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NEW APPROACH TO THE DESIGN OF SCHOTTKY BARRIER DIODES FOR THz MIXERS

Subfemtofarad GaAs Schottky barrier diodes especially designed for mixing applications in the THz frequency range are presented.

A diode fabrication process of $0.6\mu\text{m}$ diodes with near-ideal electrical characteristics will be described. This process is based on the electrolytic pulse etching of GaAs in combination with an in-situ platinum plating for the formation of the Schottky contacts. Schottky barrier diodes with a diameter of $1\mu\text{m}$ fabricated by the process have already shown excellent results in a 650 GHz waveguide mixer at room temperature. A conversion loss of 7.5 dB and a mixer noise temperature of less than 2000 K have been obtained at an intermediate frequency of 4 GHz.

The optimization of the diode structure and the technology was possible due to the development of a generalized Schottky barrier diode model which is valid also at high current densities. The common diode design and optimization are discussed on the basis of the classical theory. However, the conventional formulas are valid only in a limited forward bias range corresponding to currents much smaller than the operating currents under submillimeter mixing conditions. The generalized new model takes into account not only the phenomena occurring at the junction, such as current dependent recombination and drift-diffusion velocities, but also mobility and electron temperature variations in the undepleted epilayer.

Calculated diode I/V and noise characteristics are in excellent agreement with the measured values. Thus, the model offers the possibility of optimizing the diode structure and predicting the diode performance under mixing conditions at THz frequencies.

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ESR AND MAGNETIC STUDIES OF Rb AND K DOPED C_{60}

C_{60} powder prepared in various conditions were doped with potassium and rubidium in sealed ampoules filled with helium. After annealing in various conditions of heat treatment the samples were measured by means of ESR spectrometer and SQUID magnetometer. Superconducting properties were detected observing microwave absorption and measuring low magnetic field susceptibility. The ESR measurements made at 4.2 K and up to room temperature show, that in samples exhibiting superconducting properties, two various electron states with different g-factor and different line width were created by alkali metals doping. It was observed strong dependence of these parameters on annealing procedure and dopants concentration. From low field susceptibility measurements critical temperatures and first critical fields were determined for various dopants concentration and annealing procedures and some attempts were made to estimate the critical current density.

Tekst wystąpienia opublikowany będzie w materiałach z konferencji

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OZNACZANIE ŚLADOWYCH ILOŚCI ZANIECZYSZCZEŃ W MATERIAŁACH ELEKTRONICZNYCH METODĄ EMISYJNEJ SPEKTROMETRII ATOMOWEJ

W Instytucie Technologii Materiałów Elektronicznych opracowywane są metody otrzymywania metali czystych i materiałów półprzewodnikowych. Podstawowym elementem kontrolnym stosowanych technologii jest analiza chemiczna. Służy ona nie tylko do oceny jakości produkowanych materiałów ale umożliwia również uzyskanie informacji na poszczególnych etapach procesu technologicznego.

W ramach prac badawczych opracowano w ITME metody analizy wielu materiałów i metali wysokiej czystości stosowanych w przemyśle półprzewodnikowym między innymi gal, ind, fosfor, arsen, chlorek arsenu, srebro, tellur, bizmut, trójtlenek boru, kwas borowy, kwas szczawiowy, ruteniany ołowiu i bizmutu itp.

Opracowane metody umożliwiają oznaczanie zanieczyszczeń metalicznych na poziomie 10^{-4} - $10^{-8}\%$. Optymalne warunki prowadzenia analizy osiągnięto dzięki wykorzystaniu matematycznych metod planowania doświadczeń. Zasadę kolejnych zmian poszczególnych czynników zastąpiono zasadą jednoczesnej zmiany wszystkich czynników według określonego algorytmu - macierzy planowania. Ten sposób postępowania umożliwił otrzymanie opisu matematycznego badanych zależności. Znajomość modelu matematycznego i statystyczne opracowanie wyników doświadczeń ułatwiają wyciąganie wniosków z badanych wielkości fizykochemicznych i umożliwiają optymalne kierowanie doświadczeniem przy niepełnej znajomości badanego procesu.

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THE IMAGES OF DISLOCATIONS IN SYNCHROTRON BRAGG-CASE SECTION TOPOGRAPHY OF DIAMOND

The Bragg-case section topographic images were obtained in a 0.7 mm thick diamond plate cut and polished from a large synthetic diamond. The experiments were performed at the station 7.6 of SRS radiation source in Daresbury Laboratory. The topographs were exposed in 400 - 511 and 311 - reflections with the Bragg angle of 45° , and the main component of radiation respectively 1.26 \AA , 0.97 \AA , and 1.54 \AA . The sample was previously examined with number of methods and most of the existing defects were preliminary identified. The series of topographs were exposed with the position of the sample slightly shifted and the beam intersecting different regions of particular defect.

The investigated sample contained large regions with low defect density, and the topographs obtained in these regions exhibited numerous interference fringes. These included the Pendellösung fringes connected both with the incident beam and the beam reflected from the rear surface and the fringe systems connected with different defects.

The experimental images were compared with the theoretical ones obtained by the numerical integration of Takagi equations. The original numerical program adopting Epelboin's idea of variable step was developed. The deformation field of dislocation included the stress relaxation on the free surface.

Two versions of including the finite width of a slit were examined: one by adding several sections with single point of incidence and other by adding simulation with excited number of points and different angle of incidence. The later method was more adequate to the present situation where the angular divergence of the beam was limited to less than 0.5 due to the 80 m distance from the tangent point of the storage ring. Both algorithms required single calculation of deformation field.

