TECHNICS OF HYDROTHERMAL EXPERIMENT UNDER NOT UNIFORM CYCLICALLY CHANGING HEAT FIELD CONDITIONS

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Traditional approach for hydrothemal crystal growth implies the creation of as stable as possible stationary heat field. Nevertheless, sporadic temperature fluctuations affecting both dissolution and crystallization processes exist inside the autoclave. They are due to the pulsation character of heat-mass transfer in the growth medium. In order to regularize convective processes inside the autoclave a new approach based on the creation of not uniform cyclically changing heat field at the outer walls of the autoclave has been proposed [1].

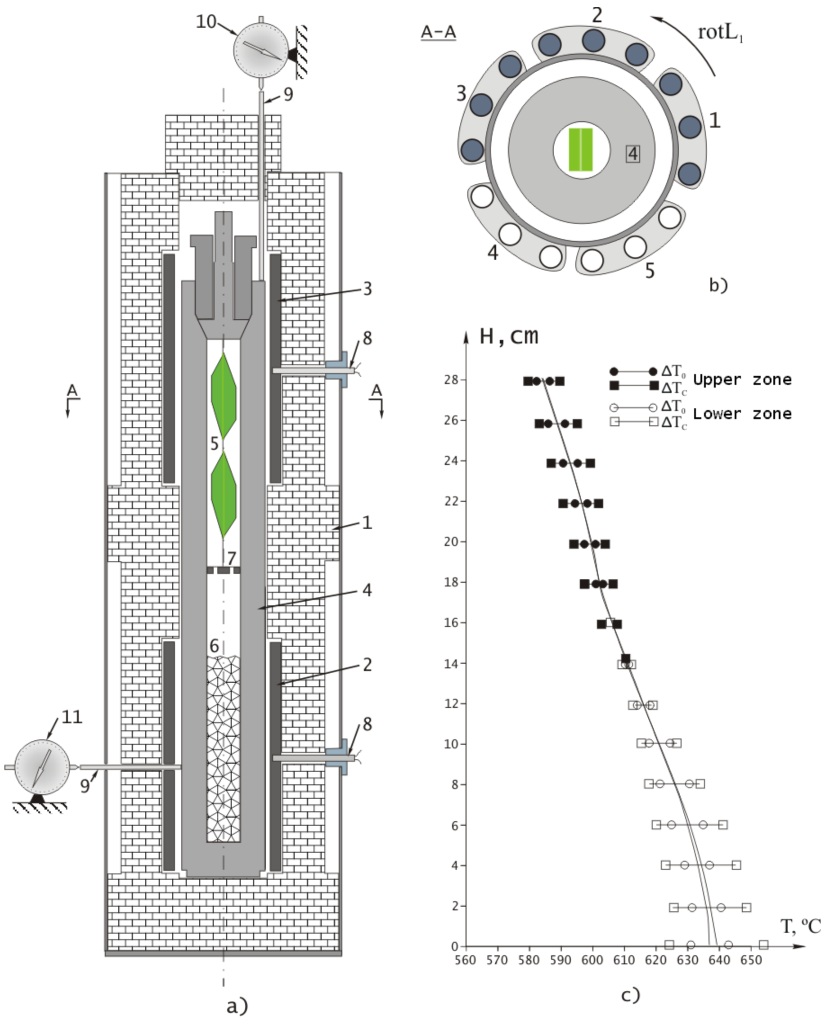

Fig.1. a) Scheme of the setup: (1) heat-insulated chamotte frame of heating furnace, (2) и (3) heaters of upper and lower zones, correspondingly, (4) autoclave, (5) the zone of crystallization, (6) the zone of dissolution, (7) diaphragm, (8) regulating thermocouple, (9) quartz stems, (10) micrometer for registration of autoclave lengthening, (11) micrometer for registration of autoclave widening; б) realization of the rotating heat field with rotL$_1$ symmetry; в) temperature distribution inside the autoclave at τ=90c: T_o - along the axis, T_c – near the inner wall.

