ODO «MICROTESTRMACHINES»

Instrument for measurement of thermostimulated current

ST1

User manual

2020

Instrument for measurement of thermostimulated current ST1: User manual. – Gomel: ODO ‘Microtestmachines’, 2020. – 16 p.

Because of constant work on the instrument improvement and update of its mechanics, electronics and control software some differences may appear between actual implementation and this manual that, nevertheless, doesn’t mean the instrument quality worsening.

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CONTENT

1 GENERAL DESCRIPTION

Method of registration of thermally stimulated currents (TSC) or thermally stimulated depolarization is one of most effective mean to study charge state in dielectrics. The essence of the method is to study the relaxation of the charge that causes the electret state. To provide quick relaxation, investigators apply thermal stimulation of the dielectric discharge at constant heating rate. The analysis is based on property of the charges accumulated by dielectrics to become free at heating and on recording the electric current arising with that. Plotting the current value as function of temperature we get so called TSC spectrum that allows to detect mechanisms responsible for the effect.

Figure 1.1 shows general view of the instrument for measurement of thermostimulated current ST1.

In extended version the instrument includes additional function of electropolarization potential application to a sample that allows use of the measuring unit thermocell directly for charging the sample placed inside it.

a *b*

Рис. 1.1. General view of instrument for measurement of thermostimulated current ST1:
a – control electronic unit; *b* – measuring unit

Functional block diagram of the device is shown in Fig. 1.2.

Complete set of the instrument for measurement of thermostimulated current ST1 (Fig. 1.1) includes:

1. Measuring unit.
2. Control electronic unit with set of connecting cables.
3. In extended version – controller of electropolarization potential applied to sample (integrated with control electronic unit).

The instrument operates under control of specialized software run on host personal computer (with OS Windows). The specialized software is available for download from site http://microtm.com (ODO ‘Microtestmachines’).

Host PC should connect with the control electronic unit via USB-port. Additional drivers necessary for connection are available at https://www.ftdichip.com/Drivers/VCP.htm.

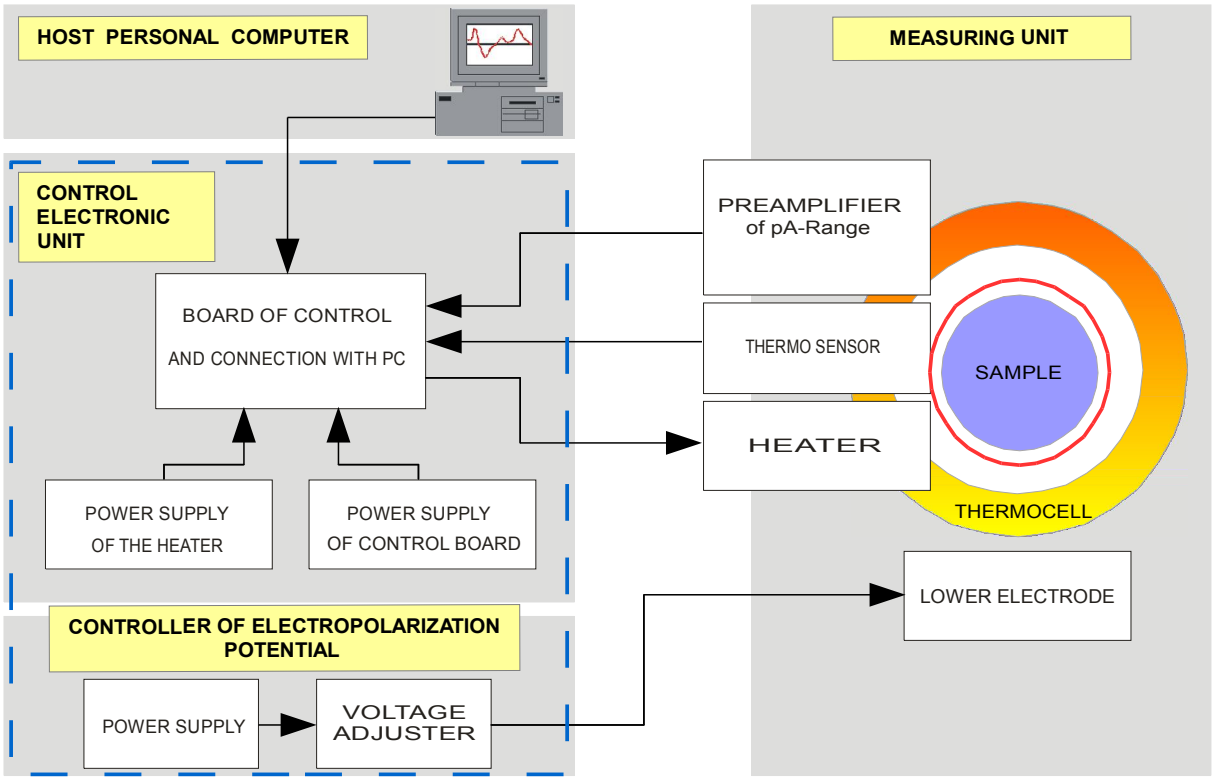


Рис. 1.2. Schematic diagram of the instrument for measurement of thermostimulated current ST1