The correspondence of experimental and simulated images was satisfactory and many of the fringe systems present in the simulated images were visible on the experimental fringes. It was also found both from experiments and simulations that limitation of the beam divergence preserves more fringe systems with increasing slit width.

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OBSERVATION OF INTERFERENCE FRINGES IN BRAGG-CASE SYNCHROTRON DOUBLE-CRYSTAL IMAGES OF STACKING FAULTS IN DIAMOND

Bragg-case double-crystal images of stacking faults obtained using double-crystal arrangements with a synchrotron source of X-rays were studied in a slab cut from a synthetic diamond. The experiment were performed at the station 7.C of the SRS radiation source in Daresbury Laboratory. The best results were obtained using the 511_{C} - 311_{D} arrangement selecting radiations of 1.54 \AA . The 80 m distance of the experimental arrangement from the tangent point of the storage ring enabled obtaining spatial resolution better than $1 \mu\text{m}$ and the probe beam divergence less than 0.3° . The images of stacking faults in topographs taken on the tails of the rocking curve exhibited well pronounced interference fringes. The fringes were strongly dependent on the angular setting; they were less spaced further from the maximum and invisible in the uppermost regions of the peak. The fringes corresponding both to Bragg-Laue and Bragg-Bragg cases of diffraction on a stacking fault were observed.

The experimental images were compared with those theoretically predicted from an application of plane-wave dynamical theory. A reasonably good correspondence between theoretical and experimental images was obtained taking into account the finite divergence of the probe beam.

The theoretical images of stacking fault were dependent on the type of stacking fault, producing some difference in the first fringe, close to the outcrop of the stacking fault on the surface. This first fringe also varied on the opposite sides of the rocking curve, providing certain possibility of identification of the stacking fault type.

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DEFECTS STUDIES IN AS-GROWN AND N-IRRADIATED PHOSPHORUS RICH GaP

Phosphorus rich liquid encapsulated Czochralski GaP crystals were studied by EPR, absorption and transport techniques. Semi-insulating, p- and n- type materials were investigated. The same measurements were carried out these materials after irradiation by fast neutrons with doses of $3.8 \cdot 10^{15}$, $4.1 \cdot 10^{16}$, $3.7 \cdot 10^{17}$, $3.0 \cdot 10^{18}$, $\approx 8 \cdot 10^{18} \text{ ncm}^{-2}$. The EPR studies of as-grown crystals showed the presence of phosphorus antisite defect P_{Ga} . The same defect was found to be introduced by neutron irradiation. The analysis of hyperfine structure of EPR P_{Ga}^{4+} signal showed that the construction of phosphorus antisite defect created by neutron irradiation in GaP crystals is identical as in as-grown crystals. The defect was determined to be an isolated phosphorus antisite surrounded by four substitutional phosphorus atoms. Besides, the EPR measurement showed the existence of another defect in n-irradiated materials. The strong, broad singlet with well resolved hyperfine structure ascribed to this defect is under studies now.

The absorption spectra of n-irradiated materials contained two bands at 0.80 eV and 1.15 eV. The correlation between them seemed to exist. Thus one can assume that these bands are connected with one particular defect, most probably one of the two observed by EPR.

The measurements of conduction showed that at lower dose the resistivity has high value of about 10^{12} cm at 300 K. At higher doses the resistivity decreased with dose and at $\approx 8 \cdot 10^{18} \text{ ncm}^{-2}$ it was equal to about 10^5 cm . The temperature dependance of resistivity indicated that the mechanism of conduction at higher doses was tunnel assisted hopping. The analysis of the parameters of that hopping conduction is under progress.

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OPTICAL AND ELECTRICAL STUDIES OF FR1 AND FR2 DEFECTS IN GaAs

On the basis of the existing experimental data it can be safely concluded that the compensation mechanism in commercially grown GaAs is not a simple balance between shallow donors, carbon acceptor and EL2. The concentration of carbon acceptor is not sufficient to explain the experimentally found concentration of ionized EL2. Therefore, quite an interest has been concentrated on the origin of possible acceptor defects in GaAs for the last few years. Using EPR technique so called FR1 and FR2 defect acceptors has been found in GaAs crystals. However, their EPR spectra have been not resolved enough to determine the origin of these defects.

In this paper the systematic EPR, optical absorption (in the energy range of 0.025 to 1.55 eV), photoluminescence (PL) and thermally stimulated current (TSC) studies were performed on bulk GaAs with the aim of better understanding of acceptor defects in GaAs. EPR measurements showed the presence of FR1 and FR2 defects in most commercially grown GaAs crystals with different relative concentration.

Parallel EPR and optical absorption experiments allowed for the first time to find the absorption spectrum due to the photoionization of FR1 defect with the threshold at 0.19 eV. The identification of this spectrum was further confirmed in Cr-doped GaAs, where FR1 behaviour under illumination is different than in undoped GaAs. PL studies showed two families of bands in the energy range of about 1.25 to 1.35 eV. We tentatively ascribed them to FR1 and FR2 complexes with shallow donors.

TSC measurements showed different peaks depending on illumination times and wavelength of excitation light. Based on this dependence two peaks at 90 K and 110 K were assigned to FR1 and FR2 respectively.

