**2nd EUROPEAN SYMPOSIUM - X-RAY TOPOGRAPHY AND HIGH RESOLUTION DIFFRACTION, Berlin, Germany 5-7/09.1994**

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# THE NUMERICAL SIMULATION OF BRAGG-CASE TOPOGRAPHIC IMAGES OF DISLOCATION AND PRECIPITATES IN GaAs EPITAXIAL LAYERS

The numerical integration of Takagi-Taupin equations was used for simulation of the back-reflection double-crystal images of crystallographic defects in GaAs epitaxial layers. In particular, the images of dislocations inclined to the surface, point-like precipitates and misfit dislocation crossings were studied in aspect of their identification in the practical examination of epitaxial layers.

The actual program used the formal assumption of the finite crystal thickness. That allowed us to use the integration grid with boundaries situated far from the defects, and less time consumable extension of the simulated area. The simulations took into account the diffraction effects both in the layer and in the substrate and the diffused lattice parameter profiles. The divergence of the beam was taken into account by adding 50-100 plane-wave images. That improved the reality of the simulations by dumping some of the interference fringes.

We found that the images of dislocations, especially those inclined to the surface, are dominated by the direct dilatation-orientation contrast. The similarity of the contrast coming from Takagi-Taupin theory and that given by Bonse's approximation was confirmed. The greatest amount of the interference effect was observed for the images of point-like defect. The difference of the images taken in the equivalent points of the layer and the substrate peak was relatively small for the dislocations inclined to the surface.

The theoretical images were compared with the experimental topographs of 4-5μm thick GaAs epitaxial layers grown with *MOCVD* technique. The GaAs substrates with low concentration of defects, topographically examined before the growth process, were used. A high perfection of the epitaxial layers was confirmed and most of the dislocations in the layers were found to be the continuation of those in the substrates. Several cases of good correspondence of simulated and experimental images of dislocations was found.

The work was sponsored by the State Committee for Scientific Research (KBN), (grant no. 1130/55).

Tekst wystąpienia prezentowano na sesji posterów.
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## INTERNATIONAL CONFERENCE ON DIFFUSION AND REACTIONS: FROM BASIS TO APPLICATIONS
## Kraków, Poland 7-9/09.1994

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## DIFFUSION EFFECTS INTO ALUMINA CERAMICS DURING ACTIVE BRAZING

Diffusion in alumina ceramics-active solder - FeNi42 alloy joint was investigated by means of XRD and XRFA,$Cu_3Ti0_5$, $Cu_2Ti_20_4$, $Cu_3Ti0_4$, $Cu_3Ti0_4$, $Fe_3Ti0_4$, $Ni_3Ti$ have been found at the ceramics surface adjacent to joint. It was also found that iron and nickel diffuses through out solder layer into ceramics.

The synthesis of the diffusional interlayers on the ceramics surface is of great importance for the production of strong bonding of ceramics to metal.

To improve the wettability of ceramics by active solder, surface of ceramics was covered by Ni and Cu layer.

Irrespective of the means of preparation of the surface ceramics, the some process parameters (temperature of 1173K, vacuum of $2 \cdot 10^{-5}$ Tr and time of 60 min.) produced the above mentioned compounds within the diffusional interlayer.

Tekst wystąpienia zaprezentowano na sesji posterów.

## ESSDERC'94 - 24TH EUROPEAN SOLID STATE DEVICE RESEARCH CONFERENCE, Edinburgh, Scotland 11-15/09.1994

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### OXYGEN PRECIPITATION IN Cz-Si UNDER UNIFORM STRESS

Phenomena related to oxygen precipitation in Czochralski grown silicon subjected to hydrostatic pressure up to 2.5 GPa at ambient temperature and up to 1.35 GPa at 1230 - 1620K were investigated by FTIR, X-ray methods, chemical selective etching and TEM. Depending on the initial oxygen concentration and preannealing conditions, uniform stress influences markedly the oxygen - related defect structure of Cz-Si.

Tekst zaprezentowano na sesji komunikatów.

## NANO'94 - SECOND INTERNATIONAL CONFERENCE ON NANOSTRUCTURED MATERIALS, Stuttgart, Germany 3-7/10.1994

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### MÖSSBAUER STUDY OF AMORPHOUS AND NANOCRYSTALLINE FeZrBCu ALLOYS

The radio-frequency (rf) collapse of the magnetic hyperfine structure (hfs) in the

Mössbauer spectra of soft ferromagnetic materials exposed to an rf field of 20 Oe at 60 MHz is employed to study the structural and magnetic properties of $Fe_{93-x-y}Zr_7B_xCu_y$ (x= 6, 8,12; y= 0, 2) alloys in the amorphous and nanocrystalline states. The rf sideband effect, induced by an rf field in magnetostrictive materials, allows one to follow the changes of magnetostriction due to the formation of the nanocrystalline phases which are formed as a result of annealing of the amorphous alloys at 500-600°C. The rf collapse and sideband effects depend strongly on the thermal treatment in the range of 400-800°C and on the alloy composition. The results show that the complete rf collapse of the magnetic hfs to a quadrupole doublet occurs only in the amorphous phase. Samples with a dominating nanocrystalline phase reveal spectral components consisting of the broadened, collapsed single line (related to the cubic nanocrystalline phase) and partly or marginally collapsed magnetic hfs (related to the phases with larger magnetic anisotropy). The unique rf Mössbauer technique allows the study of the microstructure and magnetic properties (anisotropy, magnetostriction) of nanocrystalline phases formed in FeZrBCu alloys and distinguishes them from the amorphous and microcrystalline ones.

