Control System for Chemical Thermal Processes and Its Usage for Measurement of Collagen Shrinkage Temperature

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Abstract: - This paper presents a hardware controller designed for a real-time control of laboratory thermal processes and describes its application to measurement of shrinkage temperature of collagen materials stabilized by ionizing radiation is described. Both hardware and software of the controller is presented in the paper. The controller can be used to control temperature of various technological and chemical processes. The controller contains a temperature sensor and a pulse width modulation actuator for the AC power supply of a thermal process. Fixed parameter control algorithms as well as adaptive control algorithms are programmed into the controller. Control of heating mantle is presented as an example of a real-time application of the controller. Measurement of shrinkage temperature is as an extension of heating mantle control is presented.

Key-Words: - temperature control, thermal process, adaptive control, collagen shrinkage

1 Introduction

Temperature control is a common task in many technological processes. Almost all chemical processes are temperature dependent and temperature control is required to obtain precise results. One of the simplest pieces of temperature control equipment consists of an electric heater and a temperature sensor used as a feedback. Such instruments are very common in chemical laboratories: a hot plate, an immersion heater, heating mantle.

Common drawbacks of most of available laboratory heaters, especially the low–cost ones, can be seen in the following areas:

- There is no temperature sensor providing a feedback.
- The temperature sensor measures temperature of the heating element itself, not the part that actually is to be heated (usually some liquid).
- Only a control with constant reference signal is provided. The required temperature level can be set manually.
- Controller behavior cannot be changed. The controller is hard-wired and it is not possible to define transient response of the system in terms of e.g. defensive, slow and smooth control vs. aggressive fast control with overshoots.
- The course of the reference signal (required temperature), controlled signal (actual temperature) and control signal (power of the heater) is not recorded for further evaluation.

To cope with these drawbacks a universal controller was designed and manufactured. This controller and is presented in the paper. Despite the fact that the presented controller is designed for single input single output (SISO) systems it can be applied also to suitable symmetric multivariable systems using decoupling [1].

The paper is organized as follows. Section 2 describes the controller from the hardware point of view; section 3 shortly presents controller's software. A heating mantle as an example of a controlled system is presented in section 4. The temperature control of the heating mantle is described in section 5. Practical application of the developed control system was measurement of shrinkage temperature of collagen materials stabilized by ionizing radiation which is presented in section 6. A short conclusion is provided in section 7.

2 Hardware of the controller

2.1 Controller overview

The controller is intended for usage with simple heating elements that can be controlled by switching their power supply on and off. Advanced heating systems equipped with microcontrollers are not, in general, suitable for usage with the proposed controller.

A simple scheme of the controller is presented in Fig. 1.

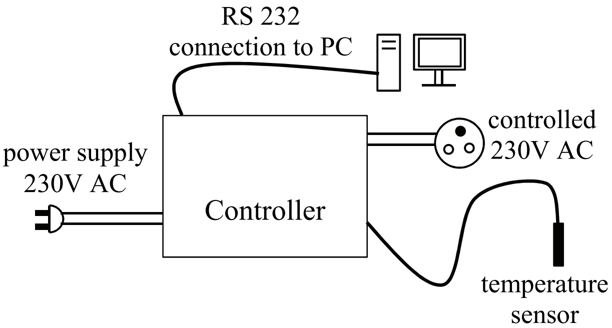

Fig. 1 Scheme of the controller

The controller is supplied by 230V AC and controls 230V AC socket which is used to plug-in the heating equipment. A pulse width modulation (PWM) is applied to this socket to control the output power of the heater. A temperature sensor Pt1000 is connected to the controller to provide feedback for the control system. A personal computer (PC) or a laptop can be connected to the controller to achieve the ability of on-line recording of controller input, output, states and time. The controller itself contains very simple user interface as can be seen from the photograph of the controller which is presented in Fig. 2.

Fig. 2 Photo of the controller

2.2 Hardware elements

The following main components were used to manufacture the controller.