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SUPERCONDUCTIVITY IN INDIUM DIFFUSED GaAs

Superconductivity of indium diffused GaAs has been investigated. The superconductivity in these samples has been identified by the Meissner effect and the characteristic field modulated microwave absorption (FMMA). The static magnetic susceptibility has been measured from 40K down to 2.5K. The results shows three distinctive diamagnetic contributions within 10K - 2.5K range. The first pronounced contribution has been found for temperatures lower than 3.5K. This temperature corresponds well to the critical temperature of indium metal and suggest that this signal is related to superconductivity of indium inclusions present in the material. In addition to this signal, another strong diamagnetic contribution has been found for temperatures lower than 5.5K. A characteristic FMMA signal near zero magnetic field found in all indium diffused samples seems to be connected with this superconductive phase. The third and relatively weakest diamagnetic contribution has been found for temperatures lower than 10 - 7K. The FMMA signal connected with this phase give rise to a local maximum observed near 260G at the lowest temperatures. In contrast to the characteristic absorption at zero field found in all measured samples, the 260G maximum was present only in some of them. It is shown that the excess of gallium leads to generation of the 260 G signal. The observed transition temperature suggest that it could be due to decoration of extended defects by stable amorphous phase of gallium.

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APPLICATION OF DEEP LEVEL TRANSIENT SPECTROSCOPY FOR MONITORING
POINT DEFECTS IN III-V SEMICONDUCTORS

The main electrical and optical properties of III-V semiconductors strongly depend on their defect structure which is formed by various point defects generated during material growth and device manufacturing. In the vapour phase epitaxial layers of GaAs_{1-x}P_x and GaP with low resistivity, which are used as starting materials for electroluminescent devices, deep defect centres are studied by transient capacitance spectroscopy. A number of donor-related deep-level defects have been found. Apart from doping level, the defect structure of these materials is affected by the III-V ratio, dislocation density and growth temperature. The reactions leading to formation of the grown-in deep-level defects are discussed. The effect of these defects on the radiative recombination efficiency is presented. In the high-resistivity bulk crystals of GaAs:Cr, undoped GaAs and InP:Fe, which are starting materials for monolithic microwave integrated circuits and high-speed digital circuits, deep defect centres are studied by photoinduced transient spectroscopy (PITS). The characteristics of thermal emission rate of charge carriers as a function of temperature and activation energies of deep centres have been determined. The effect of the Fermi level position on the PITS signal has been established and a tentative identification of the deep-level centres has been performed. The defect structure of semi-insulating GaAs crystals with various carrier mobility is compared.

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NUMERICAL CALCULATIONS OF TEMPERATURE FIELD FOR THE CASE OF SIMPLE CONVECTION MODEL IN AUTOCLAVE

A numerical study of temperature field for the case of 8.5 litre autoclave, filled with water, was performed using an iteration method. The natural convection in liquid was assumed to be steady, axisymmetric and laminar. A simple staircase function was taken to describe the radial profiles of the flow velocity in both chambers of the vessel. Moreover a linear functional relationship between the radial temperature gradient and the flow velocity was included in the model.

Two thermal steady states of the vessel were considered, with weak cooling and with intense heat exchange between the surroundings and the interzone (baffle) region. For both states almost identical water pressure (1400 bar) and identical local temperatures, under the plunger (350°C) and under the bottom of the vessel (390°C) were achieved experimentally. The distributions of the temperature on the outer steel wall and along the centerline of the reactor were measured by means of thermocouple set. The boundary conditions were defined by the above distributions in numerical computations.

The temperature fields were determined for three flow velocities 2m/h, 1m/h and .5m/h (upper zone). All results are reported in the form of isothermal contours. They show several common features for both thermal states. Firstly, the velocity of water flow is few times higher in the upper than in the lower zone. Secondly, the strong concentration of isotherms exists in the interzone region. Thirdly, the radial temperature gradients become greater when additional cooling is applied to the baffle region.

Tekst napisany dla "Materiałów Elektronicznych".

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THE BRAGG-CASE IMAGES OF DISLOCATIONS AT DIFFERENT ABSORPTION

The images of dislocations for X-ray multocrystal topographic arrangements were simulated and compared with experimental ones in some situations differing mainly in absorption of radiation. These situations corresponded to experiments in high quality synthetic diamond, silicon and gallium arsenide realized both with conventional and synchrotron X-ray sources.

The theoretical images were obtained by numerical integration of Takagi equations. Some different numerical algorithms assuming both finite and infinite crystal thickness were used. Simulations taking into account the finite divergence of the probe beam were also performed by averaging of up to 100 plane-wave simulations differing with a small change of the angle of incidence and weighted with a rocking curve profile. The formation of different elements of the simulated images was examined by visualisation of the intensity distributions in the plane of incidence.

It was found that at low absorption the simulated theoretical plane-wave images are dominated by numerous interference fringes. Obtaining a satisfactory correspondence with the experimental topographs in diamonds was possible taking into account the divergence of the probe beam. In this case most of the fringes were averaged, but at good collimation of the beam some fringe systems were visible both in theoretical and in experimental images of dislocations. In many cases the averaged fringe systems produced dark extinction contrast, also visible in experimental topographs.

The growing absorption resulted in the decreasing amount of interference fringes and in the occurrence of the dilatation-orientation contrast. The including of the probe beam divergence produced smaller effect and was not so important in obtaining of the correspondence to the experimental images. At very low absorption the assumption of the finite thickness of the crystal provided additional sets of fringes. The formation of these fringes was found as the result of bending of the fringes forming in the perfect crystal slab. At higher absorption the images of dislocations soon become not depending on the increasing thickness and the finite thickness algorithms produce the same results as that assuming the infinite thickness. The finite thickness algorithms provide advantages in placing of the dislocation line far from the start line and in smaller increase of computing time with increasing area of simulation.

