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## **THERMOLUMINESCENT SYSTEM FOR LOW TEMPERATURES\***

Luiz Antonio Ribeiro da Rosa<sup>1</sup>, Linda V. Ehlin Caldas and Nery Gejuiba Leite<sup>2</sup>

### **ABSTRACT**

A system for measurements of the thermoluminescent glow curve, the thermoluminescent emission spectrum and the optical absorption spectrum of solid samples, from liquid nitrogen temperature up to 473 K, is reported. A specially designed temperature programmer provides a linear heating of the sample at a wide range of selectable heating rates, as also long term steady-state temperatures for annealing and isothermal decay studies. The system operates at a pressure of  $1.33 \times 10^{-3}$  Pa. Presently it is being used for lithium fluoride low temperature thermoluminescent studies.

### **SISTEMA TERMOLUMINESCENTE PARA BAIAS TEMPERATURAS**

### **RESUMO**

Um sistema capaz de determinar a curva de emissão termoluminescente, o espectro de emissão termoluminescente e o espectro de absorção óptica de amostras sólidas, desde a temperatura do nitrogênio líquido até 473 K, é descrito. Um programador de temperatura, especialmente projetado, permite que se varie linearmente a temperatura da amostra a diferentes taxas de aquecimento, bem como possibilita mantê-la a uma temperatura constante para tratamentos térmicos ou estudos de decaimento isotérmico. O sistema opera a uma pressão de  $1,33 \times 10^{-3}$  Pa. Atualmente, ele está sendo utilizado para o estudo da termoluminescência do fluoreto de lítio a baixas temperaturas.

### **INTRODUCTION**

The research and application of the thermoluminescence (TL) phenomenon has considerably increased during the last two decades<sup>(8-11,15-18)</sup>. The wide-spread use of TL phosphors in personnel dosimetry, environmental dosimetry, dating, diagnostic radiology and radiotherapy has resulted in

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the development of a large number of TL instruments for routine thermoluminescent dosimetry (TLD). Julius<sup>(6)</sup> summarized the salient features of some of the well known commercial TL readers and Oberhofer<sup>(13)</sup> described several additional necessary accessories for TLD. Some commercial TL systems include automation to a great extent and utilize microprocessor based electronics and on line computers to handle very large numbers of samples expeditiously<sup>(3,6,12)</sup>. These instruments, however, have little use in experiments that are designed to understand the basic mechanisms of TL processes. For example, investigations at low temperatures are necessary in order to suppress ionic mobilities and observe the role of electronic processes explicitly<sup>(1)</sup>. Instruments that are able to carry out these investigations are not available commercially, although some researchers have developed systems with this capability for their own studies<sup>(1,2,4,5,7,14)</sup>. Additionally, though thermoluminescence indeed is a very interesting technique for solid state investigations, it cannot give conclusive data by itself. It is only through measurements of other correlated parameters that it is possible to exploit the extreme sensitivity of TL process to investigate and understand many obscure solid state phenomena. For example, the role played by color centers in the TL process of some alkali halides can only be achieved when measurements of the TL glow curve, the TL emission spectrum and the optical absorption (OA) spectrum are carried out. The present work describes a system which is intended to be able to carry out such measurements with solid samples in the temperature range 77-473 K. It was developed to initially study the TL mechanisms presented by dosimetric lithium fluoride, LiF:Mg,Ti, from liquid nitrogen temperature up to room-temperature.

#### **DESCRIPTION OF THE SYSTEM**

The system is basically developed around two interchangeable cryostat models and a high vacuum system. This one is composed of an oil vapour diffusion pump, model E02, backed by a high speed rotary pump, model ED50,

both manufactured by Edwards Vacuum Components, England, and enables a vacuum of  $1.33 \times 10^{-3}$  Pa to be maintained inside the cryostat sample chamber.