In addition to rf-Mössbauer experiments, conventional Mössbauer measurements allowed the identifcation of the phases formed due to annealing of amorphous FeZrBCu alloys in the temperature range of 400-800°C. The Mössbauer experiments were supplemented by DSC determination of the crystallization temperatures.

Tekst wystąpienia zaprezentowano na sesji posterów.
Pełny tekst wydrukowany będzie w: "Nanostructured Materials".

## PIEZO'94 - 8th PIEZOELECTRIC CONFERENCE Zakopane, Poland 5-7.10.1994

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## TV-IF SAW FILTERS IN POLAND - DESIGN, FABRICATION AND PARAMETERS

A family of TV-IF SAW filters has been designed and put into mass production. The filters were designed on 128° YX $LiNbO_3$ substrate. The paper will describe the design method, fabrication process and parameters of the filters. It will discuss the most important problems concerning TV-IF filter design on 128° YX $LiNbO_3$ sub-

strate. The paper will outline the filter synthesis method which enabled us to compensate efficiently for some second-order effects.

We will also present the fabrication process.

Filter measurement results will be presented and compared to theoretical predictions. Finally, filter parameters together with corresponding technical requirements will be shown.

Tekst wystąpienia zaprezentowano na sesji referatów - jako referat zamawiany.
Pełny tekst wydrukowany będzie w materiałach z konferencji.

**PIEZO'94 - 8th PIEZOELECTRIC CONFERENCE**
**Zakopane, Poland 5-7.10.1994**

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## SAW FILTERS FOR DIGITAL RADIO COMMUNICATION SYSTEMS (TYPES: FP-7010A AND FP-7030C)

Two types of 70 MHz SAW filters have been designed. The filters are currently applied in IF stages of 11 GHz digital radio receivers as selective components shaping the frequency responses of the receivers. In comparison with the domestic filters produced up to now the new filters exhibit small passband ripple and group delay ripple and high stopband attenuation.

The substrate material used for the design is 128°YX lithium niobate. The filters are packaged in a cold-welded dual in line metal case (23.5 mm × 21.0 mm × 7 mm).

The poster will present topology, parameters, amplitude and group delay responses of the filters.

Tekst zaprezentowano na sesji posterów.
Pełny tekst wydrukowany będzie w materiałach z konferencji.

**PIEZO'94 - 8th PIEZOELECTRIC CONFERENCE**
**Zakopane, Poland 5-7.10.1994**

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# CZOCHRALSKI GROWTH OF $Li_2B_4O_7$ SINGLE CRYSTALS

Surface acoustic waves (SAW) are widely used for many devices such as delay lines, resonators, oscylators and filters. Characteristics of SAW devices largely depend on piezoelectric materials so the selection of candidates as a substrate for SAW used is of great importance. Several substrates like $\alpha$-$Si0_2$, $LiNbO_3$, $LiTaO_3$ are used in commercially products. However these materials have certain disadvantages in wide applications. For instance poor electromechanical coupling for $\alpha$-$Si0_2$ makes them unsuitable for devices with large bandwidth while the poor temperature coefficient of delay (TCD) in $LiNbO_3$ makes these materials unsuitable for high stability applications.

Lithium tetraborate $Li_2B_4O_7$ (LBO) is relatively new piezoelectric material that has a zero TCD and moderatly high coupling coefficient. $Li_2B_4O_7$ single crystals belong to tetragonal structure with the 4 mm point group. Material melts congruently in 917°C so single crystals of LBO could be grown by the conventional rf heating Czochralski technique in air atmosphere.

As a starting material in our experiments, commercially available $Li_2CO_3$ and $B_2O_3$ with 99,995% purity, were used.

Transparent, and colorless LBO single crystals up to 70 mm in diameter and 50 mm in length were successfully grown. It was found that suitable pulling rate for these crystals was 0,3-0,35 mm/h with rotation 3-4 rpm: The serious problem in growing of LBO crystals was the danger of cracking. It is possible to avoid this cracking by a suitable adjustment of the thermal conditions in the vicinity of the melt surface and in the space above the melt.

Properties of acoustic surface waves of Rayleigh and Bleustein - Gulyaev types were measured and correct parameters of the crystals were confirmed.

Tekst wystąpienia zaprezentowano na sesji referatów.
Pełny tekst wydrukowany będzie w czasopiśmie "Elektronika".

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## THE INFLUENCE OF RARE EARTHS, MgO AND Fe DOPING ON PIEZOELECTRIC PROPERTIES OF $LiNbO_3$

Lithium niobate is one of the most commonly used piezoelectric and electrooptic materials. For $LiNbO_3$ doped with rare earths, it is possible to obtain lasing and modulation effects simultaneously.

In this work $LiNbO_3$ single crystals doped with Er, Er + Tm, Er + Tm + MgO and Fe were grown by the Czochralski method. The dopant distribution was uniform and crystals were free of macroscopic defects. Their dimensions were up to 80 mm in diameter and up to 90 mm in length. After growth runs crystals were poled in the separate operation.

The wafers of "Y" orientation were cut out and SAW velocity was measured for propagation in "Z" direction. SAW - IF -TV filters of the type FT-386 were prepared with the use of the doped wafers and their amplitude characteristics were compared to those of filters prepared with undoped wafers.