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**TETRAGONAL-CUBIC INVERSION IN UNSTABILIZED ZIRCONIA DISPERSED
IN ALUMINA MATRIX**

The effect of sintering atmosphere on content of zirconia phases present in $\text{Al}_2\text{O}_3\text{-ZrO}_2$ ceramics was studied. As it was demonstrated, in case of high vacuum-sintered ceramics 100% of zirconia is cubic form, in contrast with 83.5% of tetragonal form in air-sintered ceramics. Appearance of transformable cubic zirconia is a result of sintering in high vacuum and accompanied oxygen nonstoichiometry. By changing of vacuum level and oxygen content in a sintering atmosphere, a critical value of oxygen nonstoichiometry was estimated, where "low temperature" tetragonal-cubic inversion of unstabilized zirconia inclusions occurs.

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BADANIA ANIZOTROPII PRZEWODNICTWA CIEPLNEGO KOMPOZYTÓW WŁÓKNISTYCH

Metodologia wyznaczania współczynnika dyfuzyjności i przewodnictwa cieplnego różniąc się od często stosowanych, opartych o koncepcję przedstawioną w pracy [1].

W zaproponowanej metodzie zastosowano modele matematyczne źródła zmiennego i stałego oraz system trójpunktowego pomiaru zmian temperatury.

Źródła ciepła tworzy się stosując odpowiedni czas nagrzewania laserem lub promiennikiem.

Pomiar zmian temperatury wywołany przejściem frontu cieplnego przez próbkę dokonuje się jednocześnie: 1) w sąsiedztwie czoła próbki (x_1), 2) w głębi próbki (x_2) oraz w jej części końcowej (x_3). Wykres zmian temperatury w odległości x , od źródła ciepła informuje o czasie zbliżania się frontu cieplnego do końca próbki. Pozwala to na dobranie warunków eksperymentu odpowiednich dla przyjętych modeli matematycznych w układzie półnieskończonym. Ten system pomiaru [2] pozwala na stosowanie obok źródła zmiennego również pomiarów i obliczeń w układzie źródła stałego.

Podane metody pomiaru i wyznaczania parametrów cieplnych zawierają kilka wariantów obliczeń. Daje to możliwość uściślenia wyników i ocenę modeli matematycznych.

Przewidziano badania anizotropii przewodnictwa cieplnego i wpływu technologii wytwarzania kompozytów na wielkości współczynnika dyfuzyjności i przewodnictwa cieplnego.

Bibliografia

1. W. J. Parker i in. Journ. of Appl. Physics 32, 1961, No.9.
2. M. J. Buda, Materiały - Conference on Surface Physics, Łódź, 1991.

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WZROST ODPORNOŚCI NA PĘKANIE NIEKTÓRYCH TWORZYW CERAMICZNYCH TOWARZYSZĄCY PROPAGACJI PĘKNIĘĆ, A MODELE ODPORNOŚCI NA PĘKANIE.

Zjawisko wzrostu odporności na pękanie ze wzrostem długości pęknięcia zostało odkryte kilkanaście lat temu. Tworzywa ceramiczne, w których to zjawisko występuje to: jednofazowa ceramika korundowa [1] ceramika korundowa z whiskerami z SiC [2], ceramika korundowa z dodatkiem ZrO_2 [3], stabilizowana ceramika cyrkonowa [4], lub ceramika z azotku krzemu [2]. Wspomniane powyżej zjawisko może w istotny sposób zmniejszać rozwój pęknięć podkrytycznych w materiałach ceramicznych [5]. Autorzy niniejszej pracy przedstawiają wyniki badań prowadzonych na próbkach z ceramiki korundowej o zawartości 99.5% Al_2O_3 i z ceramiki korundowo-cyrkonowej o zawartości 84.5% Al_2O_3 i 15% ZrO_2 (w fazie jednoskośnej). Próbkę z ceramiki korundowej w kształcie krążków nagriatano piramidką Vickers'a wprowadzając na ich powierzchniach wady o różnych wielkościach w zależności zastosowanej siły nacisku, a następnie badano ich wytrzymałość na dwuosiowe zginanie [6]. Na podstawie uzyskanej w ten sposób zależności wytrzymałości od siły nacisku piramidki Vickers'a oraz w oparciu o model odporności na pękanie [1], wykreślono zależność odporności na pękanie od długości pęknięcia. Z ceramiki korundowo-cyrkonowej wykonano dwie partie próbek w postaci belek do zginania trójpunktowego. Na belkach z karbem mierzono odporność na pękanie, a na belkach bez karbu wytrzymałość [3]. Na podstawie wyników tych pomiarów określono średnią długość "naturalnej" wady w obu partiach materiału. Następnie wyznaczono zależność odporności na pękanie w funkcji długości pęknięcia korzystając z modelu [1]. Próbkę z jednej partii materiału wygrzewano wielokrotnie uzyskując próbki o większych ziarnach oraz większej gęstości i ilości mikropęknięć. Dla każdej z tych partii wyznaczono krzywe odporności na pękanie. Przeprowadzone badania wykazały, że omawiane materiały wykazują wzrost odporności na pękanie w funkcji długości pęknięcia. Wydaje się też, że główną przyczyną tego zjawiska jest mechanizm mostkowy wzmocnienia odporności na pękanie, choć nie można do końca wykluczyć mechanizmu związanego z powstawaniem stref mikropęknięć [3].