The scheme of the experimental setup is shown in the fig.1a. Steel autoclave with 80mm external, 30 mm internal diameters and 450 mm height is put into two-zone heating furnace. Each zone consists of 15 evenly distributed heaters. They are divided into five group of three consecutively connected heaters. Each heating zone is managed by individual thermocontroller, which allows to create necessary temperature difference between crystallization (upper) and dissolution (lower) zones. Parallel Pt-Pt/10%Rh thermocouple is used as a temperature sensor. It consists of five working junctions placed into each group of heaters. The load commutator providing the connection of heater's groups according to the desired program is used in the thermal control system [2]. For example, if we consequently switch triplets of heaters 1-2-3→ 2-3-4→ 3-4-5, etc., symmetry of the rotating heat field, will be rotL$_1$ (fig.1b). At a short period of switching temperature fluctuations inside the autoclave are not measured and the heat field is nearly stationary having L_∞ symmetry. Fig.1c shows temperature distribution along autoclave axis T_o and near the

the inner wall T_c of the autoclave at a period of switching τ=90c. Horizontal lines mark the amplitude of temperature fluctuations observed in each point of measuring in both upper and lower zones. The results of measuring of temperature fluctuations at the center and near the inner wall of the autoclave depending on τ have been presented in [2]. Substantial temperature fluctuations of about 0.5°C arise only at a period of switching $\tau \geq 25$c.

The experiments on hydrothermal synthesis of single emerald crystals have been carried out via recrystallization of natural beryl charge from Izumrydnyje Kopi gem locality (Urals). Maximum temperatures (640-650°C) in the dissolution zone were defined by strength properties of autoclave. Crystals were grown in composite water solution containing chlorides (halides) and fluorides. The autoclave was filled with a calculated amount of water defining the pressure of 1200-1500 atmosphere. Both synthetic and natural beryl seed plates were used being cut parallel to (5.5-10.6) plane. First experiments have shown that under the action of a rotating heat field within lower zone heat-mass transfer proceeds at least 1.5 times faster then in stationary and stable heat field [1].

For indirect control of PT-conditions *in situ* two micrometers have been installed into the setup. Being connected with autoclave vie pushers of quartz stems they provide a possibility for registration of autoclave linear size variations – lengthening and widening (fig.2). The autoclave widening has been measured at a height of 90 mm from its bottom – the maximum widening appeared to be at this height.

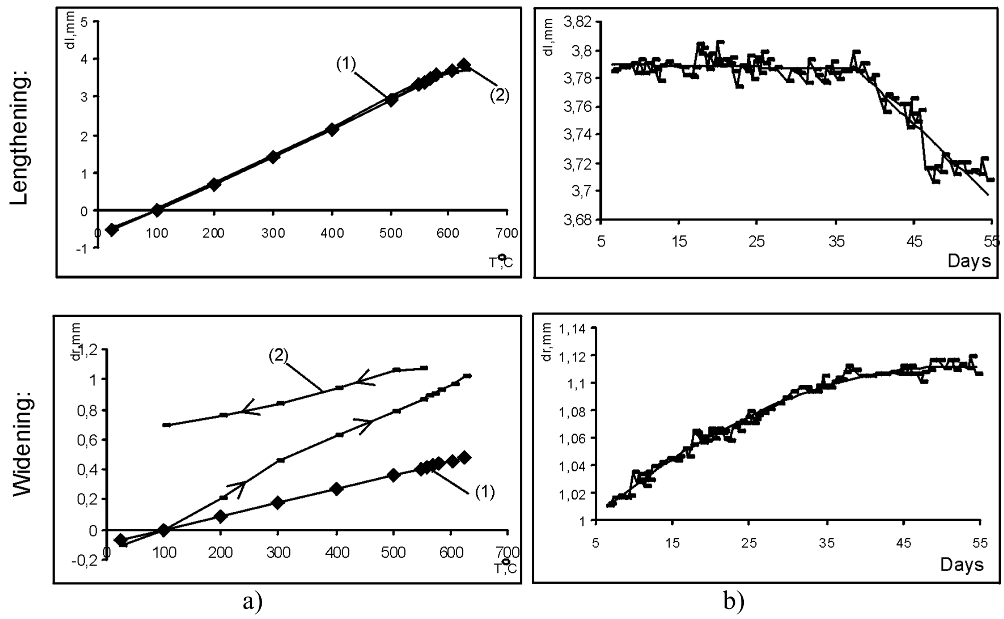

Fig.2. Micrometers indications: a) during the heating and cooling of empty (1) and filled (2) autoclave; б) during growth experiment.

According to the analysis of data in fig.2 average-integrated temperature in autoclave can be estimated from lengthening value with ±1°C accuracy. The last corresponds to the lengthening ±5 mkm. Sharp curve of lengthening in fid.2b corresponds to the insertion of cooling in upper zone with 1°C/day rate on 37th day of growth circle. Registration of the widening shows that autoclave works in creepage temperature regime [3]. Control over this parameter gives important information about safety pressure inside the autoclave. Widening should be control in a way implying residual value (after run carrying out) to be less than 0.5mm.

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