1.1 Measuring unit

Scheme of the instrument measuring unit is shown in Fig. 1.3. The sample placed between grounded and measuring electrodes is heated inside a thermocell being metal cylinder. Heater is mounted around the metal cylinder. The electronic unit controls the heater by applying direct voltage (48 V) and regulating current. Outer shell around the thermocell and heater decreases thermal losses during operation.

The electrodes being introduced inside the thermocell are of copper.

Lower electrode is made of two parts: a changeable cap (outer diam. 26 mm) may be easily taken off for cleaning or replacement. A nonremovable part of the lower electrode is equipped with a thermosensor in the center that allows to measure temperature close to the sample. Lower electrode (its nonremovable part) is mounted on the top of a movable rod. The rod together with the lower electrode may be lifted above upper edge of the thermocell and that provides easy access to the changeable cap of the electrode and allows comfortable placement of studied sample on it. The lifting and sinking of the rod with the lower electrode is performed by a gear mechanism. To rotate the hoist gear, use the knob on the right side of the measuring unit (Fig.1.4).

Fig. 1.5 show steps of installation of the changeable cap on nonremovable part of the lower electrode using the hoist gear mechanism. The same routine is used for placement a sample to be measured on the lower electrode (Fig. 1.5 *d*). After that upper electrode holder may be inserted into the thermocell to press the sample. If the sample thickness is not enough to be pressed between the electrodes, slightly lift the lower electrode (by rotating the gear knob counterclockwise) till the upper electrode holder starts own lifting.

