th INTERNATIONAL WORKSHOP ELECTRONIC
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Polanica Zdrój, Poland 11-14/09.1995**

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**GROWTH AND CHARACTERIZATION OF NEW DISORDERED
CRYSTALS FOR THE DESIGN OF ALL-SOLID -STATE LASERS**

Absorption lines of rare earth ions in crystals are usually much narrower than emission bands of semiconductor lasers. Consequently the absorption efficiency of all-solid -state lasers, based on rare earth doped crystals, is reduced and additionally a stringent control of laser diode temperature is needed. These shortcomings may be remedied by using the active media with structural disorder which tend to broaden the spectral lines of activating ions.

However, the disordered crystal must be chosen carefully since the strong inhomogeneous broadening may influence adversely the extraction efficiency. In this work we examine the crystals belonging to two large families of compounds of general chemical formula ABC_3O_7 and $ABCO_4$ ($A = Sr, Ba, Ca$; $B = Y, La - Gd$; $C = Ga, Al$) which appear to be promising matrices for rare earth and transition metal ions: Slope efficiency recorded for laser diode pumped $BaLaGa_3O_7$: Nd laser is comparable to that of YAG:Nd laser indicating that the extraction efficiency is not reduced in spite of inhomogeneous broadening of emission line.

The activator - activator and activator - host interactions have been studied using the crystal samples containing Eu^{3+} , Tm^{3+} , Pr^{3+} and Cr^{3+} in order to assess their laser performance. Based on results obtained the suitability of chromium doped $SrLaAlO_4$ and $SrLaGaO_4$ crystals for the design of tunable lasers is predicted.

Material prezentowano na sesji posterów.
Tekst został wydrukowany w materiałach z konferencji.

**EPMS'95 - THE 5th INTERNATIONAL WORKSHOP ELECTRONIC
PROPERTIES OF METAL/NON-METAL MICROSYSTEMS
Polanica Zdrój, Poland 11-14/09.1995**

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**FTIR STUDY OF OXYGEN PRECIPITATION IN HIGH PRESSURE
TREATED Cz-Si CONTAMINATED BY TRANSITION METALS**

Czochralski grown silicon single crystals (Cz-Si) contain dissolved oxygen atoms and nonpurposely introduced transition metal contaminants. Under high temperature and stress (e.g. at the surface film/ Si bulk edge during IC processing) the structural form of mentioned species can change considerably.

Present work deals with investigation of oxygen precipitation phenomena by Fourier Transform Infrared Spectroscopy (FTIR) in Cz-Si samples with the oxygen atom concentration in the interstitial positions, $c_o \leq 9 \times 10^{17} \text{ cm}^{-3}$ subjected to annealing at up to 1400K under uniform stress (hydrostatic pressure) up to 1.0 Gpa (HP - HT treatment) [1]. Deconvolution of the IR absorption curves into Gaussian profiles was performed to separate absorption originating from different oxygen containing components.

The sample defect structure before and after the treatment was checked by selective etching, X-ray methods and TEM.

The samples contaminated by Ni or Mo during HP-HT treatment with nucleation centers for oxygen precipitation created by preannealing at = 1000K were chosen.

The effects of HP-HT treatment on c_o are mostly dependent on the initial defect structure of the samples (preannealing conditions) and on particulars of the treatment. Contamination by transition metals has only changes minor effects (typically $\pm 10\%$ as compared to non-contaminated samples).

Observed effects are compared with reported for contaminated Cz-Si annealed

under atmospheric pressure [2].

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Materiał prezentowano na sesji posterów.
Tekst został wydrukowany w materiałach z konferencji.

HTSC - THE FIRST POLISH-US CONFERENCE ON HIGH TEMPERATURE SUPERCONDUCTIVITY Duszniki Zdrój, Poland 11-15/09.1995

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ELECTRON SPIN RESONANCE STUDY OF Fe³⁺ IONS IN SrLaAlO₄ AND SrLaGaO₄ HIGH-T_c SUBSTRATES

Single crystals of SrLaAlO₄ (SLA) and SrLaGaO₄ (SLG) are considered as promising substrates for the HTSC films. Crystals are grown by the Czochralski method and it was found that all of them contain iron impurity lower than 10⁻³ at.%. The sensitivity of the ESR method was enough to investigate iron ion in the undoped (as-grown) and iron doped crystals.

