Thermoluminescence apparatus using PT100 resistors as the heating and sensing elements

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A novel apparatus for obtaining thermoluminescence glow curves is described. Two standard PT100 precision resistors, which have a well-known dependence of resistance on temperature, are connected back to back to provide a sensing and heating element. The resulting hot finger has very low thermal mass, is nonreactive, and is inexpensive. With dry nitrogen gas-flow cooling, an operational range of −50–450 °C is achievable. A tailored control circuit which is easily calibrated drives the heating element, and temperature ramps are implemented in software. The simple design permits the use of modularly interchangeable hot fingers for rapid measurement of many samples.
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I. INTRODUCTION

Thermoluminescence is a well-established technique which has stimulated a number of instrument designs suited for applications in radiation dosimetry, geophysics, archaeology and biology.[1–7] The crucial element of all thermoluminescence instruments is the heating element, typically a planchet or block heater. While the planchet heater has the advantage of a fast thermal response, both planchet and block heaters present design challenges in obtaining uniform temperature distribution across the sample and, as the temperature sensor is not in direct contact with the sample, precise sample temperature measurements during the heating cycle.[4] Commercial thermoluminescence instruments offer good performance for typical applications in dosimetry but are not necessarily appropriate in design or cost for fundamental research.

Faced with exactly these problems in measuring the thermoluminescence glow curves of x-ray storage phosphors, we developed a high-performance cost-effective thermoluminescence rig based around standard PT100 precision resistors as the heating and sensing elements. The resulting apparatus is driven by a hardware proportional-integral controller with the thermal ramp controlled in software by computer. The instrument also permits the measurement of x-ray induced photoluminescence at an arbitrary temperature within the operational range of −50–450 °C during sample irradiation.

Measurement from temperatures below room temperature was a specific requirement of the design because determination of the thermoluminescence properties of x-ray storage phosphors at ambient temperature are often complicated by afterglow following irradiation. The afterglow originates from shallow traps which are of interest in themselves. Irradiation at, and measurement of glow curves from, room temperature cannot yield consistent measurements of low-lying traps. Reducing the sample temperature before irradiation and measurement permits characterization of these traps.

II. INSTRUMENTATION

The thermoluminescence rig is comprised of three elements: the heating and temperature sensing hardware, the detectors used to measure the luminescence, and a computer to control and record the thermoluminescence glow curves in software. A system diagram is shown in Fig. 1, and the components are described below.

A. Sensing and heating element

The heater and sensor are miniature PT100 resistors of dimensions (width × length × height) $2 \times 5 \times 1$ mm^3, which are combined into a single interchangeable hot finger, shown in Fig. 2. The sample is placed on top of the sensor, which gives 10 mm^2 maximum available sample area. The relatively small sample area poses no significant problems for the measurement of storage phosphor materials, which typically exhibit strong thermoluminescence signals after x-ray irradiation. Silicone oil is used to ensure good thermal contact between the sample and the sensor; both powdered and solid samples may be mounted in this manner. This configuration possesses the distinct advantage that the sample and temperature sensor are in direct thermal contact, unlike the typical planchet heater in which a thermocouple is spot welded to the bottom of the heating element and may not be

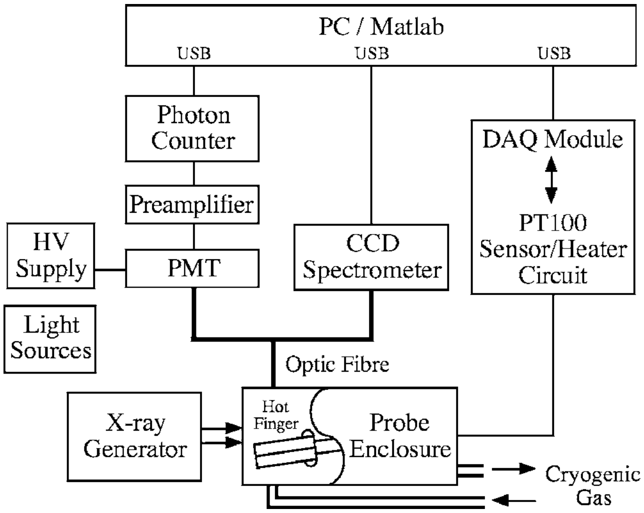

FIG. 1. System diagram of the thermoluminescence rig.

at the same temperature as the sample due to thermal gradients and inhomogeneity across the sensor-heater-sample interfaces.