- **Power supply**. A transformer was used to obtain 9V DC to supply for CPU and other electronic parts.
- **CPU**. A Freescale MC9S08AC128 microcontroller was used a as heart of the controller. It operates at 40 MHz and contains 128 kB of FLASH memory and 8kB of RAM memory. Peripheral devices can be connected via SPI or SCI serial interfaces. This CPU is equipped with 16-channel AD converter with 10 bit resolution. Moreover it contains one dual-channel and two 16-channel timers which can be used for PWM. More detail can be found in [2].
- **External AD convertor**. The controller is equipped with MCP3551 AD converter produced by Microchip Technology. It communicates with CPU using SPI interface. The resolution of the converter is 22 bits which ensures sufficient preciseness of temperature control even for wide range of operating points [3]. Detail concerning connection of the AD converter can be found in [4]
- **EEPROM**. A 32kB EEPROM memory by ATMEL is used to store controller parameters and other user settings. It is connected to SPI. Detailes are available in [5].
- **RS232 converter**. A MAXIM RS232 converter is used for interface of the controller and a PC.
- **SSR**. A solid stare relay (SSR) by Carlo Gavazzi company is used as an actuator for the 230V AC output of the controller [6]. The maximal switched current of the relay is 25A which is enough for a laboratory deployment of the controller. The changing of state of SSR (switching on or off) is performed in synchronous way: the switching is delayed until the power-line voltage is passing through zero.

The controller contains several printed circuit boards (PCB). All of them were designed using CadSoft EAGLE PCB Design Software.

3 Software of the controller

This section briefly describes software equipment of the controller. The first subchapter is focused on user interface, remaining two subchapters a cope with controller algorithms and on-line identification.

3.1 User Interface

The user interface of the controller consists of the following items as depicted in Fig. 2:

- display
- three LEDs (power on indicator, output indicator, malfunction)
- on/off switch
- master stop button
- Esc and OK buttons
- arrow buttons

The arrow buttons together with Esc and OK buttons are used to browse the menu and to change settings.

The setting sections serves for defining various parameters of the controller. The sample time is used in measurement and RS232 communication; each controller has its own sample time. It is possible to define up to 10 reference signal courses. Each reference signal is piecewise linear consisting of up to 15 sections. It is also possible to define up to 10 settings for each of the three types of the controller. Controller types are discussed in the next subsection. The thermometer calibration can be used to precisely define relation between resistance of the Pt1000 sensor and the temperature in °C.

The menu structure is presented in Fig. 3.

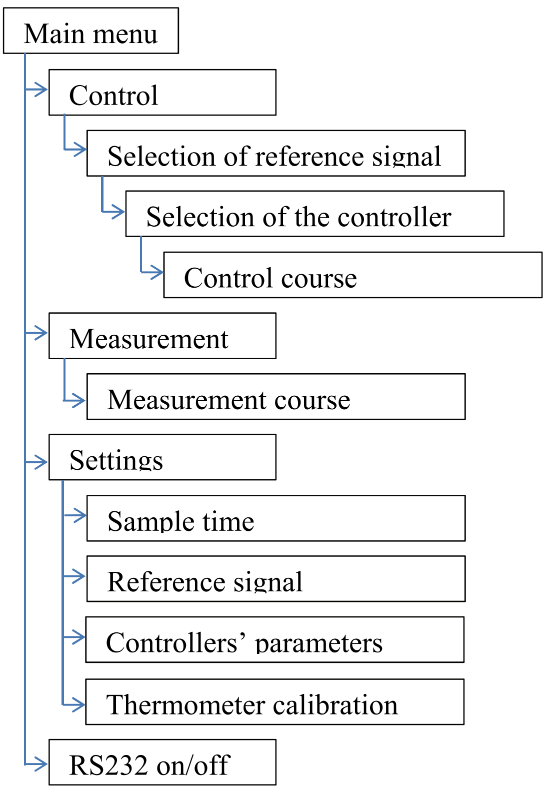

Fig. 3 Menu structure

The controller submenu is used to select appropriate reference signal, controller and to start the control process.

The measurement menu is used to measure the temperature only. This function can be used for example for step response measurement.

3.2 Control Algorithms

The controller contains the following three different control algorithms:

- adaptive dead-beat controller
- adaptive pole-placement controller
- discrete PID controller

The first two controllers belong to a self-tuning controllers group [7], [8]. Adaptive controllers are often used in chemical control problems [9]. An on-line identification is used to obtain ARX model of the controlled system. The structure of the control loop is depicted in Fig. 4.