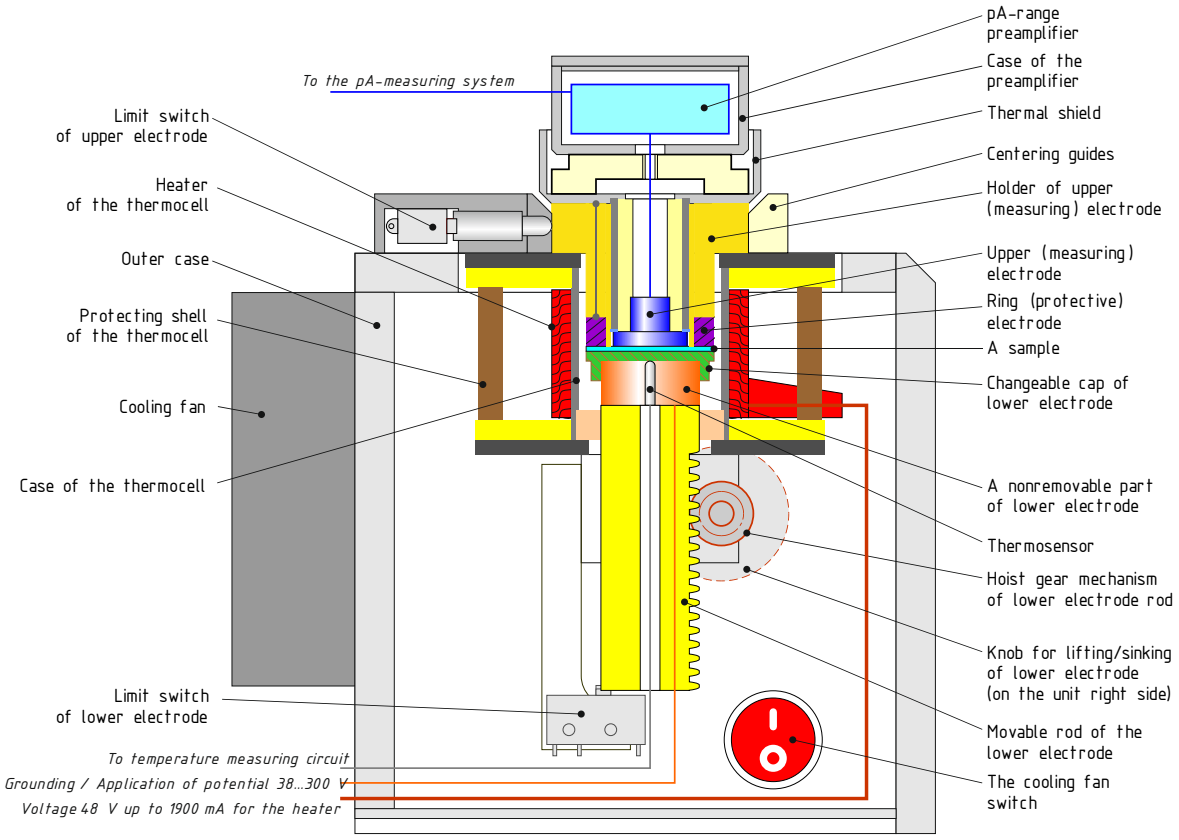


Fig. 1.3. Schematic diagram of measuring unit of the instrument for measurement of thermostimulated current ST1 (right view)

Fig. 1.4. The measuring unit.
Holder of upper electrode installed in regular position (case of the integrated preamplifier is above the unit upper cover). Knob of hoist gear mechanism to lift/sink lower electrode is on the unit right wall

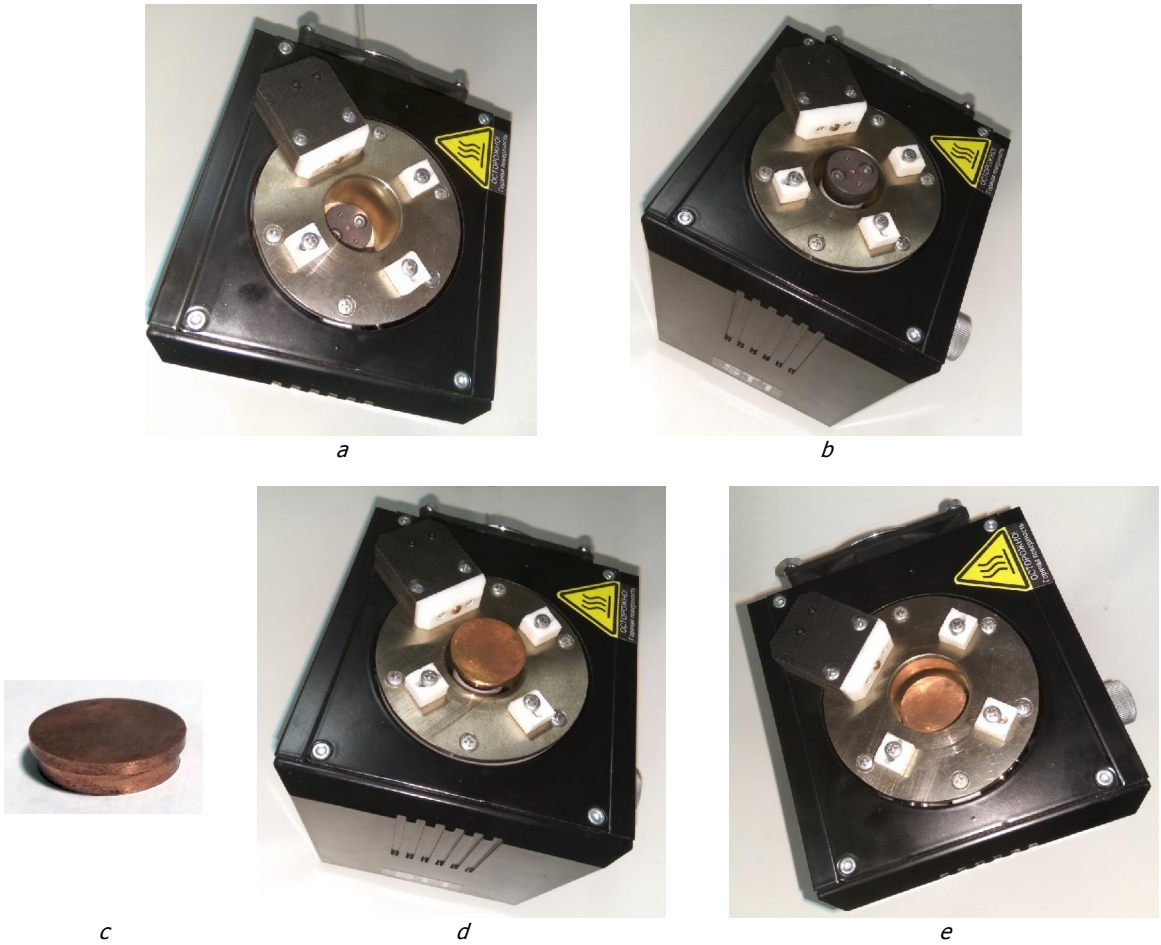
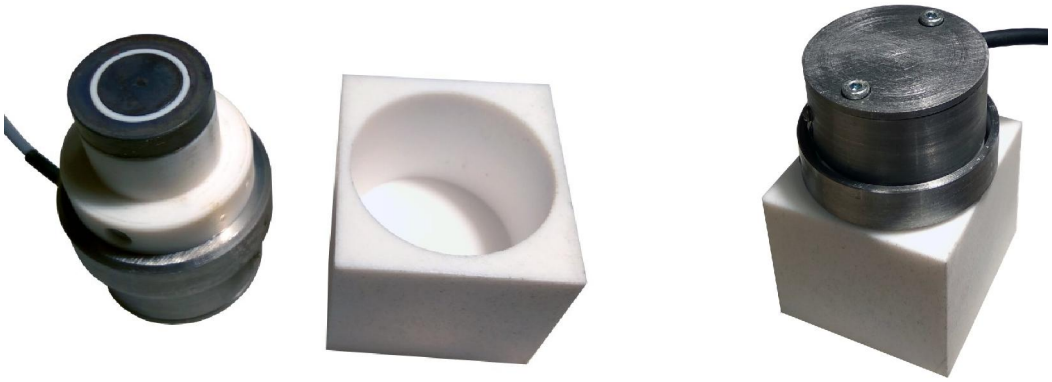