The hot finger is plugged into a simple mount and placed within a holder which is designed specifically for use on a Philips PW 1720 x-ray generator. The holder also provides an enclosed area with inlet and exhaust apertures to allow cooled nitrogen gas to be circulated over the heater-sensor head. Nitrogen gas is obtained from a high purity gas cylinder and passed through a copper coil which is immersed in precooled isopropyl alcohol. The cooled gas is introduced to the heater-sensor head through the sample holder gas inlet. The gas flow also functions to reduce the hydration of hygroscopic samples.

With this configuration, static temperatures as low as −50 °C are easily obtainable. The maximum temperature is dictated by both the choice of power supply and the hot finger thermal dissipation at high temperatures. With a little over 10 W power delivered to the heater, a maximum temperature greater than 450 °C was achievable, giving an operational range of 500° between −50 and 450 °C. Over this range, the PT100 sensor resistance changes by 183.87 Ω, from 80.31 Ω at −50 °C to 264.18 Ω at 450 °C. Due to the low thermal mass of the PT100 resistors, the system exhibits fast thermal response, taking less than 90 s to cool from 450 to 30 °C without gas-flow cooling.

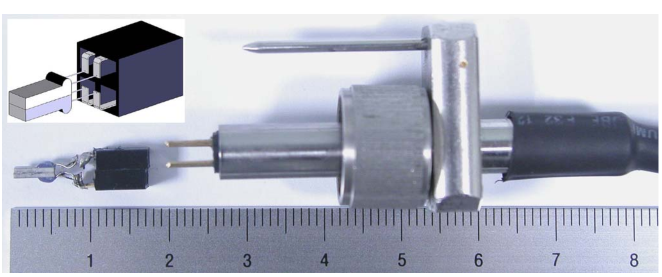

FIG. 2. (Color online) PT100-based hot finger configuration. Two PT100 resistors are glued back to back with thermally conductive paint and soldered to modified 2.54 mm (0.1 in.) pitch jumper connector blocks for use with the heater-sensor mount. The mount secures the hot finger for irradiation with the x-ray generator. Inset: Schematic of the interchangeable PT100 hot finger.

B. Digital acquisition unit

A digital acquisition unit (DAQ) provides the necessary interface between the computer and the heater-sensor hardware, to read the voltage drop across temperature sensor and write the temperature set point voltage for the heater. The choice of DAQ influences some aspects of the sensor-heater circuit design, as noted in the following section.

A National Instruments USB-6900 DAQ was selected as a compact, readily available, and cost-effective DAQ with up to eight analog inputs. The temperature sensor resolution obtained with this DAQ varies from 0.028 to 0.033 °C/bit between −50 and 450 °C. The remaining analog input ports of the DAQ are connected to read the voltage at test points in the circuit for the purpose of self-diagnostic tests in software.

The set point voltage corresponding to the desired temperature, provided by the DAQ's analog output, gives a set point temperature resolution of 0.1 °C over the operational range.

C. Sensor-heater circuit

Thermal control is provided by a calibrated proportional-integral circuit which compares the difference between a temperature set point, generated in software and provided from the analog output port of the DAQ, and the temperature of the PT100 sensor. The difference controls a 30 V power supply connected to the PT100 heater element.

