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MODEL BASED AUTODIAGNOSTICS OF SEMICONDUCTOR COMPOUNDS SYNTHESIS AND POLYCRYSTALLIZATION PROCESS

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Abstract – The process of synthesis and polycrystallization of InP is performed in hermetic welded quartz ampoule placed in the two zone furnace. The temperature of synthesis and crystallization zone is up to 1100°C. The pressure of phosphorus vapor inside the ampoule results from the temperature of low temperature zone which rises up to 570°C and can reach 3.0 MPa. Because of high pressure of the vapor inside the ampoule it should be compensated by pressure of neutral gas outside the ampoule and thus the whole furnace should be located in the autoclave. The measurements of temperature distribution and pressure of the reactor during multi-step process are in industrial conditions practical unfeasible. To avoid implosion or explosion of the ampoule the autodiagnostics is applied. The autodiagnostics makes use of the process reference model based on the indirect measurements of measurable state variables.

Keywords: autodiagnostics, indirect measurement, reference model, semiconductor compounds synthesis

1. INTRODUCTION

The semiconductor compounds such as indium phosphide and gallium arsenide are used for production of new generation semiconductor elements. Indium phosphide is used for production of very fast electronic elements, e.g. integrated circuits for data transmission reaching 80Gb/s [1]. Analysts predict for the next ten years demand for InP products of billion dollars value [2]. The great increase of demand for optoelectronic gallium arsenide elements is estimated as well [3].

The initial steps of production process of the mentioned compounds are synthesis of the liquid compound and its crystallization in the form of polycrystals by method of horizontal gradient freeze (HGF). The polycrystals are the material used for production of monocrystals in separate process. The monocrystals after cutting into wafers are material for semiconductor elements production.

In the paper the problem of autodiagnostics of the process of synthesis and polycrystalization is described using indium phosphoride (InP) processing example. The process is realized in horizontal tubular multi-section furnace placed inside the autoclave (Fig. 1). As process reactor the hermetic welded quartz ampoule is used. In low-temperature zone of the reactor is realized the controlled emission of phosphor vapor. For such emission in the phosphorus zone of the reactor should be changed temperature from 400°C up to 570°C. The phosphorus vapor migrates to high-temperature part of the reactor and reacts with fluid indium. The temperature of indium zone of the reactor is about 1100°C. The value of the pressure of phosphorus vapor depends exponential on temperature of the most cool place of the reactor due to condensation process. The inner pressure of the reactor required during the process is in 2÷3 MPa range (20÷30atm.).

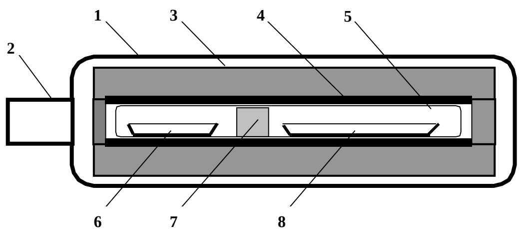

Fig. 1. The system for synthesis and polycrystallization of InP: 1 – autoclave, 2 – loading hutch,3 – thermal insulation, 4– multi-section heater, 5 – quartz ampoule, 6 –boat with phosphorus, 7 – convection barrier, 8 – boat with indium

The permissible difference between the pressure inside and outside of the ampoule is about 0.2 MPa and thus the pressure compensation by the neutral gas inside the autoclave should be applied. Because of technical reasons the direct temperature and pressure measurements inside of the ampoule during the normal production process is impossible. It should be emphasized that in case of insufficient pressure balance the explosion or implosion of the ampoule can occur and in consequence the destruction of the furnace. The necessary evacuation of the destroyed furnace out of the autoclave is difficult because of few kilograms of the phosphorus deposited on the furnace elements and on the walls of the autoclave. During such operation a fire, contamination of environment, poisoning and burn of the operators can occur.

The paper concerns the problem of the assurance of safety of the process of synthesis and polycrystallization of semiconductor compounds, especially InP, trough autodiagnostics. The described autadiagnostics makes use of the process model based on the indirect measurements. It is applied in the industrial version of the system for InP polycrystals production worked out by Industrial Institute of Electronics in Warsaw. The accompanying technology of the InP production is worked out by Institute of Electronic Materials Technology in Warsaw.

2. THE CONSTRUCTION AND TECHNOLOGICAL PROCESS

As it has been mentioned the construction of system for synthesis and polycrystallization of InP is shown in Fig. 1. The system consists of the autoclave and two zone tubular resistance heated furnace placed inside the autoclave. Temperature inside the furnace is shaped by sixteen heating sections. For the sake of aggressiveness of phosphorus (or arsenic vapor by GaAs compound) vapor the synthesis process is carried out in the hermetic welded quartz ampoules. The ampoule is placed inside the furnace (Fig. 1).