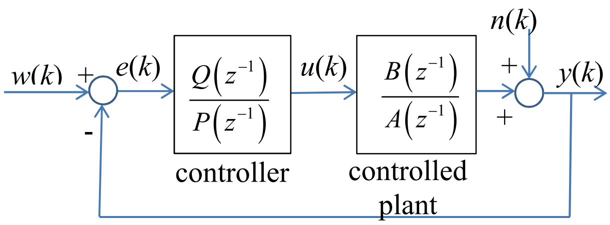

Fig. 4 Control loop

The reference signal is represented by $w(k)$; symbols $e(k)$, $u(k)$, $y(k)$ and $n(k)$ represent control error, control signal, output signal and disturbance respectively. $Q(z^{-1})$ and $P(z^{-1})$ are polynomials of the controller and $B(z^{-1})$ and $A(z^{-1})$ are polynomials of the model of the controlled system. The coefficients of controller polynomials are computed in each sample step when adaptive controllers are used. The discrete PID controller uses constant coefficients. The transfer function of both adaptive controllers is as follows:

$$G_r\left(z^{-1}\right)=\frac{Q\left(z^{-1}\right)}{P\left(z^{-1}\right)}=\frac{q_0+q_1z^{-1}+q_2z^{-2}}{\left(1-z^{-1}\right)\left(p_0+p_1z^{-1}\right)} \tag{1}$$

The following transfer function describes discrete PID controller:

$$G_r\left(z^{-1}\right)=\frac{Q\left(z^{-1}\right)}{P\left(z^{-1}\right)}=\frac{q_0+q_1z^{-1}+q_2z^{-2}}{1-z^{-1}} \tag{2}$$

Parameters of the controllers can be set by a user and therefore various control design techniques can be used e.g. robust control [10] or advanced PID tuning [11]. On the other hand application of artificial intelligence [12] is beyond the limits of controller hardware.

3.3 On-line Identification

Parameters of adaptive controllers are computed on basis of the ARX model of the controlled system. These adaptive controllers are based on the self-tuning approach where on-line identification is used to obtain ARX model [13]. The ARX model is described by equation (3)

$$y(k) = \phi^T(k)\theta(k) + e_s(k) \tag{3}$$

where $\phi^T(k)$ is the data vector and $\theta(k)$ is the vector of model parameters

Parameter estimates are updated in each step:

$$\hat{\theta}(k) = \hat{\theta}(k-1) + \frac{C(k-1)\phi(k)}{1+\xi(k)}\hat{e}(k) \tag{4}$$

where

$$\xi(k) = \phi^T(k)C(k-1)\phi(k) \tag{5}$$

and $\hat{e}(k)$ is prediction error

$$\hat{e}(k) = y(k) - \hat{y}(k) \tag{6}$$

Covariance matrix is updated in each step:

$$C(k) = C(k-1) - \frac{C(k-1)\phi^T(k)\phi(k)C(k-1)}{1+\xi(k)} \tag{7}$$

This basic form of on-line least squares method can be further enhanced by exponential or adaptive forgetting [14] to obtain more precise model of the current behavior of the system. These modifications are useful especially in case of nonlinear or time-varying controlled system.

4 Heating mantle

4.1 Description of the Heating Mantle

The heating mantle is one of the often used pieces of equipment in chemical laboratories. It consists of spherical heating element connected to electric power supply, a case and optionally a temperature sensor with hysteresis which is used to switch the power supply on or off. Such a heating mantle can be used in connection with the proposed controller.

There are also more sophisticated heating mantles in the market which are controlled by microcontrollers but there are not suitable for the proposed controller.

A scheme of heating mantle is depicted in Fig. 5.

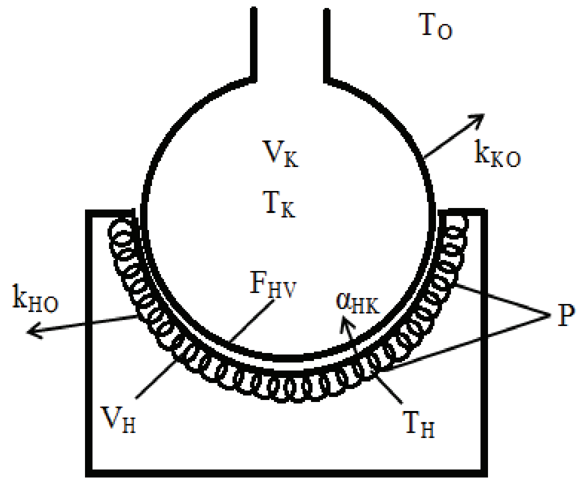

Fig. 5 Heating mantle scheme

The following parameters of the mantle are presented in the Fig. 5:

- T_O – ambient temperature [K]
- T_H – temperature of heating element of the mantle [K]
- T_K – temperature of the liquid [K]
- V_H – volume of the heating element [m^3]
- V_K – volume of the liquid [m^3]
- P – electric power of the mantle [W]
- F_{HK} – area of the bulb which is in contact with the mantle [m^2]
- k_{HO} – constant representing heat transfer from the mantle to its environs. It contains transfer from the heating element to the inner space of the mantle, accumulation of the heat inside the mantle as well as transfer from the heat case to its environs. [$W \cdot K^{-1}$]
- k_{KO} – constant representing heat transfer from the liquid to the environs. It is dependent on the level of the liquid. [$W \cdot K^{-1}$]
- α_{HK} – constant representing heat transfer from the heat element to the liquid. Heat capacity of the bulb is neglected which is valid in case if water level is not bellow the mantle margin [$W \cdot m^{-2} \cdot K^{-1}$]

The first principle model of the mantle contains also some other parameters:

- ρ_H – density of the heating element [$kg \cdot m^{-3}$]
- ρ_K – density of the liquid [$kg \cdot m^{-3}$]
- c_{pH} – specific heat capacity of the heating element [$J \cdot kg^{-1} \cdot K^{-1}$]
- c_{pK} – specific heat capacity of the liquid [$J \cdot kg^{-1} \cdot K^{-1}$]
- m_H – weight of the heating element [kg]
- m_K – weight of the liquid [kg]

Typical laboratory heating mantle is depicted in photograph in Fig. 6.

Fig. 6 Heating mantle

4.2 Mathematical model of the heating mantle

The first principle model [15] of the heating mantle is based on heat transfer balances. The heat balance of the mantle:

$$\begin{Bmatrix} \text{Heat from the} \\ \text{heating element} \end{Bmatrix} = \begin{Bmatrix} \text{Heat transferred} \\ \text{to the liquid} \end{Bmatrix} + \begin{Bmatrix} \text{Heat transferred} \\ \text{to environs} \end{Bmatrix} + \begin{Bmatrix} \text{Heat accumulated in} \\ \text{the heating element} \end{Bmatrix}$$

$$P = F_{HK} \cdot \alpha_{HK} \cdot (T_H - T_K) + k_{HO} \cdot (T_H - T_O) + V_H \cdot \rho_H \cdot c_{pH} \cdot \frac{dT_H}{dt} \tag{8}$$

Heat balance of the bulb is described by the following equation:

$$\begin{Bmatrix} \text{Heat transferred} \\ \text{from the mantle} \end{Bmatrix} = \begin{Bmatrix} \text{Heat transferred} \\ \text{to environs} \end{Bmatrix} + \begin{Bmatrix} \text{Heat accumulated} \\ \text{in the liquid} \end{Bmatrix}$$

$$F_{HK} \cdot \alpha_{HK} \cdot (T_H - T_K) = k_{KO} \cdot (T_K - T_O) + V_K \cdot \rho_K \cdot c_{pK} \cdot \frac{dT_K}{dt} \tag{9}$$

This simplified model can be used to create dynamic linearized model of the system, which can serve as a fundament for the control design. It is obvious that the linearized model should be of the second order because the first principle model has two states.

The real system contains several nonlinearities and is time dependent contrary to simplified first principle model.

5 Control of Heating Mantle

The controller was verified using several heating mantles and various liquids were heated. An example of temperature control of propylene glycol in a 480W heating mantle is presented in Fig. 7. A pole placement adaptive controller was used in this case.

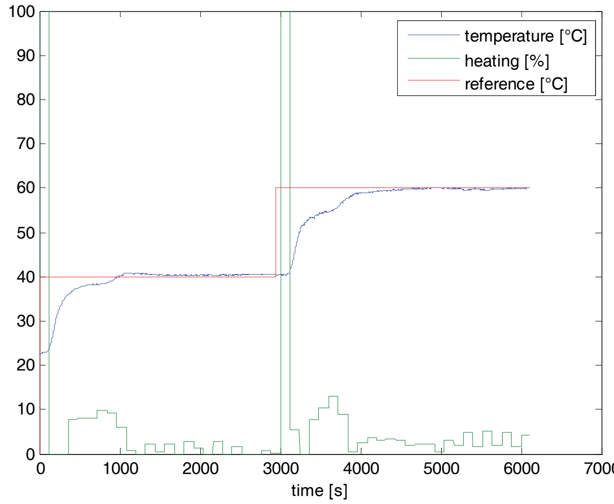

Fig. 7 Pole placement control of 1000ml of propylene glycol (480W mantle)

Heating of water in the same mantle is presented in Fig.8. In this case a discrete PID control algorithm was selected.

