

W.K. Wierzchowski

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

## **THE SIMULATION OF BRAGG-CASE DOUBLE-CRYSTAL IMAGES OF DISLOCATIONS IN GaAs CRYSTALS.**

The numerical integration of Takagi-Taupin equations was applied to simulation of back-reflection double-crystal topographic images of dislocations in GaAs crystals.

The numerical program calculates the gradient of deformation field in the isotropic approximation taking into account the stress relaxation at the free surface. The finite thickness of the crystal slab is also assumed. In the present case of the considerable absorption the last assumption practically does not affect the simulated images and it allows to increase the size of simulated image with lower cost in computation time. It also enables placing the dislocation line further from the boundaries of the integration area. Taking into account the presence of epitaxial layer was also possible.

In the present simulations the finite divergence of the beam forming the image in the double-crystal method was included. It was realized adding more than 60 images slightly differing in the assumed angle of incidence, weighted by appropriate rocking curve describing the angular distribution of radiation reflected by the monochromator.

The simulated images were confronted with the experimental images obtained in  $^{511}\text{Ge}$ ,  $^{511}\text{GaAs}$  double-crystal arrangement with  $\text{CuK}_{\alpha 1}$  radiation. The topographs in different equivalent reflections were taken and the preliminary identification was based on Lang transmission topographs.

A reasonable agreement of simulated and experimental images was obtained, especially when a finite divergence of the beam were taken into account. In that case the interference fringes appearing close to the dislocation core in the plane-wave images were averaged.

The important feature of the simulated images was the presence of characteristic black-white rosette. We proved a rough correspondence of it

to the direct dilatation-orientation contrast coming from the displacement field. The contrast of the rosette was reversible with the change of the Burgers vector sign and with the change of the rocking curve slope. The extinction rules in the reflection from equivalent crystallographic planes were significantly affected by surface stress relaxation phenomena.

Comparative simulations of dislocation images the crystals with lower absorption and smaller structure factors, as silicon and diamond, exhibited higher contribution of different interference fringes on the cost of the "rosette".

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K. Wieteska<sup>1)</sup>, W.K. Wierzchowski<sup>2)</sup>, J. Maurin<sup>1)</sup>

1) Institute of Atomic Energy  
05-400 Świerk-Otwock, Poland

2) Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

## **THE BRAGG-CASE DIFFRACTION PATTERNS OF ION-IMPLANTED LAYERS**

4-5 MeV alpha-particles implanted layer in silicon were studied using double-crystal method. In actual case most of the implanted ions is expected to be concentrated in a thin layer situated at the depth of several microns under the surface. The rocking curves of the layers exhibited distinct subsidiary maxima and the topographs contained systems of fringes.

In order to explain the results we discussed theoretical diffraction patterns obtained both with plane-wave approximation and by numerical integration of Takagi equations. In the last case we took into account the continuous distribution of the ions and related strains. In both cases we studied the influence of different phase shift introduced by the destroyed layer.

On the base of theoretical models we proved that the fringe systems observed in the topographs are due to the variation of the thickness of the destroyed layer, while the formation of the subsidiary maxima in the curves is connected with the thickness of the shot-through layer. The destroyed layer introduce additional phase factor multiplying the whole system of subsidiary maxima with reciprocal phases on the two sides of the rocking curve.

The important feature of the experimental rocking curves consisting in the location of the most maxima on low angle side and the increase of their period with lower angles was obtained assuming the strain gradient in the shot-through layer. It may be expected that the strain gradient causes the curvature of the trajectories and subsequent diminishing of the subsidiary maxima period. This effect should be stronger close to the main peak.

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W. Włosiński, W. Olesińska, K. Pietrzak

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **BONDING OF ALUMINA TO STEEL USING COPPER INTERLAYER**

The present methods of bonding alumina to steel do not allow for receiving joints with large dimensions that could withstand good mechanical properties. Very often microcracks and discontinuities occur in such joints. Another problem, in the case of using active braze metals, can be formation of microdefects in ceramics, resulting from the reactions between ceramics, active elements of the braze and steel components. The experiments carried out show that multilayer bonding using thin and very plastic material interlayer is a good solution of the problem of fabricating alumina - steel joints with large dimensions. This interlayer allows for relaxation of stresses created during bonding cycle. It was concluded that copper material interlayer is suitable for this purpose.