Fig. 1.5. Installation of changeable cap on nonremovable part of the lower electrode:
a – lower electrode (its nonremovable part) in its lower (regular) position; *b* – rotate knob of hoist gear counterclockwise to lift the lower electrode rod so that the nonremovable part appear above the thermocell upper cover; *c* – prepare the changeable cap; *d* – firmly install the changeable cap on top of the nonremovable part, at this step you may place a sample ready for measurement on the changeable cap top; *e* – rotate knob of the hoist gear clockwise to sink the lower electrode to its regular position inside the thermocell.

Upper electrode serves for measurement of depolarization current of a sample under study. Around the upper electrode of diam. 15 mm, a ring protecting electrode is mounted. Ring electrode of outer diam. 26 mm electrically connects with grounded case. Upper (measuring) electrode and its protective ring electrode are assembled in a replaceable holder (Fig. 1.6). On the holder top, integrated preamplifier of the measured signal (of pA-range) is mounted in a steel case. Such integration as well as shielding of all inner electric communications inside the holder allows to minimize outer electromagnetic influence. For measruments, insert the holder with the electrodes into the thermocell from upper side of the unit (see Fig. 1.4). Three guides and a limit switch case on upper cover of the unit serve for the holder centering insight the thermocell.

Fig. 1.6. Upper electrode holder with integrated preamplifier of pA-range signal

PAY ATTENTION! Electrodes of the measuring system especially upper one are extremely sensitive to contaminations. To prevent the electrode system contamination it is recommended do not touch its copper and PTFE elements, prevent their contact with extraneous objects and surfaces. To handle the holder take it preamplifier case or thermal shield exclusively. When the holder is extracted off the thermocell, place it on its side (Fig. 1.6) or on the preamplifier case top (i.e. with the electrodes upward) (Fig 1.7 *a*). You also can use a PTFE shell from the shipment set to keep the holder safe and protect against contaminations (Fig. 1.7 *b*).



a *b*

Fig. 1.7. Safe placement of the upper electrode holder: *a* – on the preamplifier case top with the electrode upward (PTFE shell from the shipment set shown on the right); *b* – inside PTFE shell from the shipment set

Rear wall of the measuring unit (Fig. 1.8) contains cooling fan that can be used to accelerate inner element cooling when necessary (including tested sample in the thermocell). The cooling fan switch is placed on left wall of the measuring unit (Fig. 1.1 *b*). Under the cooling fan, sockets for electric connection of the unit with control electronic unit are located. Please use for this purpose corresponding cables from the instrument complete set (pay attention on the labels). Cable integrated into the measuring unit between the sockets serves for the heater voltage supply. Its plugs should be inserted into two sockets on rear wall of control electronic unit (in center) marked as «48 V» / «SAMPLE HEATING CABLE» (polarity is indifferent).

pA-Range preamplifier mounted on the upper electrode holder has own integrated electric cable that should be connected directly to the corresponding socket on rear wall of control electronic unit and labeled as ‘THERMOCURRENT MODULE’.

Fig. 1.8. Rear wall of the instrument measuring unit

ATTENTION! Switch the control electronic unit off before connecting or disconnecting any cables of the instrument. That will allow avoiding the electronic components damage.

ATTENTION! During the operation, elements of the measuring unit may become extremely hot. Temperature of the thermocell upper cover may reach 180 °C at operation with maximum available settings. Therefore in the course of experiments it is prohibited to touch the upper surface of the measuring unit especially its unpainted area.