In these crystals the angular dependence of three lines was distinguished at X-band frequency in the temperature range from 4 K to 300 K. These lines are attributed to Fe³⁺ (3d⁵, S= 5/2). The transitions insides for allowed doublet ± 1/2, and "forbidden" ± 3/2, ± 5/2 were observed. The Fe³⁺ ions are surrounded by a distorted octahedral distribution O²⁻ ions. This octahedral lacks inversion symmetry and there are possible transitions with ΔM > 1 presumably as electrically induced. These transitions are described by axis spin-Hamiltonian with the parameters D > 1.2 cm⁻¹ and g_{||} = g_⊥ = 1.98.

Materiał prezentowano na sesji posterów.
Tekst został zamieszczony w materiałach z konferencji.

**HTSC - THE FIRST POLISH-US CONFERENCE ON HIGH
TEMPERATURE SUPERCONDUCTIVITY
Duszniki Zdrój, Poland 11-15/09.1995**

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**LOW FIELD MICROWAVE ABSORPTION IN A YBCO THIN FILMS
ON DIFFERENT SUBSTRATES**

Non-resonant microwave absorption as a function of temperature and magnetic field has been studied in epitaxially grown thin films of YBCO on SrTiO₃, SrLaAlO₄ (SLA) and SrLaGaO₄ (SLG) substrates. Results obtained showed that both single crystals of SLA and SLG look promising as the substrates for the high temperature superconducting films of YBCO.

Materiał prezentowano na sesji posterów.
Tekst został wydrukowany w materiałach z konferencji.

**HTSC - THE FIRST POLISH-US CONFERENCE ON HITH
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RAMAN SPECTROSCOPY IN SrLaGaO₄ SUBSTRATE CRYSTAL

There has been a considerable interest in the physical properties of strontium lanthanum gallate, SrLaGaO₄ (SLG) belonging to the family of compounds with the general formula ABCO₄, where A = Ca or Sr, B = rare earth element and C = Al, Ga or some transition element. SLG single crystals are interesting as substrates for high temperature superconducting thin films from the standpoint of lattice matching and their dielectric properties. Good quality of epitaxial layers requires both crystallographic perfection and appropriate physical properties of substrate material.

SLG single crystals used in our experiment were grown from nonstoichiometric melt by Czochralski method. Samples of sizes about 3x5x6 mm³ were cut from as-grown yellow colored SLG single crystals. The samples used in Raman scattering experiment were ground and polished to optical quality.

In this paper we report the Raman scattering study of SLG single crystals. Through polarization studies of oriented crystals the Raman-active vibrational modes were identified. Moreover, the study of the polarized and temperature dependencies of some of the Raman modes have been performed. The results obtained are discussed in terms of the nature of defects which might arise in the ABCO₄ lattice during crystal growth process.

Materiał prezentowano na sesji posterów.

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HTSC - THE FIRST POLISH-US CONFERENCE ON HIGH TEMPERATURE SUPERCONDUCTIVITY Duszniki Zdrój, Poland 11-15/09.1995

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OPTICAL METHODS IN ASSESSMENT OF STRUCTURAL STABILITY OF HTS THIN FILM SUBSTRATES. APPLICATION TO SrLaAlO₄ AND SrLaGaO₄

Performance of high temperature superconducting thin films depends critically on the physical properties of the material used as a substrate. In particular, any potential substrate material should combine an acceptably low dielectric constant and low microwave noise levels with the stable crystalline structure giving minimal mismatch with the superconductor used. In addition to these features which are inherent to material considered a high structural perfection is desired which usually is the most difficult to achieve. Methods of optical spectroscopy provide the information on both the structural stability and the density of lattice defects which is relevant to the development of high quality HTS substrates. Two different approaches have been used in this study in order to get a closer insight into the structure of SrLaAlO₄ and SrLaGaO₄. In the first one the behavior of structural defects have been investigated in the 5 K - 600 K region. In the second one the Cr³⁺ and Eu³⁺ ions have been used as probes of local crystal field strength and symmetry and their optical spectra have been analyzed. The results reveal that the inherent structural disorder associated with random distribution of Sr²⁺ and La³⁺ ions over the lattice sites favours the formation of point defects which are abundant in both crystals.

Materiał prezentowano na sesji posterów.
Tekst został wydrukowany w materiałach z konferencji.