Tekst wystąpienia zaprezentowano na sesji posterów.
Pełny tekst wydrukowany będzie w czasopiśmie "Elektronika".

**PIEZO'94 - 8th PIEZOELECTRIC CONFERENCE**
**Zakopane, Poland 5-7.10.1994**

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## TECHNOLOGY AND MEASUREMENTS OF ST-CUT QUARTZ SUBSTRATES

Large quartz crystals were grown by the hydrothermal technique. The infuence of growth time and the pressure of liquid on the crystal parameters were studied. ST-cut wafers, for use in surface acoustic wave (SAW) device application, were fabricated. The diameters of the wafers were equal to 76 mm (with seed) and 57 mm (without seed). The thickness of the wafers was equal to 0.5 mm. One side of the wafers was polished and the other one was lapped. It was found, that chemical cleaning of the wafers with seed was very difficult, because it was impossible to remove the polishing powder from the microchannels near the seed. Therefore only seed free wafers were used for SAW measurement. To verify the SAW properties of the wafers, two port SAW resonator was designed, fabricated and tested. At the frequency of 303 MHz the loaded Q of the resonator (in the 50 Ω system) was equal to about 10 000. It means, that the ST-cut substrates can be used in SAW technology.

This work was supported by the State Committee for Scientific Research (KBN) under grant No. 8550105104.

Tekst wystąpienia zaprezentowano na sesji komunikatów.
Pełny tekst wydrukowany będzie w materiałach z konferencji.

## CAS'94 - 17th EDITION OF ANNUAL SEMICONDUCTORS CONFERENCE, Sinaia, Romania 11-16/10.1994

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## IR STUDY OF OXYGEN PRECIPITATE GROWTH IN CZ-Si CRYSTALS DURING HEAT TREATMENT UNDER STRESS

Oxygen is the most important impurity in Czochralski grown silicon, Cz-Si, single crystals. It can exist in different states (interstitial atoms in the Si lattice and bridging atoms in the silica precipitate phase) depending on crystal growth and heat treatment conditions. A large stress can be generated at the silicon / precipitate interface affecting the processes of the silica phase formation or dissolution. Therefore,

investigation of stress influence on the behaviour of oxygen in Cz-Si crystals subjected to annealing is of special interest [1].

Experimental technique

Cz-Si samples of 600 μm thickness were cut from the wafers of 100 mm diameter, with the initial oxygen concentration, $c_o$, up to $1.2 \times 10^{18}$ cm$^{-3}$, and were preannealed at 720-1000K for up to 200 hours to create nucleation centers for oxygen precipitation. The samples were then subjected to high temperature annealing (HT) at up to 1620K under atmospheric or hydrostatic pressure ($10^7$ -$1.35 \times 10^9$ Pa) for 5 min - 5 hours (HP - HT treatment).

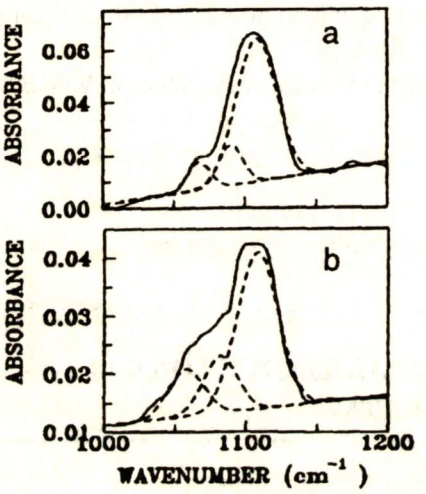


**Fig. 1.** Deconvolution of Si-O stretching band for oxygen in silicon into gaussian profiles. Sample was annealed at 1000 K for 20 hours (a) and then subjected to HT-HP treatment at 1400 K for 5 hours under $10^9$ Pa (b).

The IR - transmission spectra were measured in the range of wavenumbers 900 - 1200 cm$^{-1}$ for both Cz-Si and reference FZ-Si crystals. Deconvolution of the IR absorbance curves into the gaussian profiles was performed to separate interstitial (elementary band at 1107.5 ± 0.5 cm$^{-1}$ with full width at half maximum 33 ± 1 cm$^{-1}$ ) and bridging oxygen (the difference between spectral curve and the mentioned peak) absorption. In the last case a structural configuration of the oxygen containing components (main elementary bands at 11060 and 1080 cm$^{-1}$ ) was also determined according to [2]. The examples of spectrum deconvolution are presented in Fig.1. The oxygen concentration in silica phase, $c_p$, was estimated from absorbance of the corresponding part of the IR spectrum.

The IR data were compared with those obtained by complementary methods: chemical selective etching, X-ray topography and X-ray anomalous transmission measurements.

Results and discussion

The $c_o$ and $c_p$ dependences on the pressure applied during heat treatment are shown in Fig.2. It is seen that whereas the HT treatment results in the decrease of $c_o$, the HT - HP annealing leads to its increase. The $c_p$ value increased substantially due to the HT treatment. In the case of one-step preannealing the $c_p$ value did not depend practically on the pressure value (Fig. 2a). It increases, however, with pressure in the case of two - step preannealing (Fig. 2b).