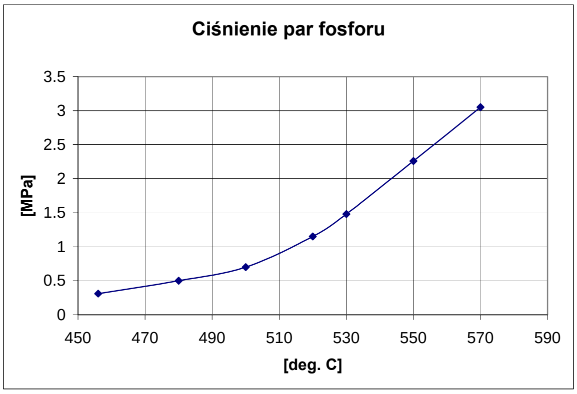

Fig. 2. Phosphorus vapor pressure vs. temperature

In the phosphorus zone of the quartz reactor is located the boat with phosphorus. In this zone during the process is applied the temperature of 400°C÷570°C range. Under the influence of temperature phosphorus sublimates and migrates to the indium zone where the boat with melted indium is placed. The value of the pressure of phosphorus vapor depends exponential on temperature of the most cool place of the reactor due to condensation process. The dependence between temperature and phosphorus vapors pressure is shown in Fig. 2. Because of strong non-linear dependence for obtaining the right pressure of the reactor the very precise temperature control in phosphorus zone is required. In Fig. 3 is shown the temperature distribution along the ampoule axis during synthesis (curve A), in some moment during HGF process (curve B) and at the end (curve C) of process. It can be observed that during the whole process the stability of temperature distribution in phosphorus zone is kept.

The initial temperature of 1100°C in indium zone of the reactor (curve A in Fig. 3) during the synthesis is stabilized. The final temperature of this zone after crystallization should by lower that crystallization temperature of 1070°C value (curve C). The polycrystal of InP is formed by horizontal gradient freeze (HGF) method. For directional crystallization process the programmed changes of heating sections should be applied. The curve (B) in Fig. 3 shows the intermediate temperature distribution during crystallization. The temperature gradient should shift smoothly with proper speed along the axis of the furnace. This can be obtained via multi-step temperature program applied for each heating section of indium zone.

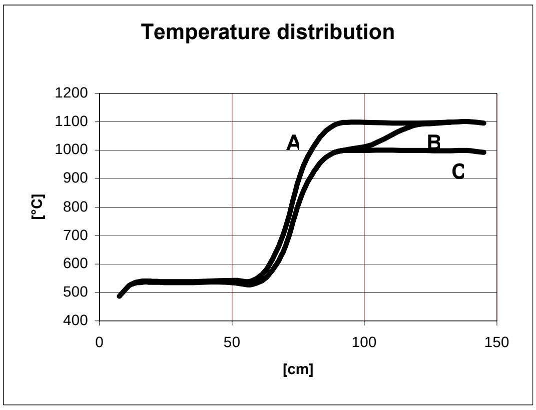

Fig. 3. Temperature distribution inside the ampoule: A – during the synthesis, B - in some moment during HGF process, C – at the end of the process

The construction of electroheat system and temperature control of the furnace for this purpose is much more complicated then in lot of electrothermal devices with horizontal tubular reactors. In described furnace dominates the heat exchange by convection. The gas properties by 2÷3 MPa pressure are similar to liquid properties and in consequence such details as thin gas barrier have very essential influence on temperature distribution. As a result of this phenomenon the temperature distribution in the reactor is essentially different from the temperature distribution in the close surrounding of the heater. The next complication results from the fact that the measurements of temperature distribution and pressure of the reactor are in industrial conditions practical unfeasible. This means that the reactor should be indirect controlled. The right inner temperature distribution and pressure value of the ampoule should be achieved using heater sections temperature control only.

For control program purpose the real temperature distribution and pressure inside the reactor during normal process should be measured. The results of these measurements are the basis for corrections of desirable temperature of the heating sections. The methods of these measurements, especially for measurements of pressure of phosphorus vapor, are complicated [4] and in consequence these measurements are very difficult and can be done in laboratory conditions only. Additional complication of these measurements is dynamic nature of the physical and chemical processes inside the ampoule. These problems should be the subject of separate paper and because of this reason they are not described in details here.

As a result of the mentioned measurements and corrections of the temperature distribution during the preparation of the process the identification of required temperature of heating sections is carried out. This enables the determination of the time program of temperature for the sections during the normal process combined with time program of the pressure in the autoclave.

Because of the difficulties connected with pressure control in dynamic conditions the procedure of increasing of the phosphorus pressure before synthesis and polycrystallization and also the procedure of pressure decreasing after the process are realized as multi-step sequences with stabilization phases. Taking additionally into consideration the necessity of multi-step HGF control process it can be stated that the whole process of synthesis and polycrystallization is carried out as the following main stages:

multi-step heating up with pressure stabilization for few intermediate levels – for each level the right temperature distribution in phosphorus sublimation zone should be achieved,

synthesis of InP that requires proper pressure of vapor and temperature distribution in synthesis zone,

multi-step temperature changes in crystallization zone for shaping the right value and speed of temperature gradient shift for horizontal freeze of InP crystals,

multi-step cooling down with pressure stabilization for few intermediate levels – for each level the right temperature distribution in phosphorus sublimation zone should be achieved.