For TL glow curve and TL emission spectrum measurements, a HNDT-5 model cryostat is utilized. This cryostat was manufactured by Minnesota Valley Engineering, USA. Its sample chamber has three windows, namely two quartz windows and one aluminium window. Figure 1 presents a scheme of the cryostat and its sample chamber. The sample holder is fixed inside the sample chamber maintaining an angle of  $45^{\circ}$  with the aluminium window and one of the quartz windows. Through the aluminium window the sample irradiation is carried out. For TL glow curve determination, a photomultiplier tube (PMT) is connected to the quartz window that forms an angle of  $45^{\circ}$  with the sample holder and is used to detect the TL light displayed by the heated sample (Figure 1). The second quartz window is covered with a copper plate in order to prevent the access of spurious light into the sample chamber. For TL emission spectrum measurements, the photomultiplier tube is replaced by a monochromator to which the same PMT is connected. In order to obtain the emission spectrum of a TL glow peak, the sample temperature is maintained stable at a value between the initial and the maximum glow peak temperatures, while the monochromator analyzes the light displayed by the sample at a constant wavelength rate, within the desired wavelength range (Figure 1).

For optical absorption studies, a HNDT-3 model cryostat is utilized. This cryostat was manufactured by Minnesota Valley Engineering, USA, as well. Its sample chamber has four windows, namely two parallel aluminium windows, used for sample irradiations, and two parallel quartz windows. Figure 2 presents a scheme of the cryostat and its sample chamber. The sample holder can be placed parallel to the quartz windows or to the aluminium windows by means of a mechanism that enables the rotation of the sample chamber. An ultra-violet (UV) radiation high pressure mercury lamp SR-200, manufactured by Bausch & Lomb, USA, is connected to one of the quartz windows and is used to illuminate the sample. A GM 200 double