Direct bonding using active products of reduction reaction of  $\text{CuO}$  ( $\text{Cu}_2\text{O}$ ,  $\text{O}_2$ ) can be the adequate process of bonding joints with copper interlayer. Using this method, we can partly to render the relation between the quality of the bond (homogenous microstructure at the whole bonding surface) and a protective atmosphere.

This paper contains the conditions of alumina - steel joining process and the results of strength tests. Another part of this work are microstructure tests, showing the homogeneity of bonds. The elaborated method can be used for joining alumina to copper and copper to steel in one thermal cycle. Such bonded joints have high mechanical strength and homogenous microstructure. Comparing to other joining methods this process does not need to use any special joining conditions (low bonding temperature  $\approx 1340\text{K}$ , protective atmosphere of  $\text{N}_2$  containing about 40 ppm  $\text{O}_2$ , bonding time  $\approx 90$  minutes).

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K. Pietrzak

Institute Electronic Materials Technology  
ul. Wólczyńska 133, O1-919 Warszawa, Poland

## **JOINING OF CARBON FIBRE - COPPER COMPOSITE TO METALS**

Fibre reinforced composites belongs to the group of composites with programmable properties, which are meant to be the most attractive kind of advanced materials. Carbon fibre - copper composites are used f.e. for electric contacts, dilatation bases for power semiconductive elements and also for brazes with enhanced mechanical strength.

The important problem is joining of this composite to metal technical alloys. It can find the application in diffusion bonding, vacuum brazing or in protective atmosphere brazing.

This paper contains the results of investigations on bonding carbon fibre - copper composite to low carbon steel or to tungsten.

The carbon fibre - copper composite was formed by volume bonding using the following conditions: bonding temperature 850°C, bonding time 30 minutes, pressure 300kG, vacuum  $2 \times 10^{-5}$  Tr. The composite contained 30% vol. of carbon fibres and copper was admixed with active elements (Zr, Cr). The process of diffusion bonding was carried on with bonding parameters varying in a specified range: bonding temperature 750 - 850°C, bonding time 15 - 60 minutes, pressure 250 - 350 kG.

The presented paper includes the results of structural investigations (microstructure, linear elements distributions). It was concluded that the interface being formed between composite and steel or tungsten is in fact created between copper matrix and bonded metal.

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L. Kociszewski, D. Pysz, R. Stępień

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **APPLICATION POSSIBILITIES OF FIBER OPTIC IMAGE GUIDE RODS IN ULTRATHIN ENDOSCOPES**

Small diameter of an endoscope can be realized by using a fiber optic multirod, instead of the system of many sequently placed lenses. With fiber optic technique it is possible to manufacture rods with square and circular section, 1 mm and even below in diameter. Successful experimental manufacturing of such fiber optic image guides has been performed. Various technological problems (like image cleanness improvement, enhancing of relayed image contrast and resolution, choice of proper rod cladding and EMA glasses) were encountered.

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A. Kłos<sup>1)</sup>, A. Glubokov<sup>1)</sup>, A. Pajączkowska<sup>1,2)</sup>

1) Institute of Electronic Materials Technology,  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

2) Institute of Physics, Polish Academy of Science,  
Al. Lotników 32/46, 02-668 Warszawa, Poland

### **ON MELTING OF $YBa_2Cu_3O_{7-x}$ IN $Y_2O_3$ CRUCIBLE**

Since the discovery of superconductivity at 92 K for a  $YBa_2Cu_3O_{7-x}$  (with  $x = 0.1$ ), a great number of studies have been devoted to the elaboration of large single crystals which are necessary for physical measurements, applications and as substrate for liquid phase epitaxy to receive extremely perfect layers with quasi-atomically flat surfaces.

Recently, Japanese have found that large single crystals to 7mm in c-axis can be obtained by Czochralski method and become superconducting at 92K [1]. Crystals have been grown from yttrium oxide crucible and from nonstoichiometric solution.

