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MATERIALS ENGINEERING FOR OPTOELECTRONIC CRYSTALS RELATED TO III–V COMPOUNDS

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Abstract

An introductory glimpse of modern approaches to the application of productive and reasonable techniques for the defined electronic materials will be presented. The paper is divided into four parts. Each is supplemented with illustrations. The first part explains the meaning of materials engineering for electronics (*MEE*) and for optoelectronic crystals in particular. Its interdisciplinarity is shown and also the range of problems it can solve. Graduate courses of some *MEE* disciplines are also given.

The second part of the paper related to the feasible solution with *MEE* as to the optimal realization of the application requirements. The physical modelling, databases and characterization techniques are given. The third part deals with particular materials: III–V semiconductors. A brief survey of the best methods of crystal growth is given, stressing those which imply a possibility of creating crystals defined up to the atomic range.

The last part is devoted to our team's original crystal growth methods: *CAM-S* (A Crystallization Method Providing Composition Autocontrol in Situ) and *COM-S* (Calculation Method of Optimal Molten-Solution Composition). The combination of these methods, further modified with vibrational and magneto-hydrodinamical stirring (*VS, MHD-S*), allows us to grow crystalline ingots of ternary solid solutions (*TSS*) possessing extreme homogeneity. Illustrations on In–Ga–Sb system are supplied. We conclude with a discussion of the impact of such methods and approaches on a device quality and to other fields.

Keywords: data handling and computation, educational aids, materials science/engineering, methods of crystal growth, optical crystals, semiconductor materials-III-V, solid solutions

Introduction

This article is based on an unpublished lecture presented at 'CHISA '93' – 11th International Congress of Chemical Eng., Chem. Equipment Design and Automation (Aug. 29–Sept. 3, 1993, PRAHA) [1] and on a brief contribution to the 'CTU-Seminar '94' (Jan. 17–20, 1994): 'Materials Engineering for Electronics (MEE)' [1].

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The limiting and thus strategic significance of crystals – defined materials – for industry advancement, in general and for electronic industry in particular, is known both domestically and abroad [2, 3].

Their significance consists not in a bulk form only, but also in the form of single-crystalline substrates for the production of semiconducting heterostructures convenient for the realization of photoelectronic components (radiation sources, detectors, modulators, etc.) [4–6].

Trends in this field lie namely:

– in an achieving the maximum definiteness and reproducibility of properties of the crystals being prepared/produced,

– in a necessity to obtain a high degree of structural perfection (most of crystal properties depend on it),

– in a technological control with a possibility to gain the new properties for an application,

– in a preparation/growth of new or modified materials.

The quoted aims should be reached mainly by the new approaches to the crystal growth in conjunction with the diagnostics, modelling and adequate databases. The view on an atomic scale is desirable [7–9]. One of this sophisticated approaches is materials engineering for electronics (*MEE*) [10, 11].

The role of *MEE*

In principle, we wish the optimal satisfaction for the application requirements by a material possessing a quality which will correspond to the latest techniques. This can be explained schematically in Fig. 1 for the case how to satisfy application requirements in the preparation/growth of a semiconductor crystalline material in a defined quality [10, 11].

It is necessary for this task, to have available a maximum of adequate and reliable data. The graphical representation in Fig. 1 deals with three integral systems: 'semiconducting materials'; 'bank of databases'; and 'crystal growth methods, processes and modelling'.

The bank of databases must be supplied with critical data only. Three information sub-systems should fill in this demand: two of which relate to an equilibrium/time-independent state, for input (technological) and for output (application) properties; and the third one, for kinetic/metastable states (both for input and output information).

This information can be expressed in the analytical form, in tables, and/or in the form of state diagrams (*SD*) [10, 11], i.e., simply, in a graphical representation of sets of equations

$$Z_i = Z_i(x, x', T, P), \tag{1}$$

where Z_i are dependent properties, x, x' concentrations, T temperature and P any other independent parameter, like pressure, electrical, magnetical field, etc.

