Automatic Control System for the High Pressure CdTe Crystal Growth Furnace

Petr Praus, Eduard Belas, Jiří Bok, Roman Fesh, Jan Franc and Pavel Höschl

Charles University, Faculty of Mathematics and Physics
Ke Karlovu 5, Prague 2, 121 16, Czech Republic

ABSTRACT

CdTe and (CdZn)Te bulk single crystals have been widely used as substrates for MBE and LPE epitaxy of infrared (HgCd)Te as well as gamma- and X-ray detectors. The $Cd_{1-x}Zn_xTe$ ($x = 0.04–0.1$) single crystals with diameter up to 100 mm and height at most 40 mm were prepared in our laboratory in a vertical arrangement by gradual cooling of the melt (the Vertical Gradient Freezing method). Achievement of excellent crystal quality required full control of Cd pressure during the growth process and application of high Cd pressures (up to 4 bar) at growth temperature. An electronic control system was designed to control both temperature and internal pressure of two zones CZT crystal growth furnace by using two high performance PID controllers/setpoint programmers. Two wire current loop serial communication bus was used for the data exchange and computer control of the furnace electronics setup. Control software was written to supervise the crystal growth process and to collect all important data and parameters.

Keywords: CdTe, CdZnTe, crystal growth, Cd pressure control and PID cascade control

1. INTRODUCTION

The binary semiconductor CdTe has been in a center of intensive research since the 60^{th} mainly due to its application as a material for X and gamma ray detectors. At that time its basic physical properties were measured. After a period of certain attenuation the interest was renewed in the last decade mainly in connection with a development of new epitaxial methods (mainly molecular beam epitaxy MBE and liquid phase epitaxy LPE). It appeared, that CdTe with a small content of Zn (4–5%) is an ideal substrate for preparation of epitaxial layers of narrow-gap semiconductor (HgCd)Te, which has been widely used for a detection of infrared radiation. In the last years CdTe started to be used as a material for an electrooptic modulation of signal as well.

Fabrication of (CdZn)Te substrates is predominantly based on the growth from the melt by the Vertical Gradient Freeze method (VGF) [1,2]. In addition, the Vertical Bridgman method is also widely used [3,4]. It was found that the growth of (CdZn)Te has proven to be extremely difficult [5,6]. Due to the low thermal conductivity and low defect formation energy (i. e. stacking fault energy) [7] of CdTe, which is the lowest among compound semiconductors with the zinc-blend structure, it is very difficult to avoid the polycrystalline nature of solidification. Thus, the growth process does not usually result in a crystal with a single grain.

The crystallographic quality of the as grown crystals is a limiting factor of yield mainly in case of application of (CdZn)Te as a substrate material, when it is necessary to achieve a full width at half maximum of the rocking curves of X-ray radiation less than 50 arcs on the whole area of the substrate (typically 4×4 cm^2). The main factors influencing the process of crystal solidification are the distribution of temperature in the furnace characterized by its axial and radial gradients and the solidification speed. Our growth experiments also showed that the resulting quality of CdTe single crystal can be directly connected with the history of the melt (it means with the temperature to which the melt was heated above the melting point, with the solidification speed and with deviation from stoichiometry).

Our recently published results [8] have shown, that the growth of CdTe and CdZnTe crystals at higher Cd pressures than those typically applied in current growth experiments (1–2 bar) could lead to reduced supercooling at the crystal growth and consequently to higher crystallographic quality of boules and improvement of the yield and the substrate quality. The demonstrated effect was studied by direct DTA measurements in the high-pressure growth setup. The first results of crystal growth also clearly demonstrated, that no inclusions larger than 1 μm were observed in the boules in most cases. Therefore growth of CdZnTe single crystals at Cd overpressures in the range of 2–4 bar seems to be a promising way how to improve the current quality of substrates and increase the yield of material suitable for MBE epitaxy of HgCdTe.

