

**AUTOMATED INSTALLATION FOR PRODUCTION
OF THE THERMOELECTRIC MATERIALS PROFILED CRYSTALS**

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Abstract

The problems of the automated installation design for the industrial production of thermoelectric materials based on bismuth telluride are under consideration. The main features of technology and installation implementing it are discussed and construction of the automated control system based on industrial and microcontroller tools is presented. The paper gives short overview of the processing equipment and the special software technical design, as well as the full layout of automated installation manufacturing and testing technical design.

Key words: *Thermoelectric Materials, Bismuth Telluride, Oriented Crystallization, Shaper, Synthesis, Casting, Annealing*

1. INTRODUCTION

At present semiconductor technology of manufacturing of thermoelectric materials (TEM) are widely used around the world. Semiconductor thermoelectric devices (modules) used to generate electric power, temperature control, cold production in various industrial sectors and households, are made from them.

Some results of creation in ETU (Department of Automatic Control Systems) an automated system for manufacturing crystals of thermoelectric materials, satisfies technology requirements developed by the of OAO "Research and Production Enterprise of thermophysical instruments" OSTERM SPB.

2. FEATURES OF THE TECHNOLOGY, IMPLEMENTED IN CREATED FACILITY

There are various methods for obtaining thermoelectric materials used in certain types of raw material [1]. The basis of implemented by automated installation technology for obtaining (casting, synthesis, crystallization, and annealing) of thermoelectric materials based on bismuth telluride on the method of directional solidification, which consists in the formation of controlled temperature gradient in the field of crystallization.

Directed crystallization method, which consists in the formation of controlled temperature gradient in the crystallization is the basis of implemented by an automated installation technology for obtaining (casting, synthesis, crystallization, and annealing) of thermoelectric materials based on bismuth telluride. Control of temperature field can be performed either through mechanical movement of the shaper with a substance relative to the heater [2, 10, 13], or by adjusting the heater itself (see for example [15]) or by heaters temperature range control (and chillers) (as for example, in [9, 12]).

The last control method is realized in the installation as the most progressive. This method allows flexibly control the distribution of temperature throughout the volume of the shaper to obtain crystals with given electrophysical properties. In particular, when using the shaper of elongated forms, suggest a horizontal or vertical, as in the created installation, directions of temperature gradient (see, eg, [2, 13.15]).

Profiling generated grown crystals of thermoelectric materials is provide in the installation by making injection chambers of the shaper geometric dimensions close to the required size of materials. This is done to reduce the time and energy costs during crystallization, as well as losses of raw materials during the final mechanical processing the crystals (see for example [11, 14]). In addition, the crystallization process is carried out at a pressure of protective gas environment, such as in [6, 16].

3. DESCRIPTION OF THE CREATED INSTALLATION FUNCTIONAL STRUCTURE

The technological process of obtaining profiled crystals of thermoelectric materials based on bismuth telluride is carried out as a single process in the installation. It combines processes of synthesis, casting, annealing and crystallization occurring in a vacuum chamber using a special graphite disassembled container - reusable shaper.

The basis of the installation is working chamber (tube furnace). It is composed of graphite heaters, mixing device and the gas-vacuum system, consisting of a vacuum pump, pressure gauge, control valve and connecting hoses and vacuum provides the internal volume of the working chamber and to maintain positive pressure of inert gas. In the working chamber is placed shaper, having at the top of the boot capacity, and the bottom - injection molds (crystallization cell), connected with a loading capacity of the holes. Graphite heaters are located in the upper part of the working chamber, and the base of the shaper is on a pedestal, equipped with water cooling.

In the synthesis raw materials (chemical elements Bi, Te, Sb, Se) are fused in certain combinations and proportions in the boot area of the shaper to form a feedstock for the subsequent growth of crystals of thermoelectric materials. Synthesis is accompanied by intensive mixing of the melt, reduces the time of synthesis and, consequently, the loss of highly volatile components of the melt. To suppress the volatilization of the components is also applied filling the working chamber with argon overpressure.

The casting process is carried out at least the end of the synthesis process.