3. THE AUTODIAGNOSTICS OF THE PROCESS

It has been mentioned before that during the preparations of the process the inner pressure and the temperature distribution is measured and adjusted for proper process realization. In the same time the identification of the process course referred to the other process variables is carried out too. These additional variables are the measurable state variables and for control and autodiagnostics purposes can replace the non-measurable variables describing the process inside the reactor.

It should be emphasized that this replacement is possible for stationary cases of the system only and any change of the thermal system parameters requires repetition of the identification. Such significant changes of the thermal system parameters can be caused by small crushing of the thermal insulation during loading and unloading of the ampoule, some displacement of thermocouples mounted in the furnace etc. It should be also taken into the consideration that the control loops of the system can keep the right temperature in the points of placement of the control sensor in spite of some changes of thermal system parameters. In consequence for enlargement of the process reliability the identification for autodiagnostics purposes should be based on the measurement of temperature of the heater sections and the reactor's neighborhood as well.

The set of described indirect variables courses together with course of desired pressure outside the ampoule can be used as the reference model for the diagnostics of the process. The permissible differences with relation to the reference model for each variable is determined during the identification also. For the control of the correctness of the process the variables measured during the actual process are comparing with their equivalents in the model. Any overflow of these borders causes warning or alarm informing the operator about irregularity. In such case the operator should decide if the process can be continued or it should be used the emergency program or manual procedure e.g. program of simultaneous furnace cooling and pressure reduction. In some dangerous states the progress of the program is automatically stopped and the operator is alarmed that the system waits for the human decision.

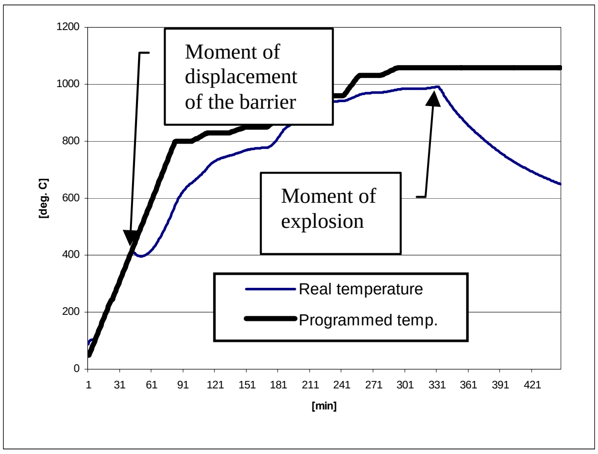

Fig. 4. Programmed and real temperature of section P02 during failure

The important consequence of the indirect control of the reactor's state variables is statement that any change of the heating system such as displacement of elements of the furnace or the reactor entails changes of the temperature and pressure conditions in the reactor. For example during the testing of the furnace the displacement of convection barrier (Fig. 1) inside the reactor toward the indium zone occurred. It changed the temperature distribution in such extent that pressure values of argon in the autoclave didn't meet the predicted levels. In Fig. 4 the programmed and real temperature of P02 heating section during this failure is shown. In normal state the convection barrier is located in the place heated mainly by this section surrounding the barrier. Because of the screening effect caused by the barrier in normal position the heat looses of this section are essentially lower than without barrier placement. After the mentioned displacement of the convection barrier the temperature of P02 section dropped below the programmed value (Fig. 4) although the section was supplied by full power. The next phenomenon connected with displacement of the convection barrier was extra heating power delivery to phosphorus zone. In the consequence in spite of some distance of section P02 from the phosphorus zone (Fig. 1) the temperature distribution in this zone changed. After some time since the displacement the inner pressure of the ampoule raised excessively, the quartz reactor exploded (Fig. 4) and the furnace become to be destroyed and contaminated by poisonous and combustible white phosphorus.

To ensure the security of the operation by avoiding the consequence of such situations it was necessary to equipped the described system for synthesis and polycrystallization of InP with appropriate autodiagnostic system. This autodiagnostic system is based on the measurements of following indirect variables:

- the temperature courses of all heating sections of the furnace,
- the average heating power values on outputs of all temperature control loops in each process step,
- the temperature courses of few additional points of the reactor's neighborhood,
- the pressure time changes of argon inside the autoclave.

4. CONCLUSION

During the works leading to right construction and control of the system for synthesis and polycrystallization of indium phosphide has been proved that the problem of safe temperature and pressure control inside the hermetic welded quartz ampoule can be solved by application of the autodiagnostics which makes use of the process reference model based on the indirect measurements.

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