Bibliografia

1. P.Chantikul S.J.Bennison B.R.Lawn J.Am.Ceram.Soc. 73, 1990 [8] 2419.
2. N.Ramachandran D.K.Shetty ibid.74, 1991 [10], 2634

3. M. Boniecki, Prace ITME z.35 1991
4. D.B.Marshall M.V.Swain J.Am.Ceram.Soc.71 1988 [6] 399
5. T.Fett D.Munz J.Mat.Sci.Lett. 10 1991r. 1103 (1991).
6. D.B. Marshall Am.Ceram.Soc.Bull.59, 1980 5, 551.

Pełny tekst wystąpienia będzie opublikowany w materiałach z konferencji.

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X-RAY TOPOGRAPHY AND DIFFRACTOMETRY OF SYNTHETIC DIAMONDS

X-ray topography reveals internal crystal growth features such as dislocations, stacking faults and growth sector boundaries in synthetic diamonds; as well as the strain fields associated with metal inclusions [1]. By employing several different X-ray reflections (of diffraction vector \mathbf{g}) the Burgers vectors (\mathbf{b}) of dislocations and the fault vectors of stacking faults can be determined from invisibility criteria (such as $\mathbf{g} \cdot \mathbf{b} = 0$). In diamond \mathbf{b} is usually parallel to one of the six $\langle 110 \rangle$ orientations. Dislocation line directions have been found parallel to another $\langle 110 \rangle$ or to a $\langle 211 \rangle$ direction; so that the dislocations are pure edge, pure screw, or of mixed 30° , 60° or 73.2° type. (Occasionally 45° and 54.7° types have also been found).

Double-crystal techniques enhance the sensitivity to lattice strains and enable relative lattice parameters to be measured with an accuracy of one part per million [2,3,4] when using well collimated synchrotron X-rays and large Bragg angles. The selective incorporation of nitrogen (100 ppm) into $\{111\}$ growth sectors increases the lattice parameter by about 10 ppm compared with the relatively pure $\{110\}$ growth sectors. For the selection of diamonds for use as monochromators of powerful synchrotron radiation, rocking curve widths less than ten seconds of arc are required. Various types of X-ray diffraction interference fringes have also been observed from crystal defects in otherwise nearly perfect synthetic diamond.

[1] A.R.Lang and M.Moore, *Proc 2nd Int.Conf.New Diamond Science & Tech.* (Ed.R.Messier, J.T.Glass, J.E.Butler & R.Roy) Materials Research Society Pittsburgh Pennsylvania (1991) 683-694.

[2] A.R.Lang A.P.W.Makepeace M.Moore and W.Wierzchowski *ibid* (1991) 557-560.

[3] W.Wierzchowski, M.Moore, A.P.W.Makepeace and A.Yacoot *J.Crystal Growth* 114 (1991) 209-227.

[4] A.R. Lang M. Moore A.P.W. Makepeace W. Wierzchowski and C.M. Welbourn *Phil Trans Royal Soc London A* 337 (1991) 497-520.

Prime novelty: *Accurate relative lattice parameter measurements*

Keywords: *Dislocations lattice parameters X-ray topography.*

Pełny tekst wystąpienia opublikowany będzie w materiałach z konferencji.

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SYNCHROTRON SPIKE TOPOGRAPHY OF NATURAL TYPE Ia DIAMOND

Natural type Ia diamond contains impurity platelet precipitates probably composed of nitrogen. They have diameters 10 to 100 nm, are only a few atoms thick and lie on {100} planes. Such platelets produce spikes in diffraction associated with some reciprocal lattice points [1]. Six spikes surround the 111 reciprocal lattice point: $\pm[100]$, $\pm[010]$ and $\pm[001]$, arising from platelets lying parallel to all three (100), (010) and (001) planes. Only two spikes, $\pm[001]$, however extend from the 331 reciprocal lattice point. This is because the platelets cause a displacement of approximately one-third of the lattice spacing: so in general there are no spikes for indices of reflexion divisible by three. Hitherto, topographic studies of reciprocal lattice spikes in diamond have concentrated upon the strong 111 reflexion. Spikes are weak and long exposures have been needed to record them: typically 100 hours from a conventional X-ray source. With a powerful source of synchrotron X-rays, such as at the SERC Daresbury Laboratory, other reflexions are now accessible. The novel feature is the topographic study of the 331 reflexion. Here the Ewald sphere intersects only one of the two spikes; rather than three of the six at the 111 reflexion. Three spikes give rise to three images on the topograph; which overlap unless large specimen-to-plate distances are employed with a consequent loss of resolution. Spike intensity follows an inverse square law with distance from the reciprocal lattice point. Associated with platelets are voidites [2]. These are {111} faceted defects of low electron density; and in certain diamonds, sheets of voidites replace platelets. Spikes in the $\langle 111 \rangle$ directions from the 111 reciprocal lattice point appear to arise from sheets of voidites [3]; and we have observed such spikes emanating from the 331 reciprocal lattice point.

[1] S.G.Clackson and M.Moore, *Industrial Diamond Review*, 49 (1989) 118-133.

[2] J.C.Barry, L.A.Bursill, J.L.Hutchison, A.P.Lang, G.M.Rackman and N.Sumida *Phil. Trans. Royal Soc. London A321* (1987) 361-401.

[3] A.R.Lang, G.Kowalski, A.P.W.Makepeace and M.Moore, *Phil. Mag.* 52 (1985) L1-L6.

Prime novelty: *High resolution 331 synchrotron spike topography.*

Keywords: *Platelets, spike topography, voidites.*

Pełny tekst wystąpienia opublikowany będzie w materiałach z konferencji.