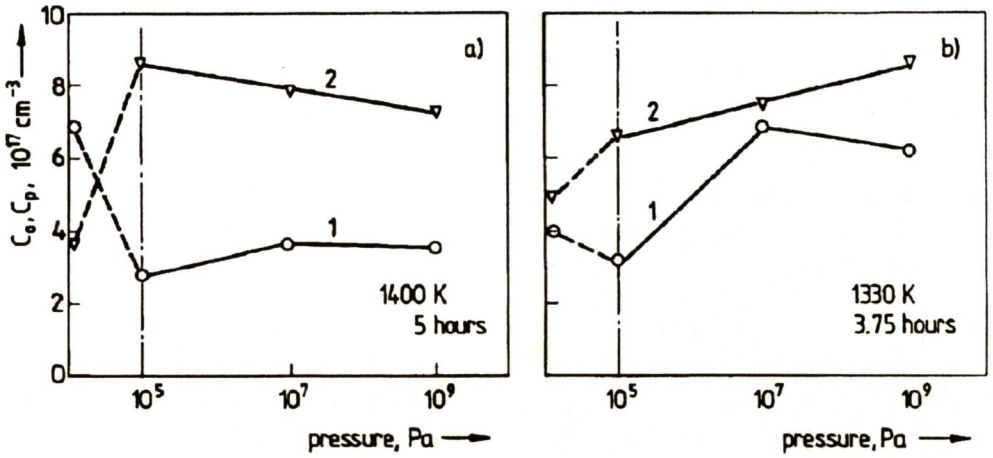


**Fig. 2.** Dependences of $c_o$ (curve 1 ) and $c_p$ (curve 2) on pressure during HT-HP treatment. Samples were subjec- ed to one-step (1000 K, 20 hours) preannealing (a), and two-step (720 K, 96 hours + 920 K, 96 hours) preannealing (b).

These changes were fast and took place even during the shortest (5 min.) HP - HT treatment (Fig. 3). Annealing at 1400K under atmospheric pressure caused the same changes in $c_o$ and $c_p$ after at least 20 hours of treatment.

In all cases the ratio of concentrations of the bridging oxygen atoms incorporated in different structural components of silica phase has been changed. The fraction of the dense and strained part of silica phase (elementary band at 1060 $cm^{-1}$, presumably connected with the 4-fold $SiO_4$ rings [2]) increased with $c_p$. The corresponding oxygen concentration value was equal to 0.25 $c_p$ for the non-treated samples amounted to 0.3 $c_p$ and 0.5 $c_p$ after preannealing and the HT - HP treatment, respectively. Taking into account that strained part of silica phase exists at the silicon - precipitate interface (in other words, it represents the outer coating of precipitate), one can conclude that the HT-HP treatment leads to formation of a large number of relatively small oxygen precipitates. This conclusion is confirmed by the data of X-ray anomalous transmission measurements: the high precipitate concentration was

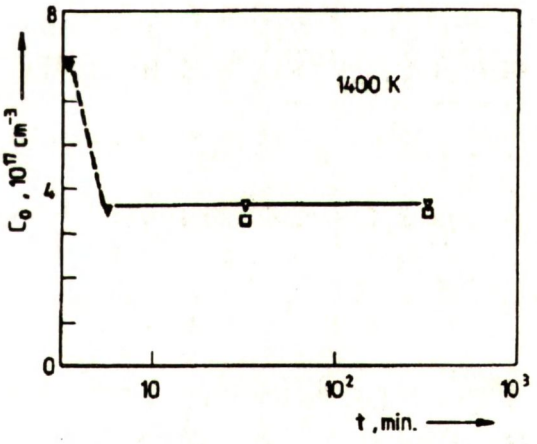


**Fig. 3.** Dependence of $c_o$ vs. annealing time for samples pre- anneled at 1000 K for 20 hours and then subjected to HT-HP treatment under $10^7$ Pa (triangles) or $10^9$ Pa (squares).

connected with the decrease of the anomalous transmission value resulting from the creation of strained areas in the crystal.

Conclusions

High hydrostatic pressure influences the concentration of oxygen in the form of interstitials and the process of precipitate phase growth. It results in introducing the oxygen atoms into the lattices both of silicon and precipitate materials and accelerates the process of silica phase creation; the latter is produced in the form of a rather large number of relatively small inclusions. It can not be excluded also that the hydrostatic pressure can influence the critical radius of oxygen nucleation centres and oxygen precipitates thus causing dissolution of the smallest ones [3].

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Tekst wystąpienia zaprezentowano na sesji komunikatów.
Pełny tekst wydrukowany będzie w materiałach z konferencji.

## 1st INTERNATIONAL CONFERENCE ON MATERIALS FOR MICROELECTRONICS, Barcelona, Spain 17-19/10.1994

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## EFFECT OF PREANNEALING CONDITIONS ON FORMATION OF DEEP ELECTRON TRAPS IN CZ-Si ANNEALED AT HIGH PRESSURE

Hydrostatic pressure (HP) applied to monocrystalline Cz-Si with oxygen precipitates (OP) can modify the state of stress at the defect/matrix boundary. As a result,

the primary Cz-Si defect structure can be altered by creation or annihilation of stacking faults and/or dislocations. Thus, the monitoring of Cz-Si defect structure after annealing at high pressure and high temperature is of great importance in terms of internal gettering processes. In this study deep level transient spectroscopy (DLTS) has been employed to monitor the effect of preannealing conditions on the formation of deep electron traps in Cz-Si during the isochronal 4-hour annealing at a pressure of 1.1 GPa and a temperature of 1120 °C. Apart from DLTS studies, the X-ray anomalous transmission $I_a$ was measured and the Bond's method was used to determine the lattice constant a. The n-type Cz-Si samples with the surface oriented along <100> direction were used. The net donor concentration was ~ 1 x $10^{15}$ $cm^{-3}$ and the interstitial oxygen concentration was in the range of (6.5 - 8.6)x$10^{17}$ $cm^{-3}$. The OP nucleation centres were generated by the preannealing processes labelled as I, II and III : I- heat treatment at 450 °C for 26 h, II - heat treatment at 450 °C for 96 h plus additional heat treatment at 650 °C for 96 h, III - heat treatment at 650 °C for 96 h.