1.4 Control electronic unit

Control electronic unit (CEU) (Fig. 1.9) provides conversion of commands from the specialized software run on host PC into final execute pulses or voltage for thermocell heater. CEU also provides reception of measured signals from pA-range preamplifier and thermosensor, their analog-to-digital conversion and further transmission to specialized software for recording. CEU additionally provides service functions (e.g. cooling fan powering). In extended version, CEU includes module for electropolarization potential application to sample in the thermocell (autonomous power supply and voltage adjusting circuit).

a

b

Fig. 1.9. Control electronic unit: a – front panel (extended version); b – rear panel

CEU contains main board controlling the instrument functions and providing connection with host PC as well as specialized power supply units for thermocell heating, sample charging (in extended version) and measuring circuits of the instrument. Fans inside the CEU case provides cooling for the mounted components.

Control electronic unit connects to the instrument measuring unit with help of two specialized cables. Plug them into corresponding sockets on rear wall of the measuring unit (Fig. 1.8) and rear panel of CEU labeled ‘CABLE 1’ and ‘CABLE 2’ (Fig. 1.9 *b*). The cable integrated in the measuring unit should be plugged into two sockets on rear wall of control electronic unit (in center) marked as «48 V» / «SAMPLE HEATING CABLE» (polarity is indifferent). The cable integrated in the preamplifier should be inserted into socket labeled as ‘THERMOCURRENT MODULE’. To connect CEU to host PC, use conventional USB (Am-Bm) cable and insert its B(m) end into socket on rear wall of control electronic unit labeled as ‘USB /B/ TO HOST COMPUTER’

CEU is powered by AC 230 V 50 Hz. Socket for power cord is on the unit rear panel. Main switch is also on rear panel of the unit. Fuse (3.15 A) seat is next to the main switch.

CAUTION! Socket of electrical outlet must have reliable grounding. If there is no grounding in the socket, you are to provide grounding for the control electronic and measuring units to prevent electric shock at the device operation.

Front panel of control electronic unit (Fig. 1.9 *a*) in extended version contains a toggle switch (‘CHARGE VOLTAGE On/Off’) that turns on/off application of electropolarization potential to the lower electrode in the measuring unit (activation of sample charging). Knob ‘CHARGE VOLTAGE Min/Max’ to the right from the toggle switch serves for adjustment of the applied voltage (in range 38…300 V). Digital voltmeter ‘CHARGE VOLTAGE x10’ to the left from the toggle switch indicates the adjusted voltage.

CAUTION! There are high voltage elements inside the control electronic unit. To avoid danger of electric shock turn the unit off and unplug the power cord before opening the control electronic unit case.

2 Control software for ST1 instrument

Instrument for measurement of thermostimulated current ST1 operates under control of specialized software **TST** run on a personal computer with OS Windows. The software package is available for download from the manufacturer site (direct link to current version http://microtm.com/download/tst.zip). To install the software, unzip downloaded file and place the resultant folder with the file package on hard drive of host computer allocated for the instrument control.

For communication of host PC with the device control electronic unit virtual COM-port drivers should be installed beforehand in the system. Download driver package from http://microtm.com/download/cdm-v2.12.28-whql-certified.zip for example or from https://www.ftdichip.com/Drivers/VCP.htm and unzip the package content on hard disk. To install drivers, connect ST1 control electronic unit to host PC with USB cable and turn on the CEU (with switch on rear panel). System will ask for drivers at maiden connection. Feed path to the unzipped driver set into the system and appropriate drivers should install automatically. After that the system will assign a virtual COM-port for ST1 device. Further, check the port settings in the TST software (see §2.4).

To start **TST** software, run file `tst.en.exe` in the package folder.

2.1 Main window

After start of **TST** software, main window appears on the screen (Fig. 2.1). At the top, the main window contains the menu bar and the toolbar below it. Area in the middle serves for showing visualization windows and other control elements used for operations with the device and measured data. At the bottom, a status line provides information about the device state and other service data (for debug purpose mainly).