Simultaneously with the temperature in the chamber rises, there is penetration of ports (gates) separating the sections of synthesis and formation, the melt flows, filling the cavity shaping crystallization.

The process of directed crystallization is provided by controlled-oriented vertical temperature gradient. At the same time to control the heating of the graphite heater with the top of the mold and cooled shaper with a water-cooled pedestal at the bottom of the mold shaper. Temperature mode of the crystallization process is measured by the thermocouples placed at the top and bottom of the shaper.

At the final stage of manufacturing profiled crystals are usually subjected to subsequent annealing at the experimentally determined annealing temperature. This temperature is always lower temperature of crystallization.

A transition to the annealing temperature exercise in two ways. In the first method the crystals rapidly cooled to ambient temperature and then slowly heated to the annealing temperature. By the second

method crystals are slowly cooled from the crystallization temperature to ambient temperature and then heated to the annealing temperature. Annealing by both methods is made directly to the shaper without unloading thermoelectric materials after crystallization.

In accordance with technical specifications compiled by "NPP TFP" Osterm SPb ", at Automatic Control System Department was developed construction documentation E SPVG.040441.01 and made a working model of the automated installation of synthesis, casting, crystallization and annealing of crystals shaped thermoelectric materials with precise casting equipment (the act of manufacturing № 444/54-2 from 21.06. 2011)

Figure 1 shows a photograph of layout automated installation, located in the educational and scientific laboratory at the Automatic Control System Department. Laboratory was founded jointly with JSC "NPF TFP" Osterm St. Petersburg ". Figure 2 shows a general drawing of the basic structural component of layout - the working chamber.



Fig. 1. General drawing of the basic structural component of layout - the working chamber

All the nodes titles of the working chamber, as shown on drawing callouts (Fig. 2), previously explained in the text.

4. THE AUTOMATIC CONTROL OF TECHNOLOGICAL PROCESS IMPLEMENTED BY THE INSTALLATION

An important part of automated installation is the development of technological process automated control system.

Automated control system provides the following functions:

- Automatic control of the main process parameters:
internal temperature of mixing, the temperature of the shaper, heater temperature, the temperature pedestal, the pressure inside the chamber mixing to ensure the stabilization of the given technological conditions;
- Automatic start of preparatory operations preceding the beginning of the cycle (gas and vacuum subsystem);

- Automatic generation of the start / stop signal for production equipment (actuators) subsystem of heating, cooling, ventilation, and mixing;
- Automatic monitoring of the technological process;
- Alarms on emergency deviations of main parameters of technological processes and the state of the actuators;
- Scheduling, registration, processing and archiving of data: visualization subsystem / SCADA.

The hardware part of the automated control system is realized as:

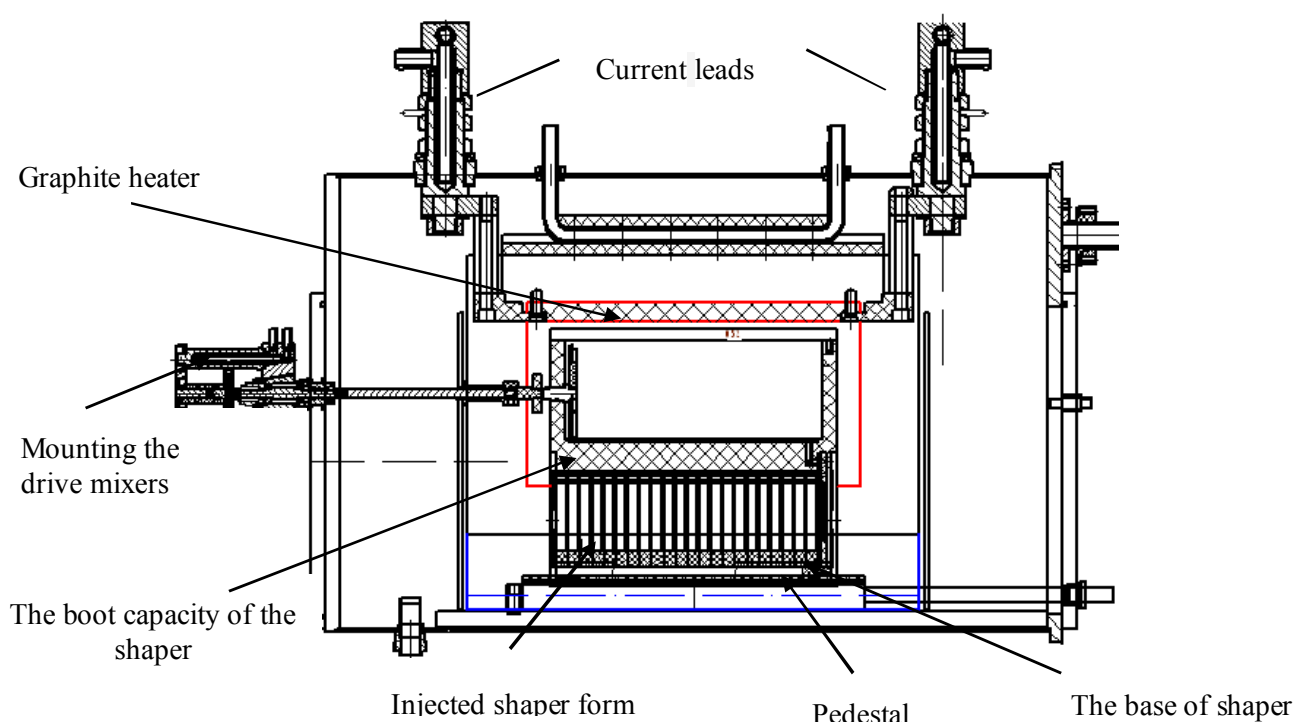


Fig. 2. Nodes of the working chamber

- Computer operator panel, based on of the industrial controller TPC 12 606 touch screen 12.1 "(shown in Fig.3);
- Specially designed control board, based on the microcontroller Atmega 128 A from the periphery and a connector for programming (shown in Figure 4.)



Fig. 3. Computer operator panel



Fig. 4. control board, based on the microcontroller Atmega 128 A

Automated control system functions are realized with special software, which organizes: automatic control of technological process with given algorithms, data exchange with interface controller, human-machine interface and real-time visualization of technological process, emergency signalization and alarm control, storing of process data.

Main window displays data of bar and temperature sensors on the plot and in the digital poles. Buttons T1-T4 are used to switch current plot. Special buttons with arrows let user to move along the time axis.

Using this window operator may supervise the technological process in the real-time: it has buttons to turn cooling subroutine on and off, to safely emergency stop the process using software procedure, to look through the archived data of the process parameters. Buttons of the selection of the algorithm of the process let operator to choose between different technological processes with preset parameters.

The amount of these programs is compliment to the set of forms precision casting tooling shaper, which are contained in the installation. Each form has its set of parameters of crystal synthesis - bar, temperature, heating and cooling rates.

Fig. 5 shows a user window interface displayed on the screen industrial computer.