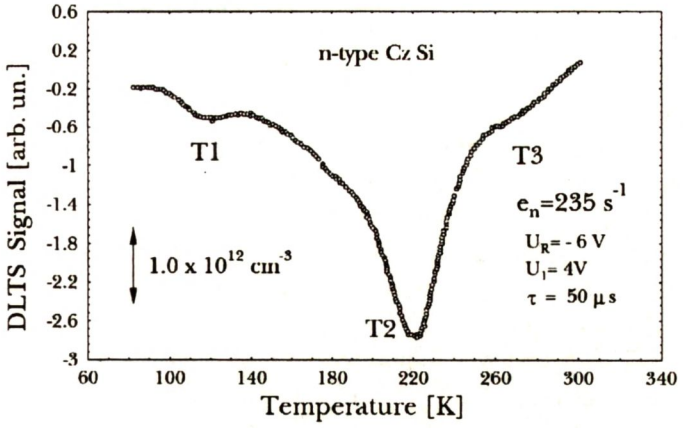


**Fig.1.** DLTS spectrum of n-type Cz-Si subjected to the 4-hour annealing at a high pressure ( 1.1 GPa) and high temperature ( 1120 °C) following the process II. $N_{T3}$= 7.7x$10^{11}$ $cm^{-3}$, $I_a$ = 60 %, a= 0.5430800 nm.

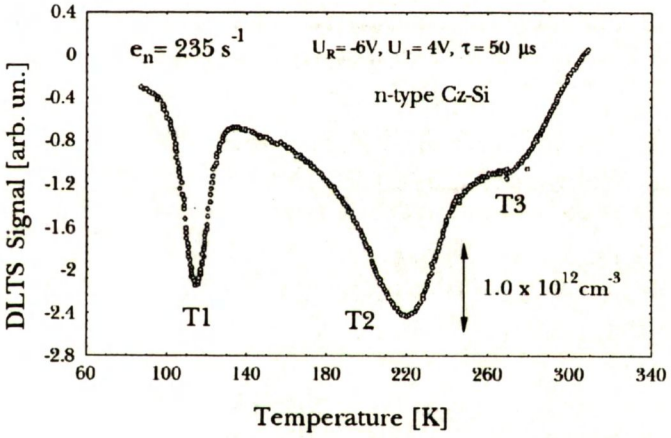


**Fig.2.** DLTS spectrum of n-type Cz-Si subjected to the 4-hour annealing at a high pressure ( 1.1 GPa) and high temperature ( 1120 °C) following the process III. $N_{T3}$= 1.4 x $10^{12}$ $cm^{-3}$, $I_a$ = 27.5 %, a= 0.5430659 nm.

In the case when high-pressure annealing followed process I, two electron traps T1 ( $E_a$= 0.20 eV, $s_a$ = 1.7x10$^{-14}$ cm$^2$ ) and T2 ($E_a$= 0.45 eV, $s_a$ = 6x10$^{-14}$ cm$^2$) were detected. The trap T1 is likely to be a thermal donor where as the trap T2 is attributed to the nickel contamination, for the piece of nickel foil was put into the high pressure chamber to getter other impurities. For the samples subjected to the highpressure annealing after processes II and III, additional trap T3 ($E_a$= 0.60 eV, $s_a$ = 2x10$^{-12}$ cm$^2$ ) was observed. The T3 centres are related to the dislocation states. DLTS spectra showing the changes in the material defect structure resulting from different preannealing conditions preceding the high-pressure annealing are illustrated in Fig.1 and Fig.2.

This work was supported by grants 8 8027 92 03 and 8 0337 91 01 from the State Committee for Scientific Research, (KBN).

Tekst wystąpienia zaprezentowano na sesji posterów.

## 11 th - CONFERENCE ON SOLID AND LIQUID CRYSTALS - MATERIAL SCIENCE AND APPLICATIONS Zakopane, Poland 23-27/10.1994

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## APPLICATION OF AUTOMATED POLARIMETER-MACROSCOPE FOR INVESTIGATION OF OPTICAL INHOMOGENEITY IN OXIDE SINGLE CRYSTALS

A computer-controlled polarimeter-macroscope, working on a novel principle, has been constructed. Two large sheet polarizers are rotated simultaneously by a step motor versus an immobile retardation plate placed between them in a parallel beam of light. Four images of the plate are grabbed by the video frame grabber (a modified Carré method) of the VFG/TV-camera detecting system at four increment angles of the polarizers' transmission axes versus the base of the arrangement. The intensities of light transmitted by the analyser in every pixel of these images undergo a suitable analysing procedure and on the basis of such calculations three maps can be plotted on the entire area of the plate: birefringence (or residual stress-induced birefringence in isotropic crystals), the principal azimuth (principal residual stress direction) and

transmission. The maps can be plotted either in grey-shaded version or as 3-D or 2-D contour plots.