2. CONTROL ELECTRONICS SETUP

An electronic control system has to be designed to control both temperature and internal pressure of two zones CZT crystal growth furnace. The schematic drawing of the control setup can be seen on Fig. 1. The furnace was built inside of the stainless steel water cooled, high pressure containment (possible pressures can reach up to 50 bar). The Cd, Te and Zn starting elements are placed inside of the evacuated silica glass ampoule. There are two independent heating elements to control the internal temperature gradient during the crystal growth process. Two high performance PID electronic controllers/setpoint programmers are used in the setup, one for the temperature and the latter for the containment internal pressure control. Both controllers enable free software wiring of all process variables and parameters.

The temperature controller is configured as two loop control system. Cascade control can be applied as an advanced control technique [9] to get the best temperature setpoint stability for one loop (see Fig. 1). This enables long time constant of the furnace to be controlled with the fastest possible response of the control system to the temperature profile perturbation caused by occuring exothermic processes and it also increases the heating elements lifetime substantially. Cascade control approach improves effectively the time lag between the temperature change of the heating element and the ampoule. This control electronics wiring enabled us to drive both heating elements of the furnace independently. However, one must be aware of influencing the temperature stability when the temperature gradient is changed rapidly. The temperature stability of both zones of the furnace is better than 0.01 °C. Phase angle firing thyristor units are driven by the controller output to regulate the heating power. Three Pt/PtRh10 inconel coated thermocouples are used to monitor the temperature inside of the containment very precisely. The main control thermocouples are close to the ampoule and the auxiliary one is close to the heating element. Extremely high sensitive differential thermocouple is also placed below of the ampoule to monitor starting of the crystallization process. This signal is collected by using high precision digital multimeter equipped by 8 channels analog scanner. The temperature value in the furnace upper zone above 800 °C causes substantial nonlinear increasing of the ampoule internal pressure above 1 bar originating from the Cd evaporation process. This overpressure must be compensated by appropriate filling of the stainless steel containment volume with an inert gas (Argon) to avoid the ampoule damaging during the crystal growth process. Argon pressure in the containment is controlled by the pressure controller which is configured in single loop control setup with 0 to ± 100% opening of the control output. It drives two proportional high-grade electromagnetic valves for the flow control of the containment filling by Argon in the interval from 1 to 5 bar. The positive output means pressured Argon input proportional valve opening while the negative output is opening the outlet proportional valve. This setup ensured, that only one valve could be opened during the pressure control. Supplementary passive flow control needle valves are used to limit the maximum flow of the gas. An absolute value pressure gauge sensor is mounted inside of the containment. Current loop (4–20 mA) is