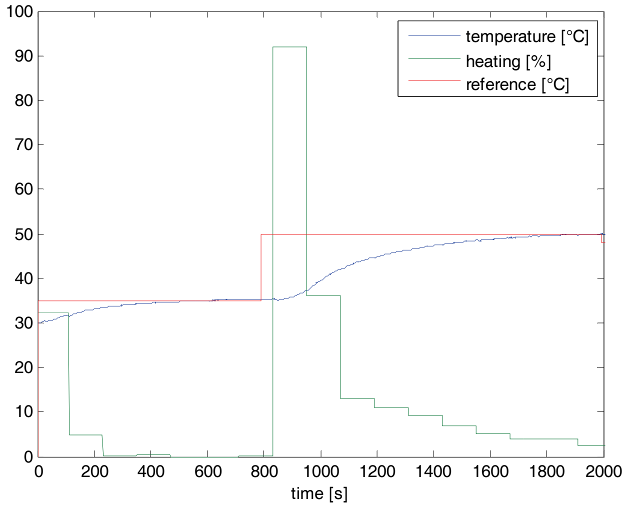

Fig. 8 Discrete PID control of 1500ml of water (480W mantle)

It can be observed, that in both cases the control algorithms coped with the task well.

6 Shrinkage of Collagen Materials

The controller was also used for control of water temperature in task of measurement of shrinkage of collagen materials. This task has been studied by many scientists and remains interesting till today [16], [17], [18].

Shrinkage temperatures of various collagen materials stabilized by ionizing radiation were measured.

Stabilization of collagen materials is important not only for example in food processing industry, but suitable and safe collagen stabilization methods have recently become subject of intensive research especially in relation to its medicinal applications. Collagen as natural material shows excellent biocompatibility, however in its native form is relatively unstable, with low mechanical strength. This so far limits its applications in particularly tissue engineering [19]. Stabilization (physical, chemical or a combination of both) leads to improvement of the material mechanical and thermal properties and substantially extends the application area.

There are several methods how to evaluate the extent of collagen stabilization. Shrinkage temperature, as irreversible phase change of collagen macromolecule and macroscopic display of denaturation, gives basic information not only on the degree of damage, but also on the degree of stabilization (e.g. the quantity of cross links formed in collagen structure). Combined effect of water and temperature leads to a collapse of organized collagen structure and to shrinkage of collagen fibres by approx. one third of its original length. The temperature at which most extensive length change occurs is called hydrothermal shrinkage temperature (TS).

In our case, the water was heated by electric plate. The plate power supply was connected to the output of the controller. The water inside the beaker was mixed to obtain approximately the same temperature inside its whole volume. Temperature was measured by Pt1000 sensor connected to the controller and the accuracy of the temperature reference tracking was verified by classical laboratory thermometer. The laboratory control setup photograph is presented in Fig. 9.

In this case, the temperature should rise slowly to be able to observe the shrinkage. An increase of temperature by 2°C/min was requested and a discrete PID controller was used to cope with this task. The controller was tuned using nonlinear optimization method to incorporate saturation to the control design. A control course is presented in Fig. 10.

Fig. 9 Setup for measurement of shrinkage of collagen materials

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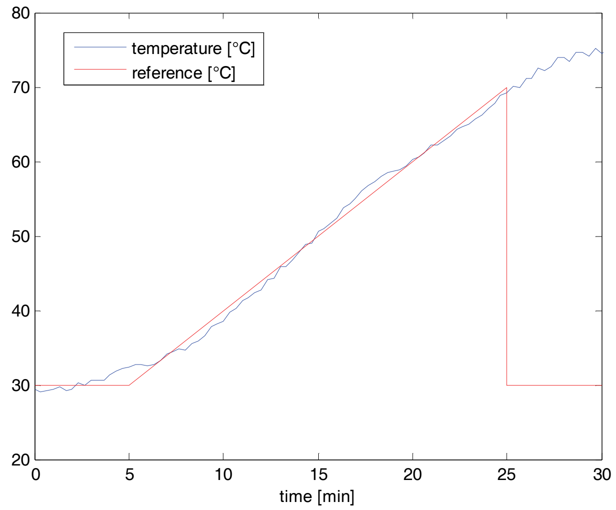

Fig. 10 Temperature control – shrinkage of collagen materials

The reference was set to 30°C in the last part of its course (after 25 min). This represents the end of experiment as the shrinkage was always observed before. The temperature of the water cannot follow this reference because no cooling was applied. Moreover, reference tracking is not important in this part as the experiment has already ended.