Some groups at the Institute of Electronic Materials Technology have experience in crystal growth by the Czochralski method and liquid phase epitaxy (LPE) and the following investigations have been performed:

- crucible of  $Y_2O_3$  has been chosen and prepared,
- seeds and substrates of perovskite structure crystals have been grown,
- crystallization of  $YBa_2Cu_3O_{7-x}$  by Czochralski method has been investigated.

The following results are presented:

- DTA/TG measurements for different fluxes using  $Al_2O_3$  and  $Y_2O_3$  crucibles,
- the corrosion of  $Y_2O_3$  crucibles in function of time and the composition of flux,
- primary results on crystallization.

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P. Kamiński, R. Kozłowski, E. Nossarzewska-Orłowska, J. Sarnecki

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **DEEP-LEVEL DEFECTS IN EPITAXIAL SILICON FOR VLSITECHNOLOGY**

Application of epitaxial silicon to submicron VLSI involves very high requirements on the epitaxial layers quality. Therefore, as integration density increases, it becomes necessary to control the concentration and properties of electrically active deep-level defects which are considered to be a major factor limiting the performance of the integrated circuit devices. So far, however, these defects have rarely been studied in epitaxial silicon.

In this work a characterization of the silicon epitaxial layers using deep-level transient spectroscopy (DLTS) and recombination lifetime measurements is presented. Our investigations were made on 25-mm-thick epilayers of n and p type grown on  $n^+$  (antimony-doped) and  $p^+$  (boron-doped) substrates, respectively. For the first time both electron and hole traps induced in the epilayers by the Si substrate contaminated with palladium have been found. In addition, the DLTS spectra for epitaxial layers deposited by using silicon tetrachloride from different vendors have been compared. The profiles of the traps concentrations in the epilayers have also been studied. It has been demonstrated that DLTS technique is a powerful tool for monitoring deep-level defects formed in the epitaxial layers due to either the Si substrate or the silicon source contamination.

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W.K. Włosiński<sup>1)</sup>, W.Olesińska<sup>2)</sup>

- 1) Warsaw Technical University,  
ul. Narbutta 85, 02-524 Warszawa, Poland
- 2) Institute of Electronic Materials Technology,  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **AN INFLUENCE OF TITANIUM ON ACTIVITY AND PENETRATION OF AgCuTiIn SOLDER COMPONENTS INTO ALUMINA CERAMICS**

Study on technological and microstructural conditions for brazing of alumina ceramics and FeNi42 alloy, by means of filler alloys contain active elements, are presented. Diffusion of titanium into alumina ceramics within the area of intermediate layer between ceramics and Ag72,5Cu19,5Ti3In5 solder, was specially investigated. Effect of titanium on thermal stability of ceramic-to-metal joint was experimentally verified. Vacuum tight ceramic-to-metal joint having a high mechanical strength was produced as a result of this study.

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M. J. Buda

Institute Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland.

**CORRECTION BY TEMPERATURE GRADIENT OF DIFFUSED  
FLOWS ACROSS CERAMICS-METAL BOUNDARY**

Diffusion processes were studied across the alumina-metal boundary. The influence of direction and value of temperature gradient on intensity of the system component fluxes were analysed.

Comparison was carried out between isothermal diffusion processes and the results due to thermodiffusion processes. Diffusion and thermodiffusion parameters have been determined.

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H. Tomaszewski

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

**EFFECT OF OXYGEN CONTENT IN SINTERING ATMOSPHERE  
ON T-C INVERSION OF UNSTABILIZED ZIRCONIA GRAINS DI-  
SPERSED IN ALUMINA MATRIX**

The effect of sintering atmosphere on the content of zirconia phases present in  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  ceramics was studied. As it was demonstrated, in the case of high vacuum-sintered ceramics 100% of zirconia is found in the cubic form, in contrast with 83.5% of tetragonal form observed in the air-sintered one. Appearance of transformable cubic zirconia is a result of sintering in atmosphere with very low oxygen partial pressure and accompanied oxygen nonstoichiometry.

By changing of oxygen partial pressure, a critical value of oxygen content in a sintering atmosphere responsible for "low temperature" tetragonal-cubic inversion of unstabilized zirconia inclusions dispersed in alumina matrix was estimated.