For example, if $Z_i = T_{tr}$, or P_{tr}; where T_{tr}, P_{tr} are respective the phase transformation temperatures or pressures; one receive phase diagrams (*PD*) of *T-X-X'* or *P-X-X'* type, respectively [10, 11]. Analogically, Z_i can mean G (Gibbs energy), (mobility) and many other technological or application properties, too.

The above given forms far not exceed all information being necessary for qualified *MEE* approach. The *PD*-s for stoichiometry and defects control [12], the correlation of dislocation density with 'output' properties [12], the kinetic

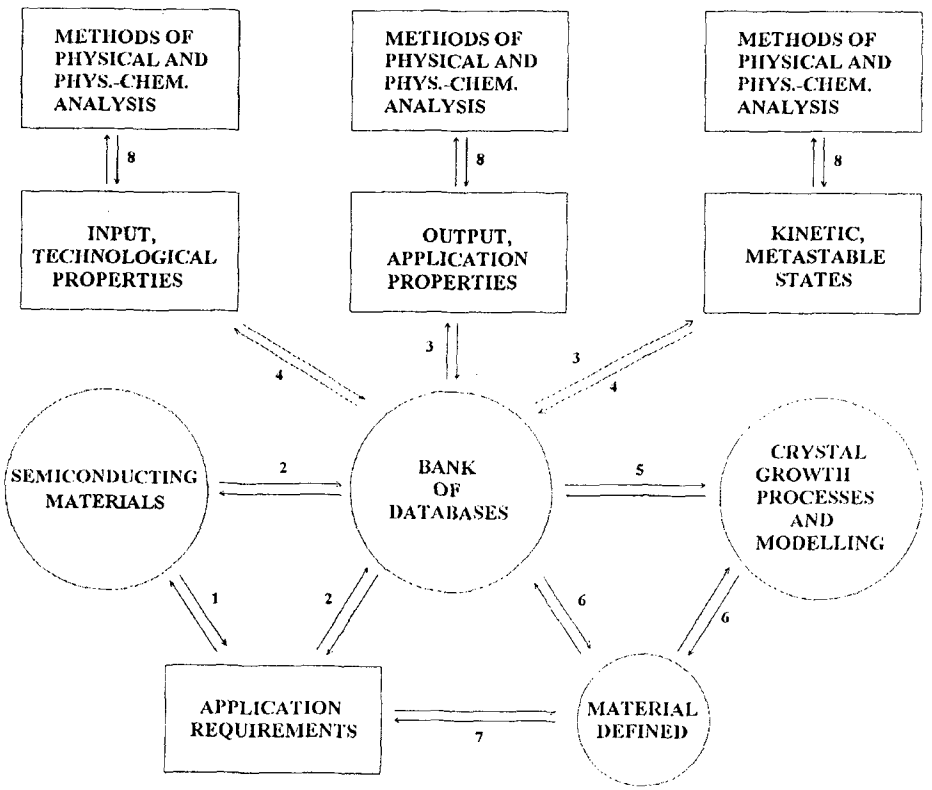

Fig. 1 A model demonstrating *MEE* approach. Paths from application requirements to their optimal realization marked: (1) choice of a group of materials, approximately in the first cycle; (2) optimization of this choice; (3) determination of material composition based on output (application) properties; (4) corrections based on technological properties; (5) selection of the growth method, process modelling; (6) defined material growth; (7) final optimization for the meeting application requirements; (8) current supply of the critical data

PD-s [13, 14], an atomic scale point of view [7], modelling on the same principle [8] are to be incorporated in these databases. The databases must also contain other information which can be logical included into the *MEE*. Special, comprehensive and topical monographies are valuables as well; see [14, 15] for example.

Each of the above mentioned information subsystems will be supplied with the reliable and verified experimental data by using the corresponding methods of physical and physico-chemical analysis [10, 11].

The paths marked by arrows correspond to the *MEE* treatment.

Cyclic repeating of this procedure may improve and optimize the meeting of application requirements. This approach brings the chance to save time and expense, and can be evidently modified for other materials too.