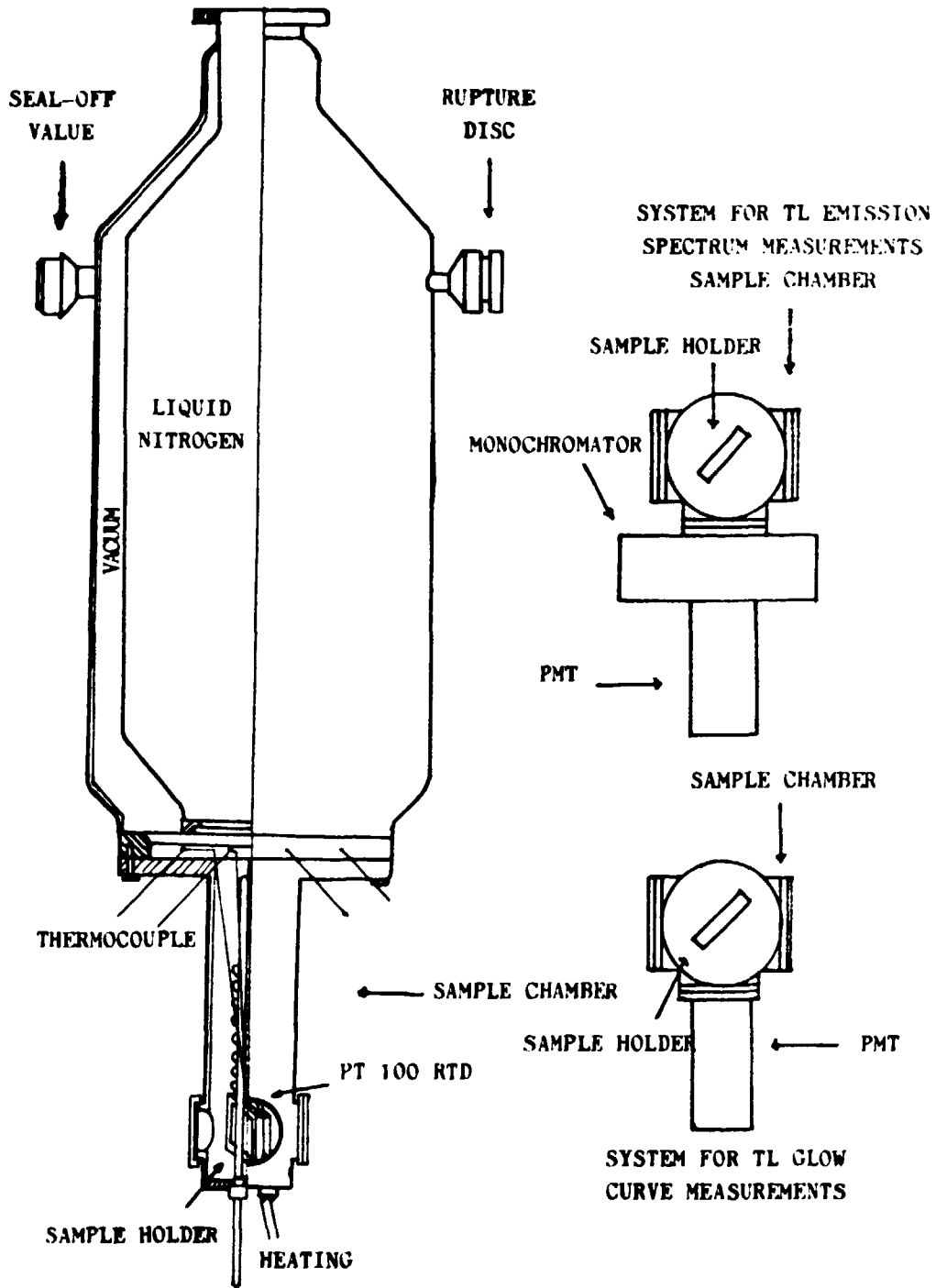


FIGURE 1 : Scheme of the cryostat HNDT-5 and its sample chamber

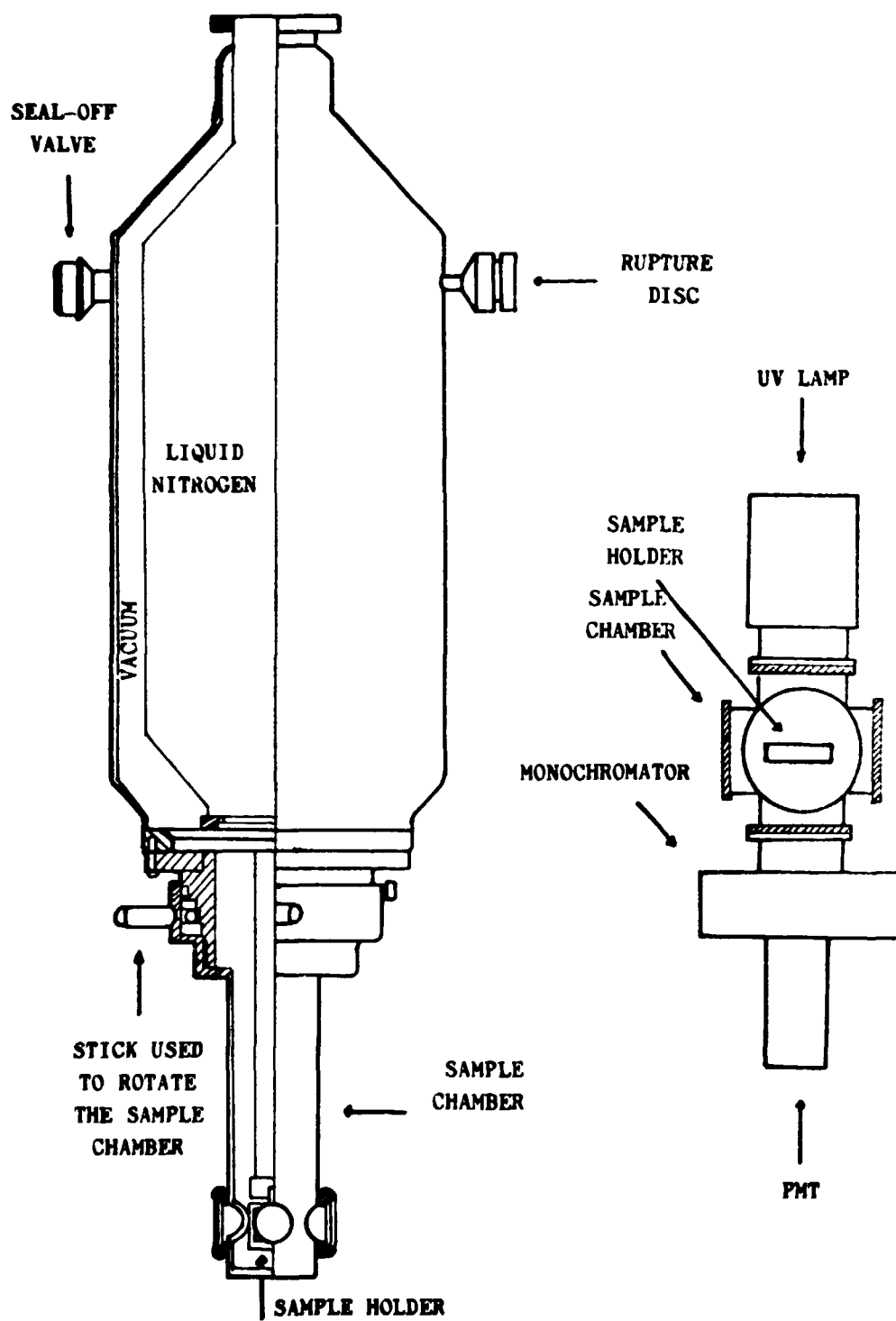


FIGURE 2 : Scheme of the cryostat HNDT-3 and its sample chamber



grating monochromator, manufactured by Kratos Schoeffel Instruments, USA, is connected to the other quartz window and is used to analyze the light that crosses the sample (Figure 2). A photomultiplier tube is connected to this monochromator. The GM 200 double grating monochromator is also utilized for TL emission spectrum measurements.

A photomultiplier tube model EMI 6256, USA, is used for light detection. It is fed by a TECTROL TCH 3000 model high voltage (HV) power supply, Brazil, and is connected to a Keithley 610C electrometer, USA, which amplifies the PMT signal to be sent to a graphic recorder.

The sample is irradiated with X-rays. They are generated by a Rigaku Denki X-ray machine (60 kV), Japan.

The sample holder is heated by means of an electric resistance and its temperature is measured with an iron-constantan thermocouple. The thermocouple measuring junction is placed very close to the sample in order to be obtained an accurate determination of its temperature.

A specially designed temperature programmer provides a linear heating of the sample at a wide range of selectable heating rates. It is also able to maintain its temperature stable for a desired period of time. Therefore, the sample annealing procedure can be carried out inside the own sample chamber and isothermal decay studies may also be executed. The temperature programmer was manufactured by S & E Instrumentos de Testes e Medição Ltda, Brazil. It uses as temperature monitor a platinum Resistance Temperature Detector (RTD). This monitor is based on the fact that the resistivity of metals shows a marked temperature dependence. In the present case the monitor is a PT 100, this means, a platinum RTD which has a resistance of 100 ohms at 0°C. The PT 100 RTD is also placed very close to the sample in order to measure its temperature accurately.

## CONCLUSIONS

The above described thermoluminescent system for low temperature investigations is presently under tests. The TL properties of single

crystal dosimetric lithium fluoride samples, LiF:Mg,Ti (TLD-100), are being studied from liquid nitrogen temperature up to room-temperature. The initial results seems to indicate that the system presents a reasonable performance.

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