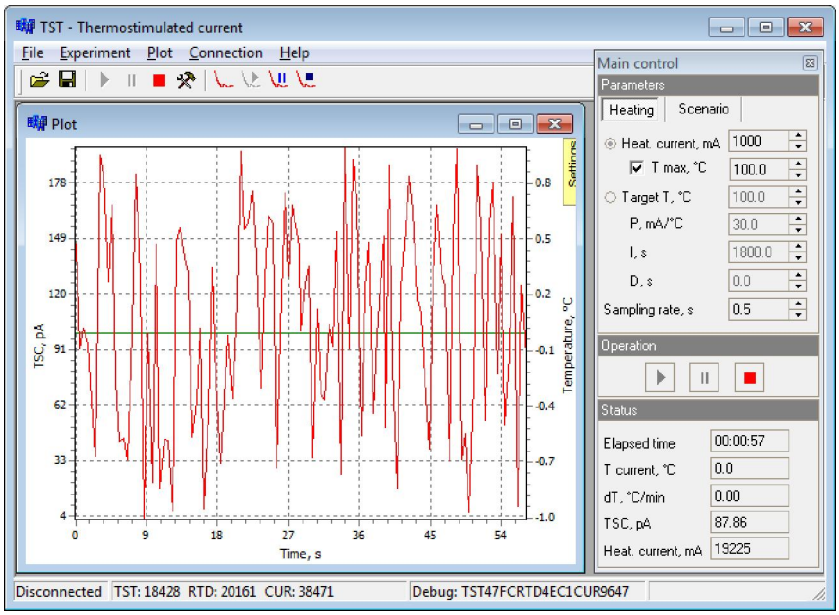
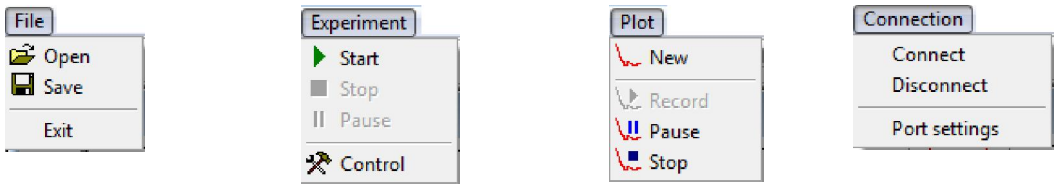


Fig. 2.1 – Main window of **TST** software. Main control panel and data plot window are activated

The program menu provides access to all control functions of the software. Menu '**File**' (Fig 2.2 *a*) contains commands for operations with data files and program exit command. Menu '**Experiment**' (Fig 2.2 *b*) provides commands for the device function control and for bringing up main control panel over the device function parameters. Menu '**Plot**' (Fig 2.2 *c*) commands over data visualization windows – opens new window and controls data plotting in it. Menu '**Connection**' (Fig 2.2 *d*) provides access to service routines like manual connection and disconnection of host PC with the device control electronic unit and activation of serial port configuration panel. In menu '**Help**' a brief description of TST software is presented under command 'About'.



a *b* *c* *d*

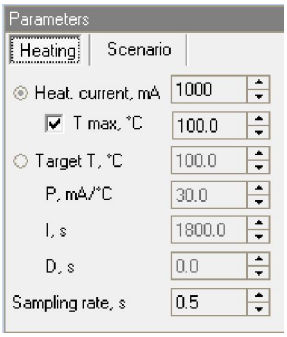
Fig. 2.2 – Items of menu 'File' (*a*), 'Experiment' (*b*),"Plot' (*c*) and 'Connection' (*d*)

Toolbar (Fig. 2.3) contains buttons for quick access to the most frequently used operations (left to right): file opening, file saving, start of the thermocell heating process, pause in heating, manual stop of the heating process, bringing heating control panel up, opening of data visualization window, staring of diagram plotting in the visualization window, pause in the diagram plotting, stopping of diagram plotting.

Fig. 2.3 – Buttons in the program toolbar

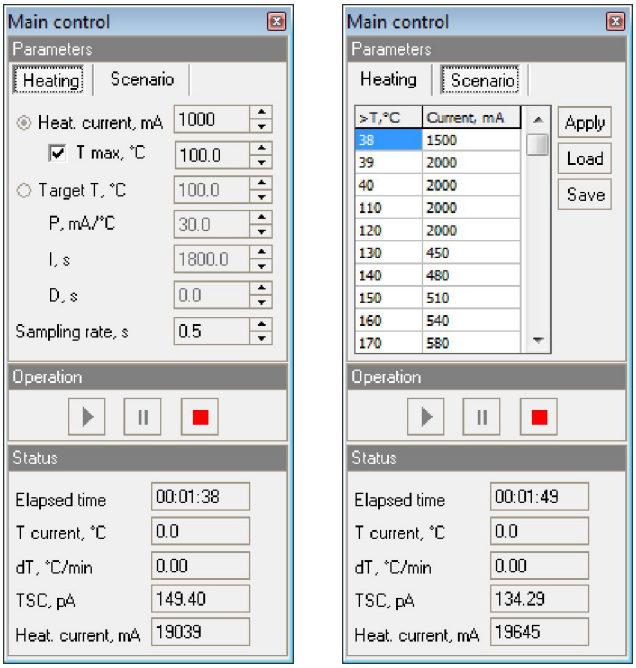
2.2 Main control panel

Virtual panel for control over the device parameters and functions (Fig. 2.4) is brought up by menu command '*Experiment > Main control*' or by pressing button  in the toolbar. Main control panel serves for setting the thermocell heating process parameters, adjustment of data visualization rate, start/pause/stop heating operation by buttons, indication of current data measured by the system.