The knowledge of the deeper insight into the relationships of structures to

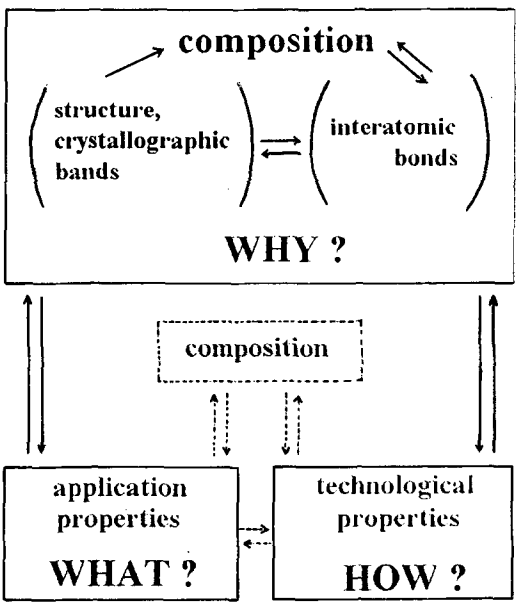

Fig. 2 'Structure – property' relationships. The empirical approach is marked by dashed lines (no detailed insight into the composition is used). Solid lines mark the rational one

properties also improves the *MEE* approach. Two methods of solution of the problem of *HOW* to reach the desired application properties are compared in Fig. 2: the empirical (dashed lines), and the rational one (solid).

One can hardly prepare, e.g. injection lasers, effectively working on 3 quantum wells, or MESFET structures, using only the empirical way [4, 7, 12, 13].

Some universities have a graduate program in Materials Science and Engineering majoring in Electronic Materials [16]. Massachusetts Institute of Technology, for example, includes similar courses. A brief selection of a few topics [MIT Bulletin, pg. 154] follows:'... the synthesis of fundamental and practical knowledge to develop, produce, modify, and apply materials to meet specific needs. ... Study of common principles and fundamental phenomena that underlie the structure and properties of a wide range of materials and computer modelling, to provide coherent description of materials structure ...'.

Our graduate courses of *MEE*, fit more or less with the MIT model, as shown bellow:

Methods and bases of materials engineering for electronics '*MB-MEE*'

Calculation of thermodynamical (*TD*) models of those phase diagrams being chosen for optoelectronics (*OE*); kinetic phase diagrams and metastable states; state diagrams (*SD*-s); smart/intelligent materials; databases for *MEE*; economic and managing peculiarities of *MEE*; interpretation and prediction of both the technological properties and utility of selected crystals; computing and model construction of crystallographic structure; a combination of known techniques and theories: → New quality. → Invention.

Application of materials engineering for electronics '*A-MEE*'

Application requirements on notable, structurally defined materials; theory and experiments applied on the growth of structurally defined materials; application of the band and quantum engineering; related experience from abroad; optimum solution of the whole path leading to the *OE* device required is performed by the selection of materials, technologies, and diagnostics; the outputs of analyses and syntheses of information are given for the needs of related application fields; illustration examples.

R. & D. of new materials '*RD-NM*'

Jackson's microscopic theory for a modelling, and its application in *RD-NM*. The principles of other outstanding theories and techniques for the defined single crystal growth; the modelling and simulation processes of crystal growth; crystal growth theory and growth techniques; etching; ohmic contacts of '*M-S*' (metal–semiconductor) junctions; nonconventional crystal growth methods; modelling of junctions in noncrystallizing substances; properties modification of polymers; R. & D. of magnetically hard alloys; development of steel type 'ATMOFIX'; dispersive solder pastes and hard-spheres; economic and managing peculiarities of *NM*.

All three courses will be supplemented with excursions to research institutions and with guest lectures by specialists.

Feasible solution with *MEE*

It is recommended:
- to follow the above given approach presented in Figs 1, 2,
- to collect adequate critical data,
- to analyze and synthesize them prior to performing the experiments.

→ Head must be ahead of hands.

Modelling and simulation of growth processes may be here of a reasonable help [8, 9, 17].