Figure 3 shows the circuit diagram for the sensor-heater hardware. The 2.5 V precision reference voltage from the DAQ, buffered through the U9 voltage follower, is used in the three branches of the circuit. From top to bottom these are (a) the sensor driving branch, (b) the heater driving branch, and (c) the sensor DAQ measurement branch.

1. Sensor driving branch

This part of the circuit is responsible for temperature measurement using the PT100 sensor resistor. The inverting amplifier U7 places a constant current of 2.083 mA (2.5 V/1.2 kΩ) through the PT100 sensor, which is connected as the feedback resistor across U7. Using an estimated dissipation constant of 0.25 °C/mW for the PT100 resistor pair in the sample mount in air,[8] the self-heating due to the sensor current is 0.05 °C at −50 °C and 0.16 °C at 450 °C.

The voltage drop across the PT100 sensor is measured by operational amplifiers (op-amps) U4, U5, and U8 configured as a standard differencing amplifier[9] with unity gain, the output of which, at test point 1 (TP1), is provided to the heating and DAQ measurement branches of the circuit. Separate sensor current and voltage wires are used to eliminate stray resistances from the cabling as much as possible. The final op-amp in the sensor driving branch, U6, is configured as an inverting amplifier with a gain of 10/2.083=4.8, thus scaling the sensor voltage such that 1 V corresponds to a sensor resistance of 100 Ω. This voltage is made accessible