SMM'12 - SOFT MAGNETIC MATERIALS CONFERENCE Kraków, Poland 12-14/09.1995

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FMR STUDY OF Co SUBSTITUTED YTTRIUM IRON GARNET FILMS

$Y_{3-z}Ca_zFe_{5-x-y}Co_xGe_yO_{12}$ films ($7 + 10\mu m$) grown on (001) plane GGG substrate were investigated vs. temperature using FMR technique. The measurements were carried out on X-band spectrometer equipped with the cryostat, enabling investigations at 4-300K temperature range. The resonance field H_r amplitudes were measured for external magnetic field applied in two characteristic planes: (001) and (110). Essential influence of the film speed of growth on magnetic anisotropy constants was found which is discussed taking into consideration Pb ions contribution. Angular dependencies of the H_r field became more complicated with lowering of temperature. For the explanation of obtained results it was necessary to consider magnetic anisotropy description assuming three cubic anisotropy constants and four uniaxial anisotropy constants. The FMR signal vanished at temperatures below about 100 K. This temperature depended on the applied field orientation. This effect is explained by consideration of magnetic anisotropy determined from torque anisometry analyses. The temperature peculiarities are discussed taking into account contributions of different magnetic ions (with different valence and crystallographic position) to magnetic anisotropy.

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GRIN'95 INTERNATIONAL CONFERENCE GRADIENT - INDEX OPTICS IN SCIENCE AND ENGINEERING Kazimierz Dolny, Poland 13-15/09.1995

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PROBLEMY TECHNOLOGICZNE SZKIEŁ NA SOCZEWKI TYPU GRIN

Przedstawiono problemy związane z wytwarzaniem szkieł przeznaczonych do soczewek typu GRIN. Jednym z najważniejszych wymagań stawianych szkłem jest jego wysoka jednorodność chemiczna i fizyczna. Przedstawiono wyniki uzyskiwania struktur GRIN przy zastosowaniu szkieł wytapianych w warunkach laboratoryjnych w elektrycznym piecu oporowym oraz w piecu indukcyjnym. Jakość uzyskiwanych szkieł porównano ze szkłem wytapianym w Jeleniogórskich Zakładach Optycznych. Wytyczono kierunek optymalizacji jednorodności szkła przez zastosowanie jednostopniowe-

go wytwarzania prętów szklanych metodą wyciągnięcia z tygla ogrzanego indukcyjnie.

Tekst zaprezentowano na sesji posterów.

Tekst wystąpienia wydrukowany będzie w: "Proceedings of SPIE" 1995 r.

SYMPOZJUM: ZASTOSOWANIE CZUJNIKÓW ŚWIATŁOWODOWYCH W MECHANICE, Warszawa, Poland 16/09.1995

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OPTYCZNE WŁÓKNA SZKLANE DO CZUJNIKÓW ŚWIATŁOWODOWYCH

Szkła wieloskładnikowe w odróżnieniu od powszechnie stosowanych szkieł krzemionkowych dają dużo większe możliwości modyfikacji parametrów (np. współczynnik załamania światła, współczynnik rozszerzalności termicznej). Rozszerza to możliwość tworzenia struktur o skomplikowanym rozkładzie naprężeń wewnętrznych, profilu współczynnika załamania, apertury numerycznej.

Metoda mozaikowa, możliwa do zastosowania przy wytwarzaniu światłowodów ze szkieł wieloskładnikowych, pozwala na wytwarzanie światłowodów wielordzeniowych, posiadających puste lub wypełnione obszary nie przewodzące światła (wprowadzające naprężenia), wielopłaszczyznowych oraz struktur włóknistych. Zarówno rdzenie jak i pokrycia mogą mieć różne własności optyczne, mechaniczne i termiczne.

Możliwe jest wytwarzanie struktur posiadających kierunkowo zmieniającą się aperturę numeryczną na skutek umieszczenia w warstwie kory światłowodu obszarów o skokowo zmienionym współczynniku załamania. Stosowana w przypadku szkieł wieloskładnikowych technologia jest prostsza i zapewnia niższe koszty wytwarzania. Wymagania co do własności stosowanych szkieł są bardzo wysokie. Dotyczą takich parametrów jak transmisja spektralna, jednorodność szkła, naprężenia wewnętrzne, współczynnik załamania, współczynnik rozszerzalności termicznej, przebieg krzywej lepkości. Stwarza to szereg problemów technologicznych przy wytwarzaniu szkieł na korę i rdzeń światłowodu.

Optymalne warunki daje metoda indukcyjnego wytapiania szkieł, która została opracowana i zastosowana w ITME do prac w dziedzinie szkieł i światłowodów.

Materiał prezentowano na sesji posterów.

Tekst został wydrukowany w materiałach z konferencji.