Examples of measurement of collagen shrinkage temperature for several different collagen samples are presented in Fig. 11, Fig. 12 and Fig 13. It can be seen that shrinkage occurred in all cases but different samples had different shrinkage temperature depending especially on the applied ionizing radiation.

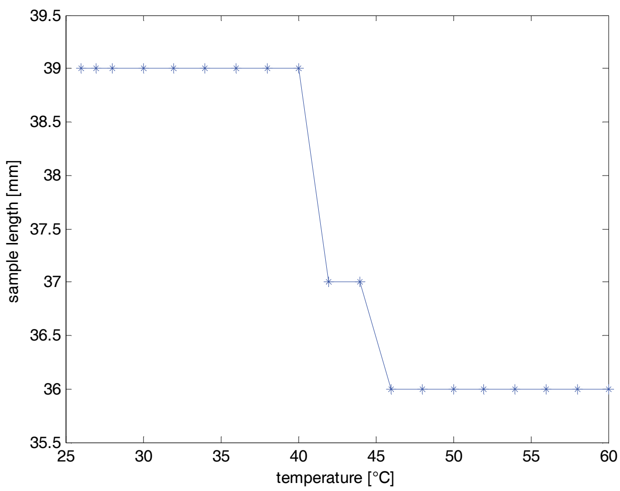

Fig. 11 Measurement of collagen shrinkage temperature (sample 1)

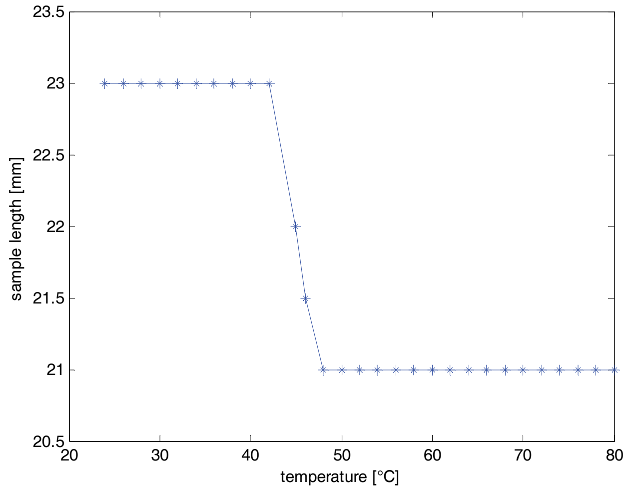

Fig. 12 Measurement of collagen shrinkage temperature (sample 2)

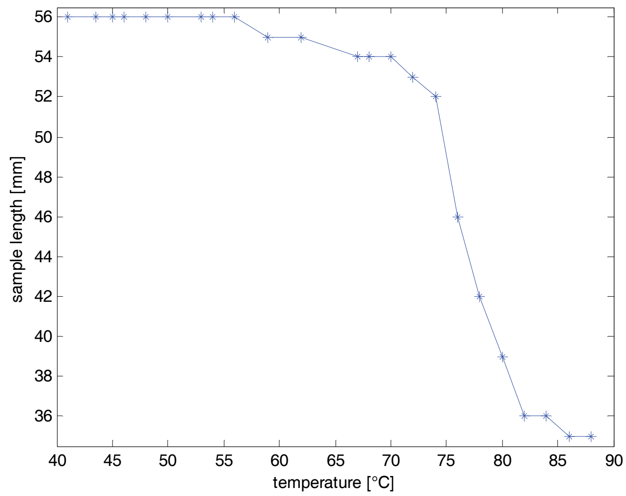

Fig. 12 Measurement of collagen shrinkage temperature (sample 2)

Measurement of shrinkage temperature was performed using a collagen material of known length which was fixed on a glass provided with a scale line and put in water the temperature of which was around 25°C. The water was heated in the said rate and collagen length was recorded at every time when the temperature changed by 2°C. The dependence of the material length on temperature was plotted in the graph and the shrinkage temperature was determined as the temperature of most dramatic length change.

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8 Conclusion

A controller for laboratory thermal processes was presented in this paper. Both hardware and software of the controller were described.

One of the main advantages of the designed controller can be seen in its applicability to a current simple laboratory heating or cooling systems. These simple systems either do not contain feedback at all or use just a feedback from the heating element and simple control algorithm. The presented controller allows for more sophisticated control algorithms based on a feedback from the heated liquid.

Real time experiments of control of a heating mantle and control of water temperature as a support for the measurement of the shrinkage temperature of collagen material were presented. Real-time experiments proved applicability of the controller in chemical laboratory.

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