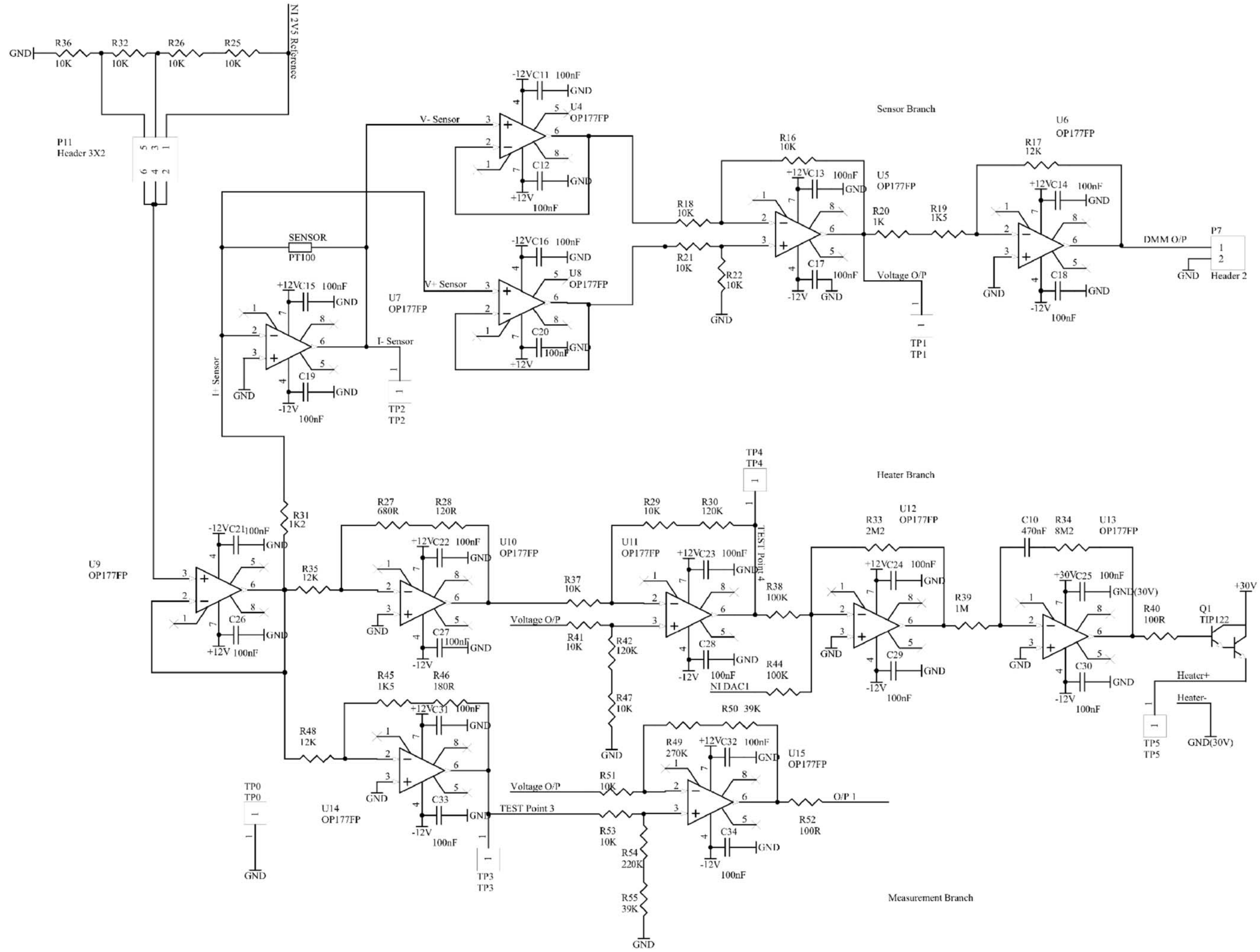

FIG. 3. Circuit diagram of the heater driver and temperature sensor module.

external to the unit at the digital multimeter output (DO/P) port shown in Fig. 3, permitting independent direct reading of the PT100 resistance.

2. Heater driving branch

Thermal control of the sensor and sample are provided by the heater branch, which measures the difference between the sensor voltage and the set point voltage from the DAQ unit's analog output port and sets the heater voltage as appropriate.

Op-amps U10 and U11 convert the sensor voltage to lie within the range 0 to -5 V for comparison with the 0–5 V analog output from the DAQ. U10 provides a reference offset voltage equal to the voltage drop across the sensor at -50 °C. This is is compared with the sensor voltage obtained from the sensor branch by op-amp U11, configured as a differential amplifier of gain 13, thus giving an output voltage at test point 4 (TP4) which is the sensor voltage scaled to the range 0 to -5 V. Op-amp U12 is configured as a summing amplifier of gain 22, the inputs to which are the scaled sensor voltage and the DAQ analog output voltage representing the temperature set point; when the sensor temperature and the set point are the same, the output of U12 is 0 V.

Combined proportional-integral control is provided by U13, with the 0.47 s time constant in the feedback loop chosen based on the experimentally determined response time of the sensor head. For ramp rates up to 5 °C/s, overshoot and ringing due to the lack of differential control is less than ±0.05 °C (see Sec. III).

The proportional-integral output voltage drives a TIP122 Darlington transistor Q1 which acts as a power amplifier to control the heater current. The heater power supply provides +30 V maximum across the PT100 heater resistor, giving a maximum current around 370 mA and maximum power just under 11 W. A separate ground is used for the heater power supply to avoid the problem of ground loops superimposing a signal from the heater on the temperature sensor voltage.

3. DAQ measurement branch

This branch, comprised of op-amps U14 and U15, is used to offset and scale the sensor voltage for connection to the DAQ analog input. The inverting amplifier U14 produces an offset reference voltage which is compared to the sensor voltage from the sensor branch by the differential amplifier U15. The differential amplifier gain gives an output voltage