Section 'Parameters' of the control panel has two modes switched by tabs – '**Heating**' and '**Scenario**'. Tab '**Heating**' (Fig. 2.4 *a*) joins regular parameters necessary for the heating process control. Mode '**Heat. current, mA**' provides heating by constant current set in mA in corresponding input box. Parameter '**T max, °C**' (when activated) sets maximum temperature of the thermocell after reaching which the heating process will be stopped automatically by setting heating current to 0. This mode is recommended for regular measurement of thermostimulated current due to minimum effect from the heating element on pA-range system.

Mode '**Target T, °C**' turns on feedback system for reaching and further supporting the preset temperature (typed in the input box). Under this option, you may set proportional, integrated and differential coefficients for the feedback system set in activated input boxes '**P, mA/°C**', '**I, s**' and '**D, s**', correspondingly. Correct setting of PID coefficients allows fast heating of the thermocell and graduate reaching of the target temperature with successive support of the target temperature at constant level. Such option is recommended for the sample charge operations.

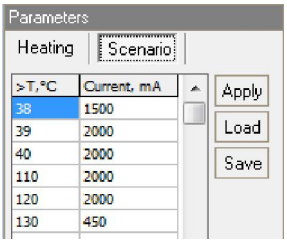


a *b*

Fig. 2.4. Main control panel with active tab 'Heating' for regular parameters (*a*) and tab 'Scenario' for setting the heating program via table (*b*).

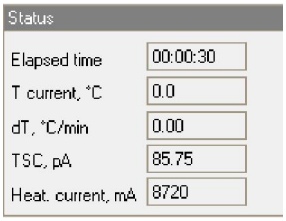
Parameter '**Sampling rate, s**' sets period of data plotting in the diagram. Minimum value is 0.5 s that equals to the data acquisition rate in the system. You may set bigger values for the sampling rate multiple of 0.5 s that will result in corresponding decrease of plotted data point in the recorded diagram.

Tab '**Scenario**' (Fig. 2.4 *b*) realizes additional option for user defined program of the heating process by typing in the table necessary heating parameters namely, the heating current corresponding to measured temperature conditions. Service buttons 'Apply', 'Load' and 'Save' to the right from the scenario table are used, correspondingly, for activation of the set heating program, loading of previously saved heating program and saving current heating program in a file on a hard drive.



Section 'Operation' contains buttons to start the thermocell heating process, to pause the heating process and to stop the heating manually, correspondingly.

Section 'Status' has indication function and shows current values for:



'**Elapsed time**' – time elapsed after start of currently performed heating process;

'**T current, °C**' – current temperature measured by the thermosensor inside lower electrode;

'**dT, °C/min**' – current heating rate calculated based on data obtained during last minute of the heating;

'**TSC, pA**' – currently measured value of thermostimulated current;

'**Heat. current, mA**' – electric current supplied to the heater at the moment. The electronic unit maximum capability is 1900 mA.

2.3 Visualization window

Start of heating process automatically brings up in main window a specialized visualization window (Fig. 2.5) plotting the acquired data in graphical form. If additional visualization window is necessary, open it by menu command ‘Plot > New’ or by pressing button  in the toolbar.