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ASSESSMENT of Fe-DOPED SEMI-INSULATING InP CRYSTALS AS THE MATERIAL FOR MICROELECTRONIC DEVICES

Semi-insulating (SI) InP is becoming technologically important as the substrate material for integrated optoelectronics and high-speed microelectronic devices. In electronic applications, the InP crystals are used either as substrates for the growth of epitaxial layers, such as for integrated optoelectronic applications, or directly in ion-implantation-based technologies for the realization of such devices as metal-insulator-semiconductor field-effect transistors (MIS FETs) and integrated circuits.

Characterization of the material for these applications contains:

- Hall effect and conductivity measurements to estimate carrier concentration, mobility and resistivity,
- compensation ratio estimation,
- inhomogeneity of resistivity and Fe concentration,
- dislocation structure revealing.

Some SI-InP crystals obtained in ITME by Czochralski technique (LEC) have been characterized. The following methods have been applied:

- Hall effect and conductivity measurements at room temperature and in the range of $300 \div 400$ K,
- near-infrared absorption measurements allowing the estimation of the total iron concentration and the concentration in different charged states,
- sheet resistivity inhomogeneity measurements by the two-sonde method,
- dislocation structure revealing by chemical etching.

The obtained results are presented and discussed.

Material będzie przedstawiony w formie posteru

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CHARACTERIZATION OF EPITAXIAL SILICON FOR MOS VLSI INTEGRATED CIRCUITS BY DEEP LEVEL TRANSIENT SPECTROSCOPY AND MINORITY CARRIER LIFETIME MEASUREMENTS

Epitaxial silicon is widely used as starting material for small geometry CMOS devices. However, during the epitaxial growth a variety of native defects and impurities are introduced into the layers. When located in the active device regions, these point defects, particularly transition-metal impurities, have strong detrimental effect on the device performance. So, for the quality assessment of epitaxial silicon for MOS VLSI integrated circuits it is necessary to control the type and concentration of point defects as well as the minority carrier lifetime.

In this paper we present the results of characterization of epitaxial silicon by deep level transient spectroscopy (DLTS) and measurements of the generation lifetime in MOS structures. The epitaxial layers were deposited in a Gemini 1 vertical CVD reactor on 4 inch diameter p- and n- type (111) substrates. Both the majority and minority carrier traps with high capture cross-section were detected. The effect of the epitaxial growth conditions on the concentrations of deep-level defects and the generation life-time was established.

Material będzie przedstawiony w formie posteru

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CRYSTAL GROWTH OF SEMI-INSULATING INDIUM PHOSPHIDE

Semi-insulating indium phosphide (SI-InP) is very promising material for microelectronic devices and very high-speed integrated circuits. For this purposes a high-quality material is required characterized by a high resistivity, high carrier mobility and low dislocation density.

High partial pressure, high melting point temperature and chemical activity cause difficulties in growing InP crystals which fulfill the above requirements.

The main technological problems are:

- high purity raw material with carrier concentration low enough ($n \leq 10^{16} \text{ cm}^{-3}$) to be compensated by iron doping (maximum Fe solubility in InP is 10^{17} cm^{-3}),
- heating system construction allowing single crystal growth in $\langle 111 \rangle$ or $\langle 100 \rangle$ direction, without twins and thermal stresses,
- inhomogeneity of electrical and structural properties.

In this paper we present the results of our research concerning synthesis and crystallization processes.

The following synthesis methods have been applied:

- 3-zone melting,
- injection,
- gradient freeze.

The influence of the manufacturing method on the InP polycrystal quality has been investigated.

Czochralski method (LEC) has been applied for growing large-diameter InP crystals $\phi \geq 51 \text{ mm}$. Growing parameters have been established for crystallization in $\langle 111 \rangle$ and $\langle 100 \rangle$ directions. The influence of doping has been estimated.

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HEAT TREATMENT INFLUENCE ON THE MODIFICATION of SI-GaAs PROPERTIES

LSI and FET technology development requires high quality semi-insulating substrate materials. With regard to electrical and structural parameters and their homogeneity, SI-GaAs crystal properties have been modified by heat treatment. The influence of the temperature, annealing time and cooling rate have been analysed.

The material have been characterized before and after each heat treatment cycle by the investigation of:

- Hall effect, allowing the estimation of the resistivity, carrier mobility and concentration,
- near infrared absorption for the definition of EL2 centers concentration and distribution,
- LVM absorption for the assessment of carbon concentration,
- inhomogeneity of resistivity.

Electrical parameters changes, after the heat treatment, have been analysed as the result of the annihilation and generation of electrically active defects.

The results of our investigations are presented and discussed.

Material będzie przedstawiony w formie posteru

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METHODS OF INVESTIGATION OF REAL CRYSTALLOGRAPHIC STRUCTURE OF InP SINGLE CRYSTALS.

Structural defects in substrates of III-V materials strongly reduce the performance of devices made by ion implantation or epitaxial technique. For this reason it is important to determine the origin of these defects and their relation to growth conditions.

In this paper we report the results of complex structural studies performed on InP wafers. The InP single crystals were grown by Liquid Encapsulated Czochralski (LEC) method in $\langle 100 \rangle$ direction. The crystals investigated were undoped or doped with Sn, Fe, Zn, S. The defect structure of InP crystals has been studied using the following experimental techniques:

- selective chemical etching,
- integral catodoluminescence patterns,
- X-ray topography.