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M. Moroz<sup>1)</sup>, S. Achmatowicz<sup>2)</sup>

1) Joint Stock Company "ELVA"

Gabijos g-ve, 5-13, 2022 Vilnius, Lithuania

2) Institute of Electronic Materials Technology

ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **THE INFLUENCE OF RuO<sub>2</sub> POWDERS PREPARATION METHOD ON THE PARAMETERS OF FIRED RESISTORS**

A new method of preparation of RuO<sub>2</sub> powder is presented. It has been shown that the manner of RuO<sub>2</sub> powder preparation has got a great influence on the powder parameters such as specific surface, dimension of particles, shape of particles, etc. The influence of different properties of thus obtained RuO<sub>2</sub> powders on thick film resistors has been investigated.

The manner of RuO<sub>2</sub> powders manufacturing is crucial for resistive paste composition (RuO<sub>2</sub> content) and responsible for resistors properties: resistivity, TCR, resistivity's dependence on temperature and duration of firing, repeated firings, stability of resistance under applied voltage.

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M. Primowicz<sup>1)</sup>, M. Jakubowska<sup>2)</sup>, E. Zwierkowska<sup>2)</sup>, S. Achmatowicz<sup>2)</sup>

1) Scientific Production Association "CARAT"

ul. Stryjska 202, 290031 Lvov, Ukraina

2) Institute of Electronic Materials Technology

ul. Wólczyńska 133, 01-919 Warszawa, Poland

## **BASE METAL RESISTIVE PASTES FIRED IN AIR**

A new series of base metal resistive pastes fired in air has been investigated. The obtained sheet resistivity is in the range of 20 ohm / to 100 kohm/

The resistive layers exhibit TCR + 300 ppp/°C. The pastes are fired at 650-750°C and can be applied to a broad range of substrates such as alumina, porcelainised or glaze metal and glass.

The basic parameters of the layers are presented. The influence of resistor contacts and firing temperature on the layers properties has been described.

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M. Moroz<sup>1)</sup>, S. Achmatowicz<sup>2)</sup>

1) Joint Stock Company "ELVA"

Gabijos g-ve, 5-13, 2022 Vilnius, Lithuania

2) Institute of Electronic Materials Technology

ul. Wólczyńska 133, 01-919 Warszawa, Poland

## **RESISTIVE PASTES FOR HIGH-OHM VALUE RESISTORS**

A new relationship between resistivity/TCR of Ru-based resistors and the content and origin of the conductive phase of the resistive composition has been observed. This led to developing of a new series of high-ohm resistive pastes. Thus, certain difficulties such as rapid changes of sheet resistivity with small changes of conductive phase content, high sensitivity of electrical properties to firing conditions and resistors parameters slump under applied voltage, have been overcome.

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S. Osieczkin<sup>1)</sup>, M. Jakubowska<sup>2)</sup>, E. Zwierkowska<sup>2)</sup>,  
S. Achmatowicz<sup>2)</sup>

1) Scientific Production Association "CARAT"  
ul. Stryjska 202, 290031 Lvov, Ukraina

2) Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **SILVER POWDER FOR ELECTRONIC PASTES**

A novel method of precipitation of silver powder from aqueous solution of silver nitrate is presented. A broad range of different silver powder can be obtained, to find appliance in many kinds of silver, silver-palladium, silver-platinum pastes. These powders are suitable for polymer thick film pastes as well as for adhesives. Some examples of application of the powders to produce silver pastes and properties of the layers obtained with use of the latter have been also described.

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Z. Frukacz<sup>1)</sup>, J. Kisielewski<sup>1)</sup>, Z. Mierczyk<sup>2)</sup>, W. Zendzian<sup>2)</sup>

1) Institute of Electronic Materials Technology  
ul. Wólczyńska 133, O1-919 Warszawa, Poland

2) Military University of Technology,  
ul. Kaliskiego 1, 00-908 Warszawa, Poland