It is estimated that the error/sensitivity of retardation measurements is not worse than $4x10^{-3}$ radians, corresponding to birefringence error of $5x10^{-7}$ for 0.8 μm wavelength and 1 mm thick plate. The transmission error is lower than 1 % in any circumstances. The principal azimuth error is negligible for moderate values of the cosine of retardation, where as it increases dramatically for retardations approaching 0, 2p, 4p etc. radians, being infinite at these boundary values.

Plane-parallel plates cut out from oxide crystals with diameters ranging from 13 mm ( $Y_3Al_5O_{12}$ ) through approximately 30-50 mm ( $CaF_2$, $BaF_2$) up to 100 mm ($LiNbO_3$) were studied in this arrangement. In most cases isotropic samples including Z-cut $LiNbO_3$ were investigated. Samples were usually cut out perpendicularly to the growth directions of the crystals. Their faces were optically polished.

In the first step of such investigations samples were examined in the usual plane polariscope (macroscope) configuration. In the crossed configuration of polarizers dark isoclinic lines and isochromatics corresponding to even orders of retardation could be observed in the polariscopic images, whereas in the parallel configuration of polarizers only isochromatics corresponding to odd orders (usually 1/2 ) of retardation were sometimes observed. In the polarimetric configuration (the angle between the transmission axes of the polarizers equals 45") the above mentioned maps were next plotted. A quasiradial stress-induced birefringence distribution was observed in wafers cut out from the YAG crystals pulled in the <111> direction and in Z-pulled $LiNbO_3$. The maximum birefringence being found at or very close to the perimeter of the samples. The absolute magnitude of birefringence in samples cut out from the bottom parts of the crystals, was typically larger than in adequate samples descending from the top parts. Such birefringence/stress distribution corresponds well to theoretical predictions of residual stress generation in Czochralski-grown crystals published elsewhere.

Tekst zaprezentowano na sesji referatów.
Pełny tekst zamieszczony będzie w: "Proceedings of SPIE".

**11 th - CONFERENCE ON SOLID AND LIQUID CRYSTALS - MATERIAL SCIENCE AND APPLICATIONS**
**Zakopane, Poland 23-27/10.1994**

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# CRYSTALS FOR FREQUENCY DOUBLING AND WAVEGUIDE DEVICES

Nonlinear optics is a vast field requiring materials with different performance features. Rapidly developing world communication demands for UV, blue or green light for computers, photolitography, colour displays and others and therefore for novel, more and more excellent nonlinear and electrooptic materials. Many inorganic and also organic crystals are presently known as the materials for this kind of applications.

Device quality NLO single crystals most widely used are: KDP, LBO, $KNbO_3$, KTP, BBO. Except KDP, which is grown from water solution (DKDP from heavy water) others are produced by flux technique. Growth process is carried on for about 2 month or longer. It is possible to grow BBO crystals by the Czochralski method without any flux.

Properties of BBO crystals in comparison with other NLO materials and conditions of Czochralski growth are discussed. Good optical quality BBO crystals obtained by Czochralski technique were verified by X-ray diffraction analysis. The samples were irradiated with Nd:YAG beam and green light 530 nm was observed.

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# THE X-RAY TOPOGRAPHY INVESTIGATION OF DEFECTS IN ERBIUM DOPED $LiNbO_3$

Lithium niobate is a material of considerable interest in view of its unusual ferroelectric, optical, acoustic and piezoelectric properties. Recently, we noticed the progress in the technology of growth of $LiNbO_3$ with rare earth dopants. It offers the possibility of obtaining crystals of modified properties suitable for using in new applications e.g. in laser technology. In this new application good quality is of great importance.

In the present work we investigated some new types of lithium niobate crystals by means of Lang X-ray topography, in particular the $LiNbO_3$ Czochralski grown crystals heavily doped with erbium. The Lang topographs were compared with microscopic observation of etch patterns.

It was found that presently investigated crystal with erbium concentration close to 0,24% contained low density of dislocation and other defects. That may be expected to be the result of doping, disturbing the glide motion and multplication of dislocations. Some of the samples contained the dislocation with concentration at the level $100/cm^2$, suitable for identification of their crystallographic type. The topographs revealed a certain concentration of small precipitates and in some cases also boundaries of ferroelectric domains. The dislocations and domain boundaries were revealed also by selective etching. A number of dislocations have been identified on the basis of topographs in reflections from sets of equivalent lattice planes e.g. (2110), (1210), (1120).

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# THE CZOCHRALSKI GROWTH OF $SrLaGa_3O_7$ SINGLE CRYSTALS AND THEIR OPTICAL AND LASING PROPERTIES

Single crystals of $SrLaGa_3O_7$ (SLG) belong to a large family of compounds of the general formula $ABC_3O_7$ where A = Ca, Sr, Ba; B = La, Gd and C = Ga, Al. These compounds crystallize in the tetragonal melilite structure which belongs to point group 42 m and to space group P42 m. The structure of $ABC_3O_7$ exhibits many distortions which are the result of the disordered occupancy of neighbouring sites by ions of significant differences in ionic radii and valence.

In this work, single crystals of $SrLaGa_3O_7$ undoped and doped with at. 5 at% and 10 at% of neodymium were grown by the Czochralski technique.

A heating system with good thermal insulation, an iridium crucible and an afterheater were used in order to achieve appropriate radial and axial temperature gradients in the crystallization chamber. Low temperature gradients and flat crystal-melt interface could provide good optical quality crystals free of internal stresses.