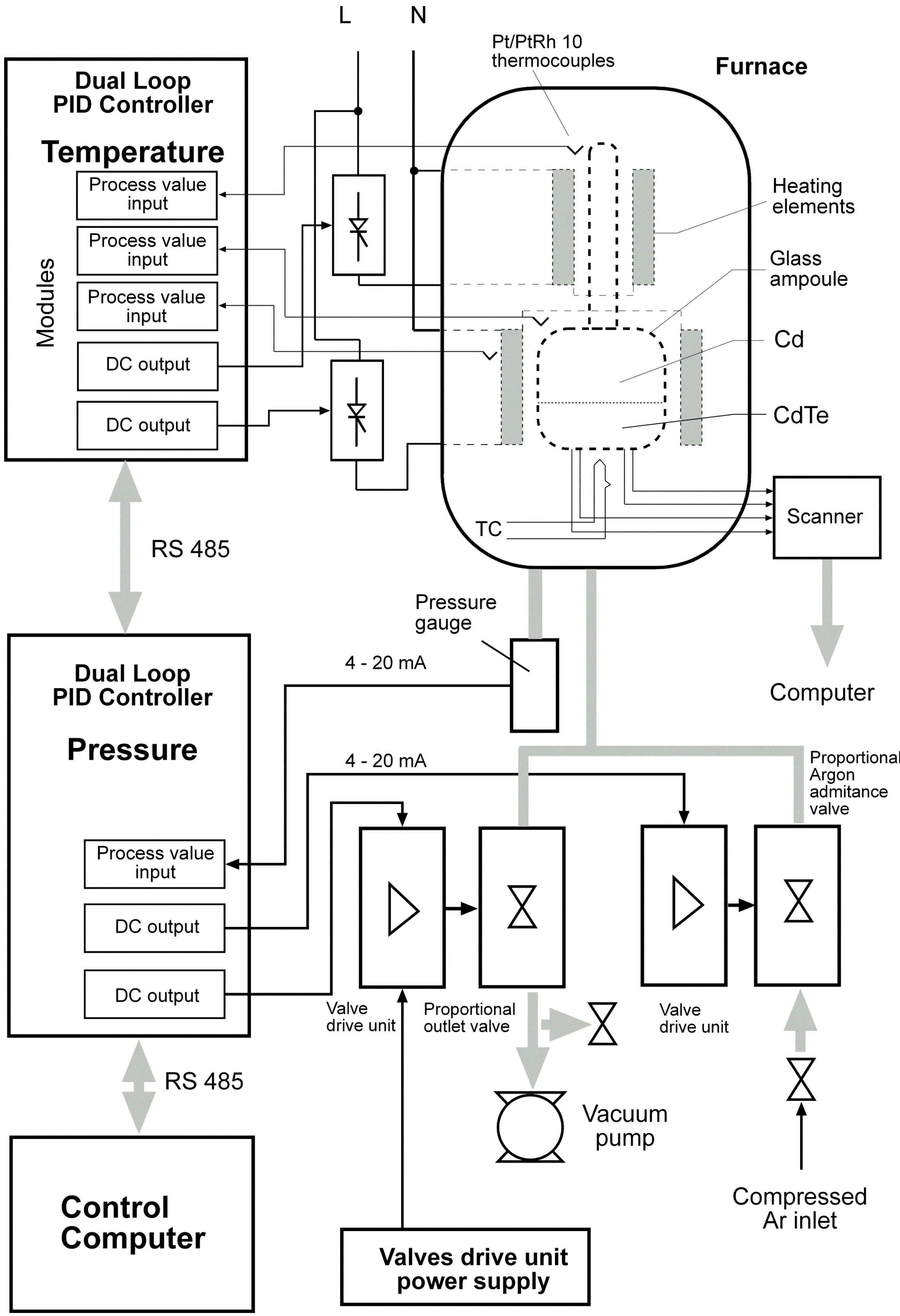

Fig. 1. Electronic control setup of the furnace

used to transmit instantaneous pressure value to the controller. The pressure controller/setpoint programmer can control the Ar pressure profile during the process. The internal pressure stability without fast deviations is a very important assumption influencing the crystal growth process. Manual tuning of PID parameters by using Ziegler-Nichols algorithm has been necessary to get over big flow control nonlinearities resulting from the dependence of the valve opening characteristics on the internal pressure variations. The pressure stability interval can reach up to 50 mbar. Three separate sets of PID parameters are assigned to specified pressure intervals. The pressure setpoint value profile during the crystal growth process can be preprogrammed to the setpoint programmer or it can be calculated by the control computer on basis of temperature data analysis read from the temperature controller and the digital multimeter.

RS 485 two wire current loop serial communication bus is used for the data exchange and computer control of the furnace electronics setup. Digital multimeter scanner is simultaneously used for recording of various safety and process monitoring signals. It communicates with the control computer by using GPIB bus.

3. SUPERVISING CONTROL PROGRAM

The temperature profile in the furnace during the crystal growth process is preprogrammed in the temperature controller. The main task of the computer program is to control Ar pressure in the stainless steel containment surrounding the ampoule and the furnace. The other function of the program is data acquisition (and storage) for the documentation of the process.

After program invocation, one can perform certain modifications in the configuration of measuring apparatus, e.g. to enable additional digital multimeter input channels and to select its measuring modes. When the "Start" button is pressed, the program activity begins. From this moment, the changes in the configuration are not possible onwards, but some parameters are still changeable. The program runs in an infinite loop. Every 10 seconds the *PressureControl* procedure is called, and after three such cycles, the *DataMeasurements* procedure is called. With the exception of the most of the *PressureControl* procedure, the Windows messages (like button clicks) are processed, and therefore the program can react immediately to button clicks in the forms.