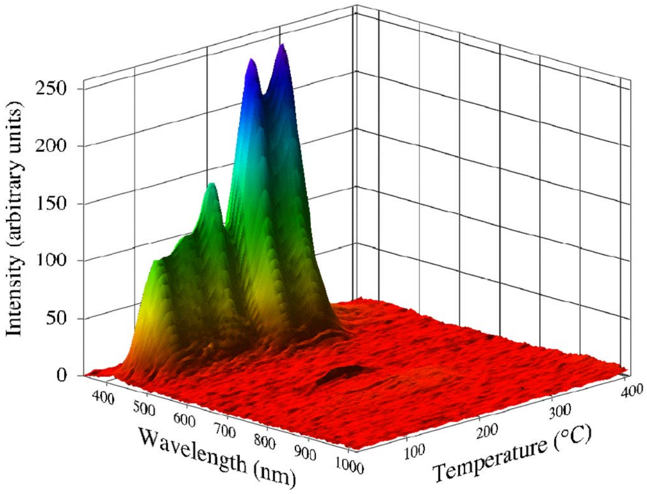

FIG. 4. (Color online) The thermoluminescence glow surface of the x-ray storage phosphor $BaMgF_4:Eu^{2+}$ obtained with the USB2000 spectrometer.

range −5–+5 V for the temperature range −50–450 °C. This voltage is connected directly to the first analog input of the DAQ for readout by the software.

D. Optical detectors

Two detectors were used with the hot finger: a photomultiplier tube (PMT) and a charge-coupled device (CCD) spectrometer. In our case, the PMT used was a Peltier-cooled Electron Tubes Limited 52 mm 9558QB PMT with a Stanford SR445A preamplifier and a Stanford SR400 two-channel gated photon counter, while the CCD spectrometer was an Ocean Optics USB2000 optimized for fluorescence measurements. The photomultiplier possesses excellent sensitivity over a wide wavelength range but provides only a wavelength-integrated measurement. Wideband filters were used with the PMT to remove unwanted signal due to blackbody radiation at high temperatures. In contrast, the CCD provides a wavelength-resolved measurement over 340–1025 nm at a resolution (full width at half maximum) of 8 nm but is about 2000 times less sensitive than the PMT. A typical result obtained with the USB2000, from an x-ray storage phosphor $BaMgF_4:Eu^{2+}$ after x-ray irradiation at 50 kV/20 mA, is shown in Fig. 4.

An optic fiber was used to pipe the thermoluminescence signal to the detectors and permits the interchangeable use of one or, with the use of a bifurcated fiber, both detectors simultaneously. It also provides a desirable thermal and chemical isolation between the detectors and the sample. Typically, a single B&W Tek FPC Series fiber patch cord of length 1.5 m, diameter 1 mm, and numerical aperture 0.22 (field of view 25°) and rated for UV was used with the system. The optimal fiber-to-sample distances for sample extents of 3–5 mm are thus 7–11 mm. Similar to previously described thermoluminescence apparatus using optic fiber coupling,[5] there is no net advantage to using coupling optics between the sample and the fiber.

E. Photostimulation and photobleaching capability

The bifurcated optic fiber also permits the optical connection of a light source to the sample for either photostimulated luminescence measurements or photobleaching. This system was tested with two types of light source: high-intensity light emitting diodes (LEDs) and a miniature 1/4 m focal length monochromator with a high-intensity incandescent light source. Philips Luxeon high-power LEDs of wavelengths 470, 530, and 625 nm and spectral width at half peak maximum of 20–35 nm (Ref. 10) were typically used for selective trap photobleaching.

The measurement of photostimulated luminescence is possible with either of the PMT or CCD detectors, with a suitable filter at the detector end of the fiber necessary to remove the stimulation signal. Photobleaching does not require simultaneous detection, and there is no requirement for optic filtering at the detector.

F. Control software

Software running on a standard personal computer (PC) controls the heating ramps and measurement of the sample temperature and thermoluminescence. The routines were implemented in MATLAB, but there is no reason why the software could not be implemented in a lower-level compiled language. The USB-6009 DAQ controlling the heater-sensor hardware was connected via the universal serial bus (USB) interface, while the Stanford SR400 photon counter and Ocean Optics USB2000 spectrometer were controlled via serial interfaces connected to the PC with USB-to-serial converters; the instruments were addressed in software by use of the NI-DAQmx drivers and the serial interface driver provided by the MATLAB Instrument Control Toolbox.