### **CRYSTAL GROWTH, OPTICAL AND LASER PROPERTIES OF Cr, Tm, Ho:YAG MATERIALS**

Yttrium aluminum garnets doped by Cr, Tm, and Ho have been obtained by Czochralski method using iridium crucible and HF heating. An assumed concentrations of dopants are  $n(\text{Cr})=2.3 \times 10^{20} \text{cm}^{-3}$ ,  $n(\text{Tm})=7.9 \times 10^{20} \text{cm}^{-3}$  and  $n(\text{Ho})=5 \times 10^{19} \text{cm}^{-3}$ , respectively. Moreover, the distribution coefficients for chromium (Cr), thulium (Tm) and holmium (Ho) are 2.4, 1.2, and 1, respectively. Thus, the concentrations of Cr and Tm dopants change along the crystal. Monocrystals having 28% of the initial mass of the crucible ingot and 75 mm long have been obtained. The concentrations of dopants calculated with the help of the normal freeze equation at the end of the crystal are  $n(\text{Cr})=1.45 \times 10^{20} \text{cm}^{-3}$ ,  $n(\text{Tm})=7.4 \times 10^{20} \text{cm}^{-3}$ , respectively. The absorptive, luminescence as well as energetic properties of laser rods, having 4 mm diameter and the length equals to 63-67 mm, have been investigated. It has been shown that the energetic characteristics of the obtained crystals depend strongly on the annealing in the oxidizing atmosphere.

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M. Malinowski<sup>1)</sup>, W. Woliński<sup>1)</sup>, Z. Frukacz<sup>2)</sup>

1) Instytut Mikroelektroniki i Optoelektroniki PW,  
Warszawa, Poland

2) Instytut Technologii Materiałów Elektronicznych,  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### EMISJA W ZAKRESIE WIDZIALNYM I ULTRAFIOLETU W KRYSZTAŁACH YAG:Tm<sup>3+</sup>

Układ poziomów energetycznych trójwartościowego jonu tulu (Tm<sup>3+</sup>) zawiera szereg poziomów metastabilnych <sup>1</sup>I<sub>6</sub>, <sup>1</sup>D<sub>2</sub>, <sup>1</sup>G<sub>4</sub>, <sup>3</sup>H<sub>4</sub> oraz <sup>3</sup>F<sub>4</sub> umożliwiających równoczesną emisję promieniowania na szeregu długościach fal z zakresu ultrafioletu (UV), widzialnego i bliskiej podczerwieni [1]. Kryształy Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (YAG) domieszkowane Tm<sup>3+</sup> są znanym i sprawnym materiałem laserowym w paśmie 21 mm [2]. Ostatnio pokazano, że możliwe jest praktyczne wykorzystanie oddziaływań kooperatywnych i zjawisk wielofotonowych w celu skonstruowania laserów pracujących w krótkofalowym obszarze widma pobudzanych podczerwonymi laserami półprzewodnikowymi [3]. Lasery te generują promieniowanie o długości fali krótszej niż promieniowanie pobudzające. Generację promieniowania niebieskiego w wyniku konwersji wzbudzenia podczerwonego otrzymano w kryształach LiYF<sub>4</sub>:Tm<sup>3+</sup> [4], YAG-Tm<sub>3+</sub> [5] oraz szeregu aktywnych włókien światłowodowych domieszkowanych jonami Tm<sup>3+</sup> [6].

Nasze wcześniejsze badania wykazały wydajną konwersję promieniowania czerwonego (ok. 650 nm) na emisję w zakresie UV i niebieskim widma [7]. W niniejszej pracy, w celu lepszego poznania i zoptymalizowania tego procesu przeprowadzono pomiary emisji z wysokoenergetycznych stanów wzbudzonych <sup>1</sup>I<sub>6</sub>, <sup>1</sup>D<sub>2</sub> i <sup>1</sup>G<sub>4</sub> jonu Tm<sup>3+</sup> w serii kryształów YAG o różnych

koncentracjach domieszki- 0.1, 0.5 i 5 at. %  $Tm^{3+}$ . Przedstawiono widma emisji oraz wyniki badań kinetyki fluorescencji w zakresie od 290 do 800 nm otrzymanych w wyniku pobudzenia bezpośredniego promieniowaniem o długości fali 290 nm oraz w wyniku konwersji wzbudzenia promieniowania o długości fali 650 nm. Przedstawiono wnioski dotyczące roli przejść niepromienistych i oddziaływania międzyjonowego w wygaszaniu emisji wysokoenergetycznych stanów wzbudzonych jonu  $Tm^{3+}$  w kryształach YAG.