All SLG single crystals were grown in [001] direction. The pulling rate was 2-3 mm/h and the rotation rate 50 rpm. Their composition and the dopant distribution 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 distribution was uniform. The Nd 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 200-800 nm were measured, then laser rods were cut and their generation properties were evaluated. It was found that in the free-running mode laser thresholds are few

times higher for SLG compared to a good quality YAG rods, but the shape efficiency is lower only by several percent compared to YAG's. From the giant-pulse mode generation, in the passive Q-switch modulation measurements, it is evident that for SLG laser rods the output energy is twice greater than for the corresponding YAG rods. This phenomenon can be utilized in laser pulsed systems, which require high energies, e.g. surgery, stomatology, laser marking etc.

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## A NEW $V_{As}$ RELATED ABSORPTION LINE IN SI GaAs

The optical measurements of semi-insulating GaAs are presented in spectral region 1400-500 $cm^{-1}$. The new absorption lines X5 (1201.8 $cm^{-1}$) and X4 (1317.5 $cm^{-1}$) are observed. It has been found that they are related to electrically active defects and are electronic transitions in nature.The coupling of the defect responsible for these lines with the lattice is stronger than CC model predicts. 0ur results seem to suggest that the lines are related to $V_{As}$ defect.

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# SINGLE CRYSTAL GROWTH AND OPTICAL PROPERTIES OF $LiNbO_3$ DOPED WITH $Er^{3+}$, $Tm^{3+}$ AND MgO

Single crystals of $LiNbO_3$:$Er^{3+}$, $LiNbO_3$:$Er^{3+}$+$Tm^{3+}$ and $LiNbO_3$:$Er^{3+}$+$Tm^{3+}$+Mg0 were grown by the Czochralski method starting from the congruently melting composition.

Uniformly doped, good quality crystals were obtained at pulling rate 2 mm/h and rotation rate 8 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, free of macroscopic defects and pale-pink The poling of the single crystals was made in the separate operation. Electrodiffusion was not observed during poling which was confirmed by X-ray microprobe analysis.

The absorption spectra of $Tm^{3+}$, $Er^{3+}$ and Mg0 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 $Tm^{+3}$ and $Er^{3+}$ ions resulting in changing of absorption spectra structure for particular terms related to $Tm^{3+}$ and $Er^{3+}$ 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 MEASUREMENTS OF THE EPITAXIAL LAYERS THICKNESS BY MEANS OF PENDELLÖSUNG FRINGES

The determination of the epitaxial layers and superlattices thickness from the FWHM of the Bragg reflection, found in the literature, is not valid.

Much more precise method consist of analysis of the maxima location of the following Pendellösung fringes.

Dynamical theory of X-Ray diffraction implies that for the thickness less than the extinction length, the thickness of the layer can be properly evaluated employing Scherrer formula of kinematical theory.

This method is valid for nonabsorbing crystals in the symmetrical the Bragg case.

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## THE INVESTIGATION OF THE DEFECT PROPAGATION FROM THE SUBSTRATES TO GaAs EPITAXIAL LAYERS

The influence of defects existing in the substrates on the quality of deposited layers was studied for several types of the epitaxial layers, grown both by the conventional chemical deposition and *MOCVD* method.

The epitaxial layers were deposited on the substrates examined previously by Lang and double-crystal topographic methods. The same methods were used in a wide program of characterization after deposition of the epitaxial layers. The substrates used for deposition included both those with low dislocation density, obtained by intensive doping and low doped materials with distinct "cellular structure". The deposited layers were in the thickness range 3-5 μm, preserving the intense multiplication of the defects.

The topographic investigations were supported by the application of simulation of the back-reflection double-crystal images of crystallographic defects in GaAs epitaxial layers. In particular, the images of dislocations inclined to the surface, of point-like precipitates and misfit dislocation crossings were studied in aspect of their identification in the practical examination of epitaxial layers. A series of programs, both for the simulations in the substrates and epitaxial layer were produced.

We confirmed the important role of the defects present in the substrates, on the quality of the layer. That is particularly important in the case *of MOCVD* technology. High quality of the epitaxial layer grown with *MOCVD* method was confirmed and most of the dislocations in the layers were found to be the continuation of those in the substrates

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## TOWARD EPI-READY OF InP WAFERS

The epitaxial growth on semi-insulating $A^{III}B^{V}$ substrates is of large importance for the integration of high speed electronic and optoelectronic devices. Nowadays, the substrate materials for epitaxial process are wafers with surface status - *ready for*

*epitaxy.* Both, semi-insulating GaAs and InP materials are widely used in the production of multilayer structures by MBE and LP-MOVPD epitaxial methods. However, InP being twice softer and with greater chemical activity then GaAs is more difficult in preparation then GaAs with known epiready technology.

In this publication, we present the essential results of investigation for cutting, lapping and polishing process of InP:Fe 2" wafers. We indicate basic technological causes of surface imperfection of substrate wafer where epitaxial layer has been already created. We also present SEM pictures of various defects of epitaxial layer obtained by LP-MOVCD on SI InP wafer.

Presented experiments generally show wide and complicated problems in producing substrate SI InP wafers for epitaxial layer. It is a synthesis of essential factors which influence the high quality of substrate materials.