Controlling the temperature ramp from software provides significant advantages in the flexibility of the measurement and in a simpler sensor-heater circuit than a microcontroller-based system. A drawback of this approach is that the relatively slow execution speed and single-threaded execution of the MATLAB routines pose problems in the production of smooth temperature ramps. These problems were addressed by updating the temperature set point from a MATLAB timer function and independently acquiring data from the PMT and CCD detectors. All operations run asynchronously. The temperature set point function may be called at any arbitrary interval; 0.1 s—a temperature update frequency of 10 Hz—was found to be an adequate update rate for temperature ramps up to 5 °C/s, the maximum ramp rate tested. The temperature ramp set point is calculated adaptively based on the time of update execution as the period between executions cannot be guaranteed to match the desired period due to MATLAB's single-thread only execution environment.

Steady-state temperatures initially achieved for a given set point were offset by up to −1–+1 °C over the operational temperature range of −50–450 °C due to a minor residual nonlinearity of the instrument. A quasi-steady-state calibration curve was obtained by running a temperature ramp at a very slow ramp rate and calculating the deviation from the set point. A cubic-spline interpolation of this cali-

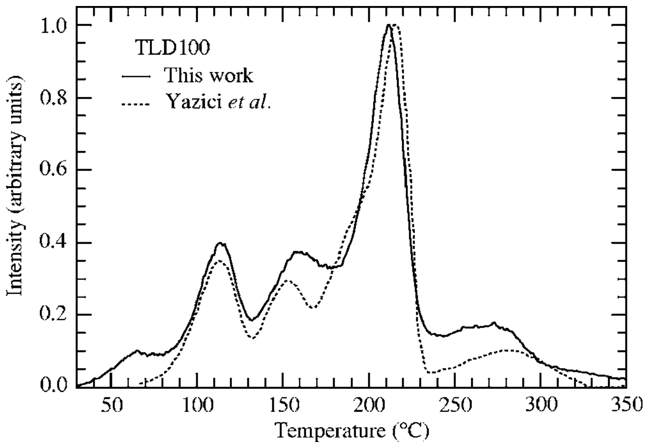

FIG. 5. A glow curve obtained from TLD100 (black line) compared with a glow curve measured by Yazici *et al.* (Ref. 11).

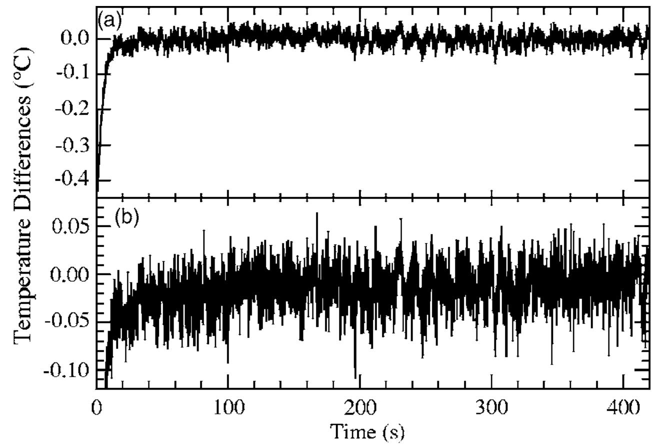

FIG. 6. (a) The residual difference between the temperature ramp and a linear fit. (b) The deviation of the sensor temperature from the previous set point temperature.

bration curve was then used as a lookup table to apply an appropriate offset to the set point in software. The calibrated steady-state deviation from the desired temperature was found to be better than ±0.05 °C.