The *PressureControl* procedure is the most important part of the program. It performs the measurements of T_1 (upper zone) and T_3 (lower zone) temperatures to compute the approximate cadmium pressure in the ampoule and then it sends required setpoint value to the pressure controller to adjust Ar pressure in the containment. The Cd pressure P_{Cd} is calculated as $P_{Cd} = a \cdot f(\min(T_1, T_3)) + b$, where *f(T)* is the analytical function published in [10]. The analytical function is supposed to be valid up to 920 °C. As we need to extrapolate this dependence up to 1100 °C, two empirical coefficients a and b were introduced to improve the agreement with the real situation. Their values were estimated during the previous experiments. Calculated P_{Cd} (in mbar) is rounded to the nearest integer value and sent to the pressure controller. At the subroutine entry, a test of four sensors (temperature and Argon pressure) is performed by calling corresponding controller functions. If any sensor is broken, or if any of the measured values T_1 or T_3 are out of prescribed interval, or if they differ from the previous values by more than 50 °C, an error message occurs. If three consecutive errors has occurred, the experiment is stopped (both controllers are switched to "Hold" state, where the temperature and the pressure remains without changes).

In the *DataMeasurements* procedure, all experimental data are read, displayed and stored to a disk file. Besides this data file, another file "History.txt" is used to log all important situations during the process running, e.g. start and stop times, changes of controllers state (either entered in the program or manually from the controller front panel keypad), all warning and error messages, etc.

The computer program was written in Microsoft Visual Basic 6, it has almost 3000 lines of