These techniques has been examined from the point of view of application to standard and special investigations. For routine control of material quality the two-step technique has been chosen:

1. Slight selective etching revealing impurity distribution, inhomogeneities, microprecipitates, faults of surface treatment.
2. Selective etching revealing dislocations and enabling determination of their density.

In order to explain the mechanisms of defect generation during the crystal growth and in the case of complex defects the more sophisticated techniques like X-ray topography and catodoluminescence studies should be used.

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MMIC'S ON GALLIUM ARSENIDE DEVELOPED IN THE INSTITUTE OF ELECTRONIC MATERIALS TECHNOLOGY

Basic MESFET technology comprising selective ion implantation, rapid thermal processing, plasma enhancement chemical vapor deposition, multilayer metallisation and submicron lithography was developed in 1989 - 1990.

In 1991 two MMIC's were designed, produced and investigated :

1. The feedback amplifier
2. The NOR gate

The feedback amplifier exhibits ultra-broadband stable performance starting from DC up to 1.5 GHz.

The two input NOR gate in the BFL (Buffered Fet Logic) arrangement exhibits excellent noise margins and time delay of less than 100 ps (fan out one).

DC and microwave parameters of IC's prove feasibility of a further development of gallium arsenide circuits in ITME.

Own layout design, pattern generation and mask making show prospect of starting activity with simple ASIC's on gallium arsenide as well. Basic cells for digital ASIC's are being developed at present under program supported by government.

Material będzie przedstawiony w formie posteru

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THE X-RAY INVESTIGATION OF DEFECTS IN LaGaO_3 SINGLE CRYSTAL

LaGaO_3 crystal belongs to Rare Earth Gallium Perowskites. Due to the lattice similarity LaGaO_3 is successfully used as a substrate for epitaxial growth of high temperature superconducting thin films. LaGaO_3 matches $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ better than other substrates. The 1.6% lattice misfit enables the growth of monocrystalline $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ film with sufficient structural perfection up to the thickness of $0.6\mu\text{m}$. The perfection of the layer is also limited by the perfection of the substrate. The structural defects in the substrate LaGaO_3 were investigated by means of X-Ray diffraction topography. It was established occurring the longitudinal volume defects. It was stated that these volume defects are crystalline as well, but with different interplanar spacing "d". Besides it was shown that in the defect region the planes (011) are continued by the planes (101) with the angle inclination of a few minutes. The separating plane is the (112). The angle between the (011) plane and the reflecting plane in the defect, estimated from our experiments is equal to the angle calculated from the crystallographic data.

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X-RAY INVESTIGATION OF THE PHOSPHORUS DIFFUSION IN MODULATED STRUCTURE

GaAs/GaAsP

The X-ray investigations were made for GaAs/GaAs_{0.7}P_{0.3} sample. The experimental diffractometric profile was fitted to the theoretical one in such a way that the profile of the interface between GaAs and GaAsP layers was changed as a function of the phosphorus concentration gradient.

The results obtained indicate the possibility of the phosphorus diffusion from GaAsP layer to GaAs layer.

The phosphorus concentration gradient was fixed.

Material będzie przedstawiony w formie posteru

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INCREASE OF LIMIT SOLUBILITY OF SOLIDS CAUSED BY TEMPERATURE GRADIENTS

An equation is enabling to compare levels of limit solubility of additives in solids under isothermal conditions and at temperature gradients were given.

A general formula to determine level of additives in solids and bring out to determine diffusion solubility in solids who was obtained by the use of well known models of solubility.

A formula to define changes of solubility levels in thermodiffusion processes at constant temperature gradient will bring out in the next time.

The concentration level of additives can determine knowing thermodiffusion parameters and limit solubility at isothermic condition. It was also shown that temperature gradient may increase or decrease concentration level of additives obtained under isothermic diffusion conditions which are depending on the direction of the concentration gradient.

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**APPLICATION OF DEEP LEVEL TRANSIENT SPECTROSCOPY FOR NONSTOICHIOMETRY CONTROL
IN III-V SEMICONDUCTORS**

The main electrical and optical properties of III-V semiconductors strongly depend on various point defects generated due to material nonstoichiometry. These defects involve a strong perturbation of the local lattice potential and create bound states with energies deep in the band gap. It is shown that the changes in the deep-level defects concentrations induced by the material nonstoichiometry can be observed using deep level transient spectroscopy (DLTS). The DLTS spectra for bulk crystals and vapour phase epitaxial layers of GaAs as well as for Si-implanted GaAs are compared. The effect of the growth conditions on the point defect concentration in the vapour phase epitaxial layers of $\text{GaAs}_{0.6}\text{P}_{0.4}\text{:Te}$ and GaP:N,S is also presented. The thermochemical reactions leading to formation of the grown-in deep-level defects in GaAs, $\text{GaAs}_{0.6}\text{P}_{0.4}$ and GaP are discussed and the experimental evidence for these reactions based on the results of DLTS measurements is given.

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THE X-RAY INVESTIGATION OF DEFECTS IN SrLaAlO₄ SINGLE CRYSTALS

The SrLaAlO₄ single crystals are used as a substrate material for obtaining high temperature superconductor epitaxial layers with uniform physical properties. Good quality of epitaxial layers requires both crystallographic perfection and appropriate physical properties of the substrate material. In the present paper we investigated the crystallographic perfection of SrLaAlO₄ crystals by means of X-ray topography. The samples were cut perpendicular to [100] direction. In some of the samples the present investigation revealed longitudinal but very thin volume defects. It was stated that these defects also contain crystalline material but with slightly different interplanar spacing.