The databases fitting optimally with *MEE* are not easily accessible, particularly if there is a demand on critical, i.e. verified data. INSPEC on disk, *EMIS* (electronic materials information service), the Gmelin database on STN seemed to be more suitable, up to now. Also, the system Internet 'Gopher' may well contribute to this demand [18]. The perfect solution of how to satisfy application requirements was presented by an unpublished contribution on ICCG-10 (Tenth International Conference on Crystal Growth). The authors used computer controlled diagnostics coupled with databases, and with a control of crystal growth process [19].

At any rate, to have reliable data available is extremely expensive, and therefore it calls for a mutual cooperation based on a valid partnership. Our country seems to have a chance to fill such conditions [18].

The small database 'INSYS' has been created. This attempt was based on R. Podmanicky's diploma thesis [20].

One of the ways which lead to the high crystal perfection is a carefully controlled solution growth. Provided the solution is localized, in a zone, there is a more chance to control the process and thus to get crystals of better quality [21–24].

These method comprise *THM* (Travelling heater method) [24–29] and also our combination *CAM-S/COM-S* (Crystallization Method Providing Composition Autocontrol in Situ/Calculation Method of Optimal Molten-Solution Composition) [21–23]. *THM* processes are also studied in orbital laboratories [28, 30].

The low growth rate (diffusion layer) limits the application of these methods to a broader range [26, 31]. Along with other reasons, this leads to studies of crystallization processes which take place in a vibrationally stirred (*VS*) melt and in a *VS* solution [32–34]. The *MHD* (magneto-hydro-dynamic) forces are also used for a *MHD-S* stirring [35–38].

Bulk semiconductor crystals

A very recent review related to the melt growth of large-diameter elemental and compound semiconductors has been presented [6]. One can also concluded from it, and from [14, 15] as well, that even a highly sophisticated technique using a stoichiometry melt growth cannot fully satisfy quality requirements of these crystals; e.g., of CdTe, (Cd.Zn)Te, (Cd.Mn)Te. These facts call for another approach, as MEE has given [39–42].

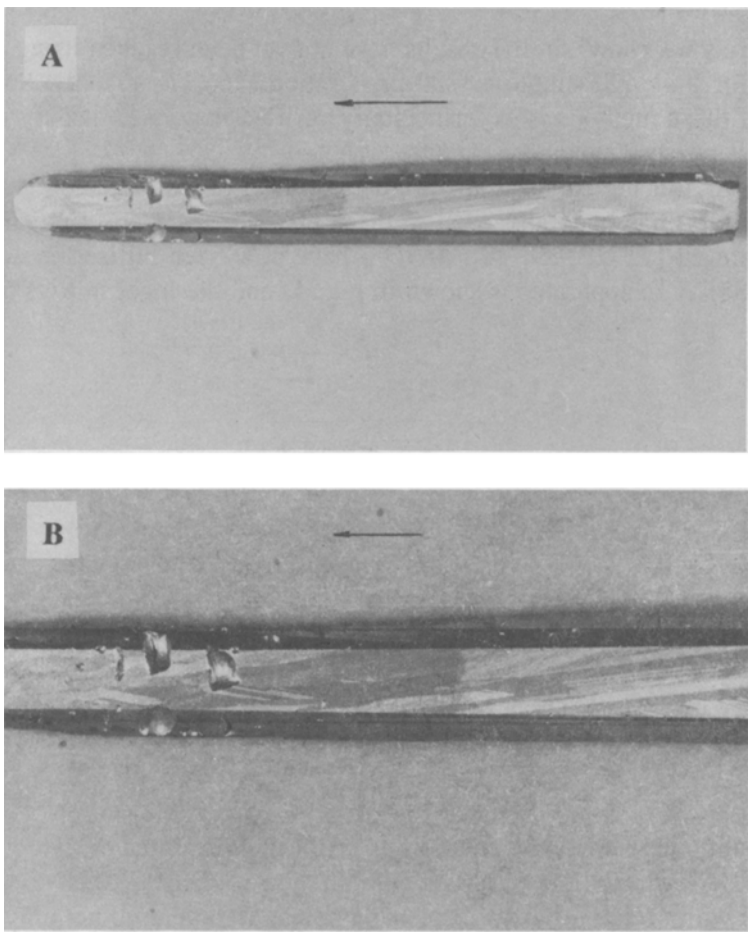

Fig. 3 Photograph of a *TSS* ingot grown with the *VS*. Parallel lamellas, being relatively long, are visible. – Scale: 2:1; arrow marks the growth direction