The routines were written to be modular, permitting their use in MATLAB scripts. Experimental profiles involving heating or cooling to specific temperatures, holding the sample temperature constant for an arbitrary time, waiting for the thermoluminescence signal to fall below a specific level, as well as repeated temperature ramps with measurement, are thus easily constructed. Similar to some previous instrument designs,[6,7] the production of arbitrary temperature ramps for more sophisticated glow curve deconvolution, such as the fractional glow technique, is also possible.

III. DISCUSSION

To ensure that the instrument provides an accurate measurement of thermoluminescence glow curves, the comparison of glow curves measured from a standard thermoluminescence dosimetry material with previously published curves is advisable and in this respect the LiF-based TLD-100 dosimetry material is a natural choice. A glow curve obtained from a powdered sample of TLD-100 after irradiation with x rays, generated at 50 kV/20 mA and filtered through the 0.65 mm aluminum front face of the probe mount, is shown in Fig. 5. This curve shows good agreement with a TLD-100 glow curve measured over a wavelength range of 410–430 nm with an optical filter by Yazici *et al.*[11] The most intense TLD-100 peak at 215 °C coincides within 2 °C, the others within 5–7 °C, which is reasonable agreement given the differences in sample annealing, irradiation (x ray versus beta ray), and glow curve measurement (wavelength integrated versus filtered for maximum transmittance at 420 nm). A thin solid sample of TLD-100 was also measured, and the same glow curve was obtained.

It must be noted that the PT100 resistor itself exhibits a thermoluminescence signal, which is visible in Fig. 4 as a weak peak, narrow in wavelength and broad in temperature, appearing just below 700 nm between 100 and 200 °C. This weak instrumental artifact poses little problem in practice and, if necessary, can be completely removed by an appropriate optical filter or by coating the PT100 with a thin layer of thermally conductive black paint, without adversely affecting the desired thermoluminescence.

The linear ramp performance of the instrument is shown in Fig. 6 for a 1 °C/s ramp from 30 to 450 °C. The detailed performance may be seen in the residual from a linear fit to the data between 20 and 420 s (50 and 450 °C) and the temperature deviation from the previous set point measured at the time the set point is updated (every 0.1 s for this measurement). The sensor temperature initially lags the set point, due first to the unavoidable response time of the system and subsequently to the lack of differential control in hardware, but decreases to below −0.1 °C within 8 s, corresponding to a sublinear ramp temperature interval from 30–38 °C. The slope of the linear fit gives the average ramp rate and was found to be $1 \pm 5 \times 10^{-6}$ °C/s, while the residual of the linear fit shows an average deviation of only 0.02 °C. Figure 6(b), the deviation from the set point, gives the instantaneous deviation of the sensor temperature from the linear set point ramp. After the initial sublinear response, the linear ramp achieved falls below the ideal 1 °C/s linear ramp by −0.01 °C on average. This constant deviation is predominantly a function of the ramp rate, arising from the system response time, and averages −0.1 °C for a 5 °C/s ramp. The ramp performance of our system thus compares very favorably to that of conventional heater systems.[4–7]

For ramp rates greater than 1 °C/s, where the sublinear deviation progressively dominates the performance, a factor of 3 reduction of the deviation magnitude was easily obtained by adding an empirically determined offset to the temperature set point at each update. The flexibility of the design also permits the use of the voltages from the sensor and diagnostic test points to provide more sophisticated manipulation of the instrument's performance.

IV. CONCLUSION

A high-performance instrument for measuring thermoluminescence glow curves has been developed, based around standard PT100 resistors as both sensing and heating elements. The linear ramp performance of the instrument is excellent, with typical average linear ramp rates within a few parts in 10^6 of the desired rate and a deviation of less than 0.05 °C (less than 0.1%) over the majority of the ramp. While this particular instrument has been optimized for the measurement of thermoluminescence from x-ray storage phosphors, other materials which exhibit thermal or optically stimulated luminescence and may be suitably prepared for mounting on the hot finger can also be measured. The apparatus thus might also be usefully applied to the measurement of samples in the fields of geophysics and archaeology.

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