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## INFLUENCE OF THERMAL, CONDITIONS ON SI-GaAs CRYSTAL GROWTH BY LEC METHOD

The aim of this work was to find the 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 were grown by HPLEC method on the high pressure puller GALAXIE MARK IV. Multiheater graphite systems for 6 " and 8 " quartz and pBN crucibles were constructed and optimized (for 5kg and 8kg of GaAs charge).

In these systems the following characteristics were made:

- 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 ambient gas pressure for Ar and $N_2$.

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

SI-GaAs crystals of 3" and 4" diameter were obtained and the influence of growing conditions on the crystal properties, especially on the structural quality, observed.

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## CRYSTAL GROWTH OF UNDOPED SI-InP

Semi-insulating indium phosphide (SI-InP) is becoming important as the material for integrated optoelectronic and high speed microelectronic devices. Large crystals are usually grown by the Liquid Encapsulated Czochralski technique (LEC).

Until now semi-insulating behaviour in InP i.e. resistivity $\rho \geq 10^7$ Ωcm, could only be achieved by doping with iron ($N_{Fe} \geq 5 \cdot 10^{16} cm^{-3}$). But the dopant contained in the semi-insulating substrates causes during the processing an out-diffusion of Fe into adjacent epitaxial layers. Thus it is very important to obtain undoped semi-insulating InP.

In this work Fe doped and undoped InP crystals were grown in [100] orientation by the High Pressure LEC technique from the polycrystalline charge 1500g. Ultra-high purity polycrystalline material for the monocrystallization process has been obtained by the phosphorus vapour injection into indium under the melted $B_2O_3$ layer. Single crystals of 600÷1200g weight and 2" in diameter have been obtained.

The properties of the crystals were measured by the Van der Pauw method, sheet resistance method, Fe concentration by NIR absorption method.

Undoped InP crystals with $5 \cdot 10^{12} \div 8 \cdot 10^{15} cm^3$ carrier concentration and compensation ratio 0.45 ÷ 0.95 have been annealed at 900 ÷ 950 °C for 8 ÷ 78 h under phosphorus pressure 1÷15 atm.

Crystals with carrier concentration $n < 3 \cdot 10^{15} cm^{-3}$ after annealing became semi-insulating with resistivity $\geq 10^7$ Ωcm. In these crystals Fe atoms are not observed by

IR absorption method and EPR. This indicates the exsitence of other factors causing semi-insulating behaviour of undoped InP crystals after annealing.

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## INFLUENCE OF CARBON DOPING ON SI-GaAs PHYSICAL PROPERTIES

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 $Si0_2$ crucible.

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

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 77 K, deep donor EL2 concentration by NIR absorption.

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

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

With $N_c = (4 \div 10) \cdot 10^{15}\,cm^{-3}$ most crystals have shown the mobility $\mu = (4000 \div 6000)\ cm^2/Vs$. 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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### APPLICATION OF A STOPBAND MULTISTRIP COUPLER IN A SAW RESONATOR FILTER

Theoretical and experimental results are presented for the amplitude frequency responses of a SAW two-pole resonator passband filter with a stopband multistrip coupler (SMSC). Two resonators are placed side-by-side and the SMSC is formed by connecting open aluminium strips of reflectors. Compared with the ordinary MSC and coupling transducers, application of SMSC, which is an integral part of reflectors, makes the resonators shorter. Therefore, the two nearest longitudinal modes are moved away and better rejection is obtained near the passband. The SMSC is especially useful in the case of quartz if relatively large inter cavity coupling is needed. The filter, fabricated on ST-cut quartz, had 600 strips in each reflector, and 200 of them were connected to form the SMSC. Other parameters were as follows: aperture - 1 mm, number of single electrodes of each apodized IDT - 61, aluminium layer thickness 0.12 μm, width of electrodes - 2.6 μm, center frequency of the passband - 303 MHz. The resonators were overcoupled and two peaks were seen in the transfer function of the filter (for the 50 Ω source and load impedances). The influence of the number of SMSC strips on the filter transfer function will be presented.

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## ZASTOSOWANIE METOD AAS I SSMS DO ANALIZY LIŚCI TYTONIU

W ramach prac koordynowanych przez Komitet Analityczny PAN, a związanych z atestowaniem materiałów biologicznych, wykonano analizę dwóch próbek liści tytoniu o symbolach CTA-OTL-1 i CTA-VTL-l. Wykorzystano 2 techniki analityczne: AAS i SSMS. Określono zawartość 26 pierwiastków z 36 wytypowanych przez organizatora. Otrzymane wyniki zostały przesłane do jednostki koordynującej.

Po przeprowadzeniu suchej i mokrej mineralizacji oznaczono metodą AAS następujące pierwiastki: K, Fe, Na, Mg, Sr, Cr, Mn, Ca, Cu, Ni. Analizę wykonano na spektrometrze AAS firmy Perkin Elmer model 430 stosując do oznaczanych pierwiastków odpowiednie lampy z katodą wnękową. Wzajemne inerferencje redukowano stosując do każdego oznaczenia metodę dodatku wzorca.

Metodą SSMS oznaczono następujące pierwiastki: Al, Cl, Co, P, Pb, Si, V, Zn, Be, B, S, Ti, Y, Zr, Nb, Ba. Odpowiednio spreparowane elektrody poddawano wzbudzeniu na spektrometrze JEOL 01-BM ze wzbudzeniem iskrowym. Do korekcji błędów analizy widm masowych stosowano współczynniki czułości - RSF.

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