Pełny tekst wystąpienia opublikowany będzie w materiałach z konferencji.

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THE PROFILE OF THE CHEMICAL COMPOSITION MODULATION WAVES IN THE $\text{Ga}(\text{As}_{1-x}\text{P}_x)/\text{GaAs}$
AND ~~$(\text{As}_{1-y}\text{P}_y)$~~ $(\text{As}_{1-y}\text{P}_y)/\text{GaAs}$ MULTILAYERED CRYSTALS BY MEANS OF X-RAY DIFFRACTION

$(\text{In}_x\text{Ga}_{1-x})$

The problem of finding the exact profile of the chemical composition in multilayered crystals by means of the X-ray diffraction consist in the best matching between experimental and theoretically calculated diffraction pattern. To this end a numerical procedure calculating the two-dimensional diffraction pattern was worked out, that requires only definition of the relative composition of P in (AsP) and In in (InGa) planes throughout the crystal. The procedure is not limited by the shape of the chemical concentration modulation wave, so that every change of the concentration could be taken into account, and its influence on the scattered amplitude calculated. The model of superlattice applied to the procedure enables calculating diffraction pattern for multilayers grown on substrates with non-zero cut-off angle.

Pełny tekst wystąpienia opublikowany będzie w materiałach z konferencji.

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THE EVALUATION OF ONE DIMENSIONAL DISTORTION WAVE IN A_3B_5 MULTILAYER STRUCTURE

From the diffraction topography studies it was found, that the lattice mismatch in the $GaAs_xP_{(1-x)}/GaAs$ multilayer structure is accomodated mostly by elastic strains. The dislocation density is much less than those required for complet strain relief. The parameters of one dimensional lattice distortion wave were found from the X-Ray diffraction profile analysis. It was established, that the best fit to the experimental data is obtained for a model in which the coherency strains are constrained by the substrate lattice.

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DETERMINATION OF THE REAL STRUCTURE OF THE GaAs(P)/GaAs MODULATED CRYSTAL

The semiconductor modulated (GaAs_(1-x)P_x)/GaAs, x=0.3 monocrystal was investigated by means of XRD. The differences were found in comparison to the spectrum of ideal modulated monocrystal. The crystal is divided into coherent scattering domains (CSD). The electron density model is presented in this case. The scattered intensity from single CSD was calculated as for ideal multilayered monocrystal. It follows from CSD model that the total intensity is a summ of the partial intensities diffracted by each CSD. According to this model, it was found that if there is a dispersion of the modulation wavelength in the crystal then it involves the broadening of the satellite reflections in relation to the nodal one. Analysis of the satellite reflections profiles was used to determinate the real coherent domain structure of the investigated crystal.

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THE INVESTIGATION OF THE DEVELOPMENT OF GROWTH SECTORS
AND OTHER CRYSTALLOGRAPHIC DEFECTS IN SYNTHETIC DIAMONDS GROWN BY
RECONSTITUTION METHOD

The recent development of the reconstitution method made possible growth of large synthetic diamonds of high perfection, containing low amount of metallic inclusions and dislocations. The principle of reconstitution method consist in the use of diamond powder as the carbon source instead of graphite. It enables keeping long crystallization processes at stable conditions. The diamonds grown with the reconstitution method are very interesting objects for studying with X-ray topography, which can provides many information about the growth process.

The important problem in synthetic diamond is connected with complicated crystallographic habit containing $\{111\}$, $\{100\}$, $\{311\}$ and $\{110\}$ faces. The various sectors contain different concentration of substitutional nitrogen causing considerable lattice spacing differences and stress at growth sector boundaries.

In present work we present some results obtained in a number of diamonds of the diameter up to 6 mm grown by reconstitution technique. These results were obtained by means of X-ray and cathodoluminescence topography and some optical methods of characterization. The X-ray topographic investigation was performed with the complementary use of different method: section, projection and double-crystal topography both with conventional and synchrotron sources of X-rays. The investigation included some original topographic experiments, as simultaneous recording of K_h and K_v beams, Bragg-case section topography and confirmation of the intrinsic stacking fault type based on Bragg-case double-crystal topographs.

The important result of present investigation was the identification of the crystallographic type of large number of dislocation. It was found that the dominant type of dislocation was mixed 30° dislocation along $\langle 211 \rangle$ directions. The dislocation usually emerged from metallic inclusions. Some highly populated dislocation bundles usually emerged from some metallic inclusions trapped close to the seed. Some

bundles were connected with particular growth sectors, according to the rule that energetically favorable position of dislocation is perpendicular to the growth surface. The observed stacking faults either originated on metallic inclusions or were formed by splitting of dislocations.

The investigation of growth sector pattern was mostly effective on the artificially introduced polished surfaces using both double-crystal and cathodoluminescence topography. It was possible to follow exactly the development of the growth sectors in large diamond of truncated octahedral habit on the four equally spaced sections made by the polished surfaces of two slabs prepared from this diamond. Some information about the configuration of the growth sectors inside the diamond was obtained from series of section topographs and from pairs of double-crystal K_h and K_o beamtopographs.

In one of the investigated diamond of cuboctahedral habit we observed additional "Maltese cross" growth feature in central (100) growth sector. We found that the centre of the "cross" was the dislocation bundle.

The double-crystal method was also used for evaluation the relative difference in lattice spacing between different growth sectors. From these measurements it was found that in the diamonds with dominated octahedral habit, the difference of nitrogen concentration between cube and octahedral sectors is greater than in the case of diamonds with dominant cube habit.