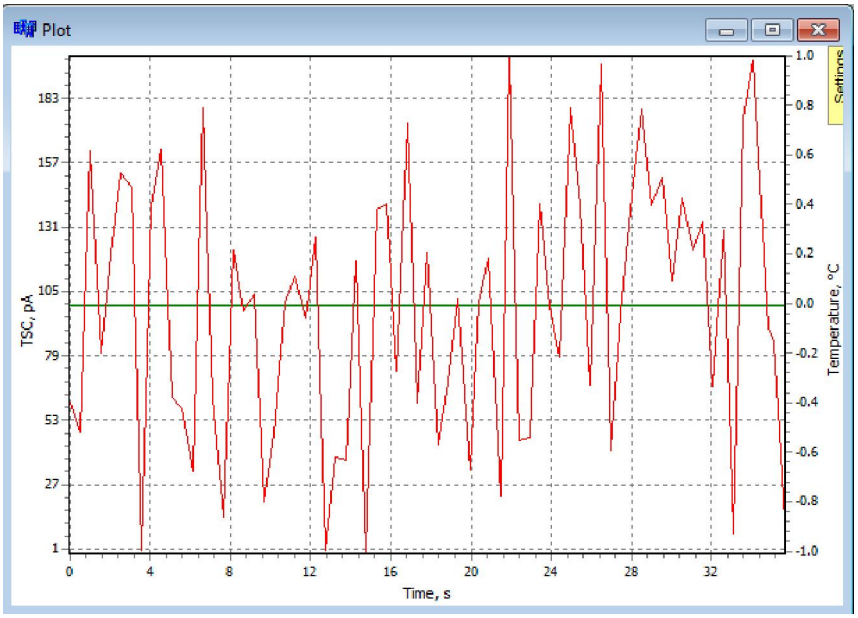


Fig. 2.5 – Example of data graphical representation in a visualization window

Vertical button Settings in top right corner of the windowy opens a panel of the plot settings (Fig. 2.6). In the plot settings panel at right boder of the window you may set:

- the plotted diagram type: TSC=f(T); TSC=f(t); T=f(t) or combined diagram TSC=f(t), T=f(t). Choose appropriate type in the selector ‘**Plot type**’;
- range on X-axis (in area X range);
- range on Y-axis (in area Y range);
- text comment that will be added to the data file. Type necessary text in area ‘Comment’.

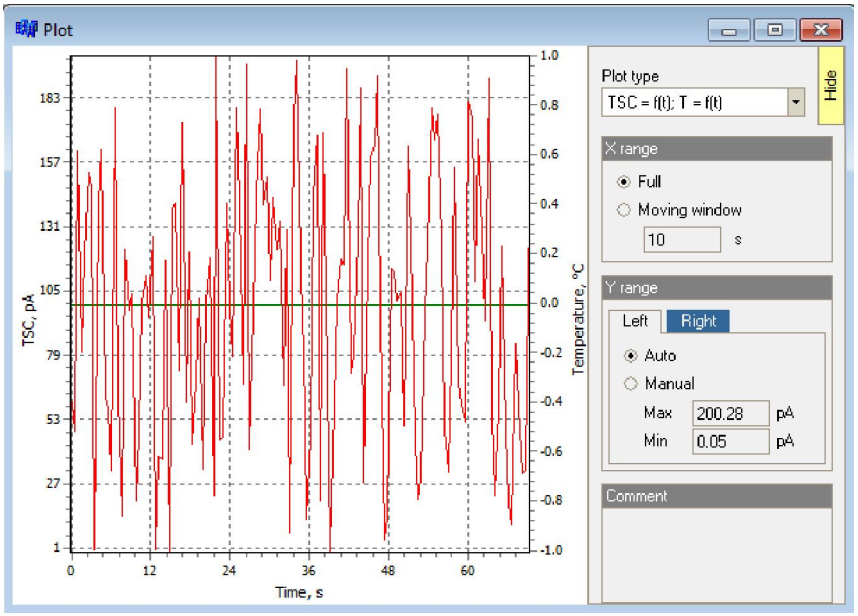


Fig. 2.6 – Окно визуализации с открытой панелью настроек

2.4 Communication menu

Menu '*Communication*' contains commands for manual connection and disconnection of host PC with the device control electronic unit and activation of serial port settings panel '**Setup**' for configuration of serial port engaged in the PC—CEU communication.

Service commands '*Connect*' and '*Disconnect*' should be used at the serial port reconfiguring.

Serial port settings panel '**Setup**' (Fig. 2.7) provides configuration of parameters defining communication process between host PC and the control electronic unit. Before working with the instrument for measurement of thermostimulated current ST1 you should correctly configure the communication parameters. To bring up serial port settings panel '**Setup**' use menu command '*Communication > Serial port settings*' (Fig. 2.2 *d*).

Communication parameters set in the panel '**Setup**' relates to the serial port in the host computer (COM-port). This port is automatically emulated in the host PC by supplied drivers when the instrument connects to it via physical USB-port and usually requires no additional adjustments in the OS control panel. Below, regular settings used in the '**Setup**' panel are cited:

Port ..... COM# (depends on PC configuration)
Baud rate 57600
Data bits 8
Stop bits 1
Parity ..... Нет
Flow control .... Нет

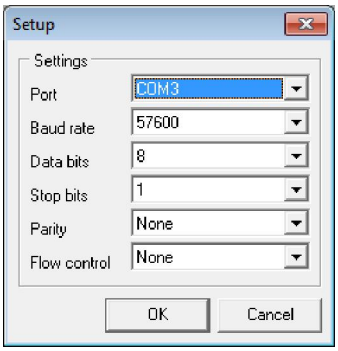


Fig. 2.7 – Serial port settings panel for configuring the host PC—CEU communication parameters

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