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W. Ryba-Romanowski<sup>1)</sup>, S. Gołąb<sup>1)</sup>, G. Dominiak-Dzik<sup>1)</sup>,  
M. Berkowski<sup>2)</sup>, A. Pajczkowska<sup>3)</sup>

1) Instytut Niskich Temperatur i Badań Strukturalnych PAN, Wrocław

2) Instytut Fizyki PAN, Warszawa

3) Instytut Technologii Materiałów Elektronicznych, Warszawa

### **SrLaAlO<sub>4</sub>:Nd - NOWE, NIEUPORZĄDKOWANE KRYSZTAŁY LASEROWE**

Wykonano badania optyczne i strukturalne monokryształów SrLaAlO<sub>4</sub>, aktywowanych neodymem, w celu określenia ich przydatności do budowy laserów miniaturowych. SrLaAlO<sub>4</sub> należy do szerokiej rodziny związków o wzorze ogólnym ABCO<sub>4</sub>, gdzie A = Ca, Sr; B = La - Gd i C = Ga lub Al tworzącej kryształy tetragonalne klasyfikowane w grupie przestrzennej I4/mmm.

Stwierdzono, że struktura kryształu zbudowana jest z warstw AlO<sub>6</sub>, a między warstwami, kationy Sr<sup>2+</sup> i La<sup>3+</sup> obsadzają statystycznie węzły o symetrii C<sub>4v</sub>. Wynikający stąd nieporządek strukturalny powoduje silnie niejednorodne poszerzenie pasm absorpcji i emisji neodymu, który podstawia lantan w sieci krystalicznej. Charakterystyczną cechą monokryształów SrLaAlO<sub>4</sub> jest duża intensywność pasm odpowiadających przejściom wewnątrz konfiguracji 4f<sup>3</sup> neodymu. W szczególności, siła oscylatora przejścia absorpcyjnego <sup>4</sup>I<sub>9/2</sub> - <sup>2</sup>H<sub>9/2</sub>, <sup>4</sup>F<sub>5/2</sub>, przy około 805 nm jest w przybliżeniu dwukrotnie większa niż w przypadku Nd:YAG. Pompując laserem półprzewodnikowym można zatem osiągnąć wysokie wydajności pompowania przy małych długościach ośrodka czynnego.

Osiągalne stężenie neodymu w monokryształach SrLaAlO<sub>4</sub> jest względnie duże, jednak prawdopodobieństwo stężeniowego wygaszania luminescencji rośnie szybko ze wzrostem stężenia neodymu. Wydajność kwantowa luminescencji bliska jedności dla stężenia 1 %, at, spada do około 0,7 dla stężenia 5% at. Potencjalną wadą kryształów jest duży współczynnik załamania światła, sugerujący wysokie wartości składowej nieliniowej, która sprzyja zjawisku samoogniskowania przy dużych gęstościach mocy promieniowania. Zagadnienie to wymaga weryfikacji.

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G. Dominiak-Dzik<sup>1)</sup>, S. Gołąb<sup>1)</sup>, I. Pracka<sup>2)</sup>, W. Ryba - Romanowski<sup>1)</sup>

1) Instytut Niskich Temperatur i Badań Strukturalnych PAN,  
Wrocław, Poland

2) Instytut Technologii Materiałów Elektronicznych,  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

### **MECHANIZM OBSADZANIA I RELAKSACJI STANÓW METASTABILNYCH JONU $\text{Er}^{3+}$ W $\text{LiNbO}_3$**

Pierwotnie przewidywano, że dzięki swym własnościom nieliniowym monokryształy niobanu litu aktywowane lantanowcami umożliwią równoczesną akcję laserową, modulację dobroci wnęki i generację drugiej harmonicznej. Przewidywania te potwierdzono uzyskując drugą harmoniczną wytworzoną w monokryształach  $\text{LiNbO}_3:\text{Tm}$  w temperaturze ciekłego azotu i w monokryształach  $\text{LiNbO}_3:\text{Nd}$  w temperaturze pokojowej, jednak zjawisko fotorefrakcji ograniczało drastycznie wydajność lasera.