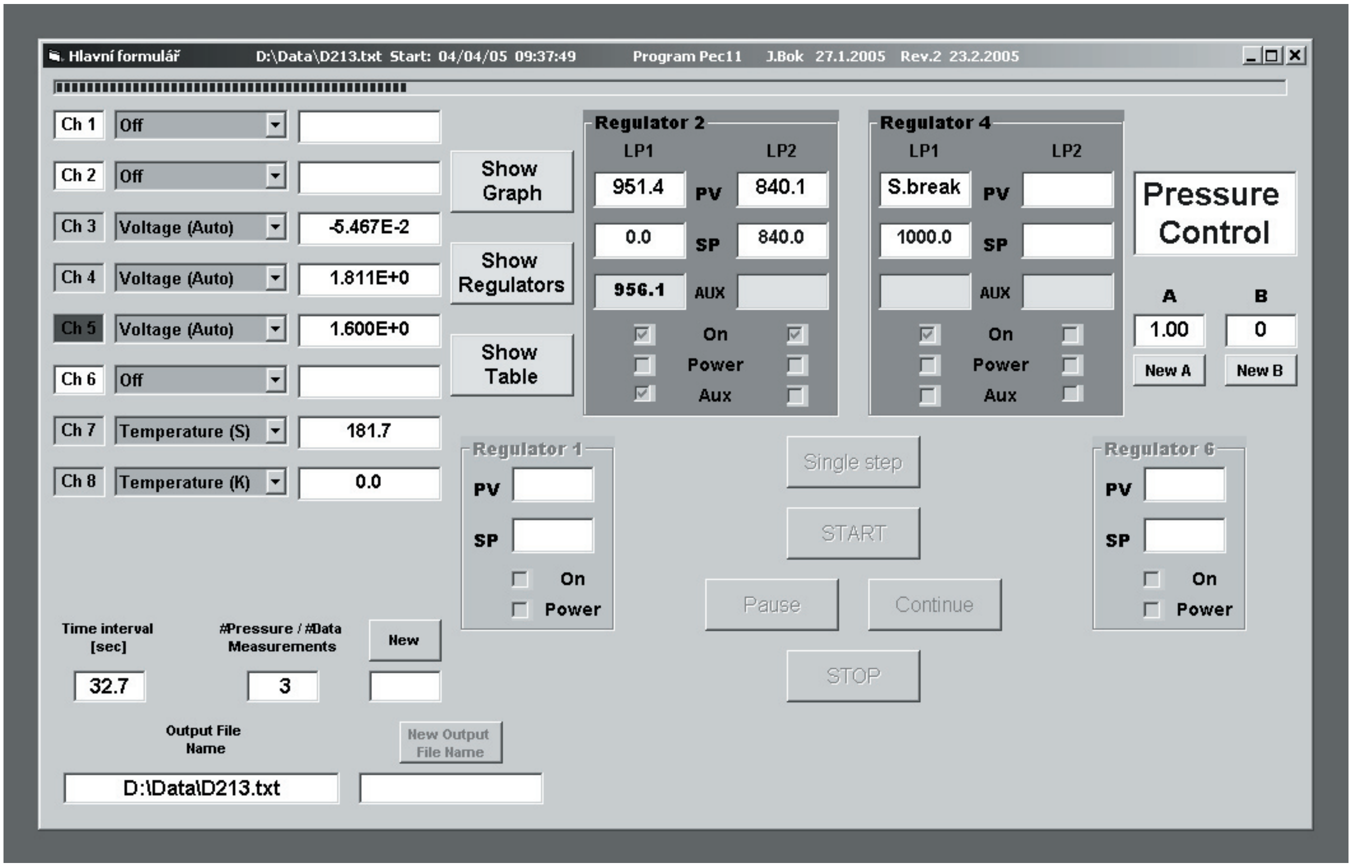

Fig. 2. *Main* form of the control program. At the left, digital multimeter channels are configured. In the centre, up to four possible controllers can be shown (two "big" are enabled by default). At the lower left, name of the data file and the "repeat factor" (number of pressure control measurements per one data storage measurement) are entered. At the upper right, empirical coefficients *a* and *b* (see below) can be changed (their default values are 1 and 0, respectively).

code. During the program run, the *Main* form (see Fig. 2) is permanently displayed, another 3 forms can be opened and closed arbitrarily. In the *ControlerControl* form, one can enter new *SetPoint* values, or to change the controller state, or to switch between the internal temperature profile programs. In the *Table* form, all data measured in *DataMeasurements* procedure are shown numerically. Finally, the *Plot* form shows a plot of 4 important quantities, each having its own vertical axis. Both horizontal axis (time) and all four displayed quantities independently can be completely controlled with zoom and shifts. When the *Plot* form is opened, the new values of all four quantities are added into the plot in real time. When operator is watching the older values, real-time mode is turned off automatically. It is turned on again by a click on the "Refresh" button, or after some time elapses.

4. CONCLUSIONS

CdTe and (CdZn)Te bulk single crystals growth furnace with high performance electronic control system was designed and constructed. The equipment enables full control of the Cd pressure during the process. A special computer program was written to control the crystal growth process and acquire all important data.

This work is a part of the research plan MSM 0021620834 that is financed by the Ministry of Education of the Czech Republic.

5. REFERENCES

[1] T. Asahi, O. Oda, Y. Taniguchi and A. Koyama, **J. Cryst. Growth** 161, 20 (1996).

[2] Yu. M. Ivanov, **J. Cryst. Growth** 194, 309 (1998).

[3] S. Sen, C. S. Liang, D. R. Rhiger, J. E. Stannard and H. F. Arlinghaus, **J. Electron. Mater.** 25, 1188 (1996).

[4] S. L. Price, H. L. Hettich, S. Sen, M. C. Currie, D. R. Rhiger and E.O. Mc Lean, **J. Electron. Mater.** 27, 564 (1998).

[5] K. Zanio, **Cadmium Telluride** (San Diego: Academic Press, 1978), p. 53.

[6] P. Rudolph, M. Muhlberg, **Materials Science and Engineering** B 16, 16 (1993).

[7] A. W. Were, S. Cole and D. T. Williams, **J. Electron. Mater.** 12, 551 (1983).

[8] Turkevych, J. Franc, R. Grill., P. Höschl, E. Belas, P. Moravec, **J.Electron.Mater.** 33, (2004) 658.

[9] **Eurotherm Engineering Handbook** Issue 3.1. April, 2002.

[10] K. Peters, A. Wenzel, P. Rudolph, **Cryst. Res. Technol.** 25, (1990), 1107.