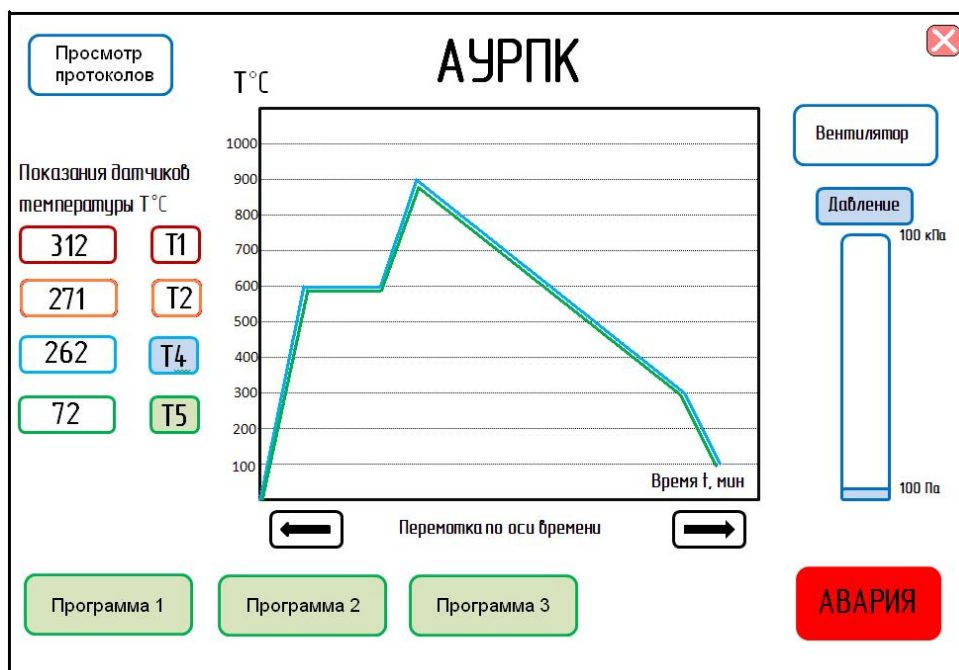


Fig. 5. User window interface

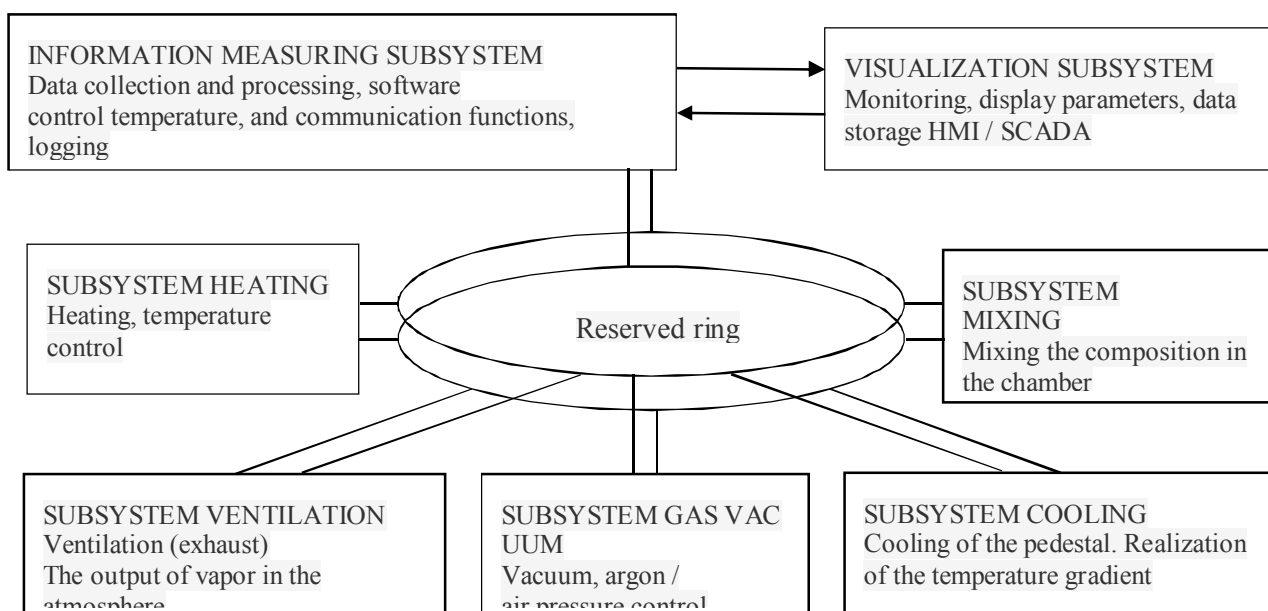


Fig. 6. The block-diagram of the system

Software includes two subroutines: one for human-machine interfacing is realized in the computer operator panel (fig. 3) and one for automatic control of technological process is realized in microcontroller installed on the control board (fig.4).

The block-diagram of the system (including control part and processing part connected with "reserved ring" network with computer operator panel as "master") is shown on the fig.6.

On the existing layout, developed automated installation production thermoelectric materials bismuth telluride (see Fig. 3, 4, 5) were made experimental studies (trial heat). They confirm the safe and reliable operation of automated installation during all modes of operation.

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