Dalsze badania wykazały, że niepożądane zjawiska można znacznie zmniejszyć przez domieszkowanie  $\text{LiNbO}_3$  odpowiednią ilością  $\text{MgO}$ , a rozwój laserów półprzewodnikowych pozwolił osiągnąć duże wydajności pompowania optycznego w zakresie bliskiej podczerwieni i wyeliminować szkodliwe promieniowanie krótkofalowe. Dzięki temu obserwuje się wzrost zainteresowania niobanem litu aktywowanym lantanowcami, w szczególności erbem ze względu na jego znaczenie w światłowodowych systemach telekomunikacyjnych.

W niniejszej pracy przedstawiono wyniki badań spektroskopowych monokryształów  $\text{LiNbO}_3:\text{Er}^{3+}$  wytworzonych metodą Czochralskiego. Obliczono prawdopodobieństwa przejść promienistych z poziomów wzbudzonych. Wyznaczono całkowite prawdopodobieństwa przejść, określono udział relaksacji wielofononowej i oddziaływania aktywator - aktywator w obsadzaniu i relaksacji stanów metastabilnych. Oszacowano wydajności kwantowe poziomów metastabilnych i wydajności potencjalnych przejść laserowych.

Anna Pajączkowska<sup>1,2)</sup>

1) Institute of Electronic Materials Technology,  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

2) Institute of Physics, Polish Academy of Sciences,  
Al. Lotników 32/46, 02-668 Warszawa, Poland

### MOLECULES IN CRYSTAL GROWTH

Molecules, complexes, clusters, aggregates are considered in the crystallization of polycomponent systems. Crystals are grown from solutions, in flux, from melt and from gaseous phase by spontaneous nucleation and on crystallographic oriented seeds.

Clusters are created mainly in polycomponent system and the induction time, supercooling, impurities, dopants, properties of crystallization medium as: viscosity, formation energy of complexes, ionic radii, concentration and composition of solution and crystal structure influence on nuclei formation and crystal growth.

The viscosity increases sharply near the crystallizing temperature suggesting some sort of association in more concentrated fluxed melts [1].

Complexes play an important role in crystallization by the chemical vapor transport. Mass transport have to be near equilibrium condition and partial pressure of chemical particles should be in similar range [2].

Crystal growth of congruent melted polycomponent system is governed by the transport (diffusion, convection) of dissociated elements, molecules or their aggregates to crystal/melt interface [3].

Investigation of flux, melt and vapor phase properties by neutron diffraction and Raman techniques should explain most questions.

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K. Pietrzak

Institute of Electronic Materials Technology  
ul. Wólczyńska 133, 01-919 Warszawa, Poland

## **THE INTERFACE BETWEEN CARBON FIBRE-COPPER COMPOSITES AS A RESULTS OF ACTIVE ELEMENTS ADDED**

The experiments were conducted to develop the technology of carbon fibre and copper - base composite. High thermal conduction and bond formability characterizes this kind of composites, and that is the reason why they might be useful as basic materials for semiconductors ( diodes, thyristors) and electrical contacts.

The main problem in the formation of this kind of composites is lack of carbon fibres wettability by copper, which makes impossible the formation of the composite of continuous structure, free from such defects as: pinholes, discontinuity etc. Therefore the influence of the addition of such elements as Zr and Cr in case to improve the composite wettability, was examined.

The experiments were conducted with the use of the additional elements content of 0.1 to 18% by weight, at the temperature between 1023 - 1323K in vacuum. The good results were reached while using Zr and Cr. Lack of wettability of carbon fibres by copper may be changed by covering fibres with relative metals or by admixing copper with active elements (carbides - forming).

Examined active elements demonstrate the ability to form carbides ( $ZrC$ ,  $ZrC_{0.7}$ ,  $C_2Cr_3$ ) on the interface. In fact, these carbides form the microstructure of the interlayer. In the light of the above mentioned statements carbon fibre-copper composite with the addition of Zr and Cr, was developed. Continuous structure and good physical features characterizes this kind of composite.

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