Novel approaches based on *MEE*

We have applied methods based on our original theories on the growth of *TSS* (ternary solid solution) crystals of III–V semiconductors. This approach brought the following results [21–23]:

– Constant lattice parameter, measured along the distance of 75 mm of the *TSS* Ga–In–Sb ingot – deviation less then 0.03%

– Hall mobility of holes and their concentration at 300 K/78 K μ_p=0.49/1.8 m^2/Vs; $p=10^{22}/10^{21}$ m^{-3} – preliminary results.

The materials used relate to *OE* [39–41]. A typical crystalline ingot is in Fig. 3.

As far as we know, similar results have not yet been reached using any other method for bulk *TSS* single crystal preparation [26, 27, 43, 44]. So the efficiency of these methods is experimentally verified now, even if only modestly.

– Crystals possess a mosaic structure..

The *VS* during a crystallization at the growth rate of 1.5 mm/h (=0.42 μm/s) has been also used in these experiments, and promising results were achieved [21–23, 39, 40]. *MHD-S* have now been utilized in addition to *VS* [37, 38]. The apparatus is shown in Fig. 4, and the ingot in Figs 5a,b [42].

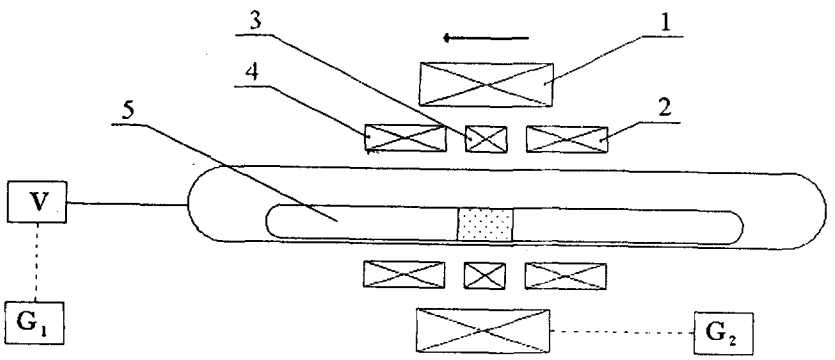

Fig. 4 The arrangement of the growth laboratory apparatus in a horizontal arrangement for both the *VS* and *MHD* stirring: 1 - induction coil, 2 - afterheater, 3 - zone heater, 4 - preheater, 5 - crystalline ingot, V - vibrator, G1,2 - pulse generators

As mentioned above, the solution growth brings advantages, which are corroborated elsewhere [24, 45]. The combination of *CAM-S*/*COM-S* with *VS* – modified with *MHD-S*, have a reasonable potential to be used not only for inorganic materials, but applied more widely [42], even to organic materials. Present theory does not exclude the use of the *VS* for crystal growth [8, 46] and experimental activity has supported it [32–34, 38–40].

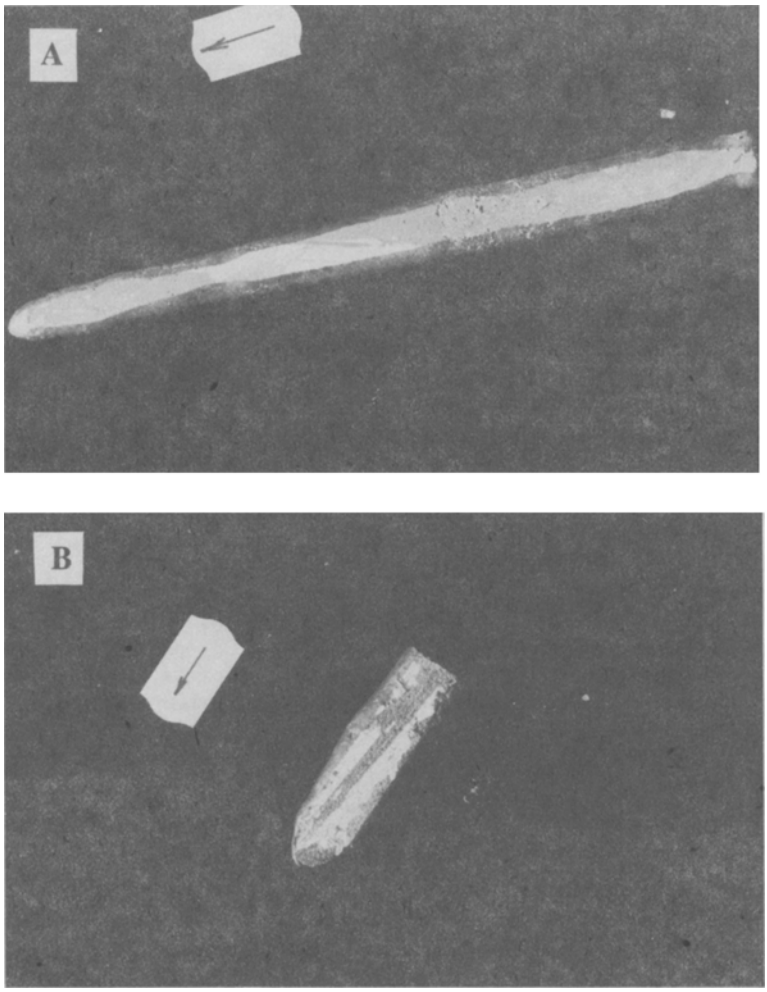

Fig. 5 Photographs of a recently grown ingot of GaSb [42] with a 105 mm length: a - the grain structure with lamellas, b - the detail of a trailing end; here, both the *VS* and *MHD* forces act. - Scale: 1,2 : 1; 2 : 1; arrows mark the growth direction

Conclusions

MEE supplemented by new approaches and novel methods, like *CAM-S/COM-S*, or in combination with *VS/MHD-S*, respectively, can meet higher application requirements. It can save time and bring higher efficiency. Moreover, it opens the possibility to bring new materials and therefore new applications. It also brings novel opportunities to other fields, which can be enriched by *MEE* and, in turn, enrich it.

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Zusammenfassung — Es wird ein kurzer Einblick in moderne Methoden zur Anwendung produktiver und akzeptabler Techniken für bestimmte elektronische Materialien gegeben. Der Artikel gliedert sich in vier Teile, wovon jeder mit Illustrationen ausgestattet ist. Der erste Teil erklärt die Bedeutung von Materialtechnik für Elektronik (*MEE*) und im speziellen für optoelektronische Kristalle. Es wird deren Interdisziplinarität gezeigt und auch der Fragenbereich, in dem sie angewendet werden kann. Es werden auch Hochschulkurse einiger *MEE* Disziplinen angeführt.

Der zweite Teil bezieht sich auf eine mögliche Lösung mittels *MEE* bezüglich der optimalen Realisierung der Anwendungsanforderungen. Physikalische Modellier-, Datenbanken- und Charakterisierungstechniken werden gegeben. Der dritte Teil beschäftigt sich mit speziellen Materialien: III-V Halbleiter. Es wird ein kurzer Überblick der besten Methoden für das Züchten von Kristallen gegeben, wobei jene besonders betont werden, die die Möglichkeit zur Schaffung von Kristallen bis zum atomaren Bereich beinhalten.

Der letzte Teil widmet sich den ursprünglichen Kristallzüchtungsmethoden unseres Forschungsteams: *CAM-S* und *COM-S*. Unter weiterer Modifizierung mittels *VS* und *MHD-S* erlaubt uns die Kombination dieser Methoden die Züchtung von Kristallrohblöcken von ternären festen Lösungen (*TSS*) mit einer extrem hohen Homogenität. Dafür werden Beispiele am System In-Ga-Sb gezeigt. Zum Schluß erfolgt eine Diskussion der Auswirkung solcher Methoden auf die Gerätegüte und andere Bereiche.