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Temperature measurement based on photoluminescence of Er^{3+} doped $Sr_{0.3}Cd_{0.7}F_2$ microcrystal coupled to scanning thermal microscopy

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ABSTRACT

Rare earth doped sub-micrometric luminescent materials are promising candidates for temperature sensing and play an efficient role in many technological fields. In this paper, a new optical sensor is developed for measuring local temperatures. This sensor is based on a thermal-resistive probe and on photoluminescence of a luminescent fluoride microcrystal. The final purpose is to develop a device calibrated in temperature and capable of acquiring images of local temperature at sub-micrometric scale. Indeed, the sensor temperature can be obtained in two distinct ways: one from the thermal probe parameters and the other from the green photoluminescence generated in the anti-Stokes mode by the active Er ions directly excited by a red laser. The thermal probe is based on Wollaston wire whose thermal-resistive element is in platinum/rhodium. Its temperature is estimated from the probe electrical characteristics and a modeling. A microcrystal of $Sr_{0.3}Cd_{0.7}F_2$: Er^{3+}(4%)–Yb^{3+}(6%) of about 25 μm in diameter is glued at the probe extremity. This luminescent material has the particularity to give a green emission spectrum with intensities sensitive to small temperature variations. Using the fluorescence intensity ratio (FIR) technique, the crystal temperature is estimated from the intensity measurements at green wavelengths 522, 540 and 549 nm by taking advantage of particular optical properties due to the crystalline nature of $Sr_{0.3}Cd_{0.7}F_2$: Er^{3+}–Yb^{3+}. The microcrystal temperature is then assessed as a function of electric current in the thermal probe by applying the Boltzmann's equations. The coupling of the scanning thermal microscope (SThM) with the photoluminescence probe reveals that the particle fluorescence signal is affected by the temperature rise of an electrical microsystem submitted to a Joule heating. The first results are presented and discussed.

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1. Introduction

Enormous interests in temperature sensors have been emerged during the two last decades because temperature is one of the most fundamental physical parameters that we must know in many fields of science and technology. Measurement techniques of this important physical quantity continue to grow especially on a small scale. For many applications, it is necessary to be able to determine the local temperature and even to map temperature variations at sub-micrometer scale. For example, knowledge of thermal behavior via knowledge of the heating of micro-components is vital in microelectronics. In microscopy, the proposed methods are based on various sensors such as the thermocouple, the composite probe, the carbon nanotube thermometer or the luminescent temperature sensor [1]. The measurement and imaging of temperature at submicroscopic scale is a sensitive topic because it addresses the notion of calibration, a subject that still requires investigation today [2]. Optical temperature sensing using the fluorescence intensity ratio (FIR) technique has been the subject of many research projects [3–6]. This sensing technique is based on the temperature dependence of visible luminescence intensity obtained from closely spaced energy levels of rare earth ions inserted in micro-nano sized materials. Such materials are present, generally, in the form of fluorides since they have low phonon energies which promote radiative emissions with higher luminescence efficiencies.

In this work, we propose the development of a device for measuring local temperature and sub-micron scale imaging by coupling two techniques, photoluminescence of Er^{3+} ions doped $Sr_{0.3}Cd_{0.7}F_2$ luminescent microcrystal and Scanning Thermal Microscopy (SThM). The temperature sensor is a probe based on Wollaston wire, whose thermo-sensitive element is in rhodium–platinum. The luminescent microcrystal is stuck at the end of this probe. In literature, the Wollaston probe without microcrystal is

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not used for application in which the components temperature is measured due to its poor sensibility. The thermal probe equipped of a microcrystal makes possible the detection of local temperature grounded on calibration. So, this probe can be used as a sensor for thermal conductivity or as a temperature sensor.

For the thermal probe as temperature sensor, we will then evaluate the probe temperature in two different ways, the one obtained from the electrical circuit's components and the temperature sensor's parameters [7,8] and the one obtained from the intensity variations of the microcrystal's photoluminescence induced by the heating. The green emitting levels of Er^{3+} ions ($^2H_{11/2}$ and $^4S_{3/2}$ multiplets) are especially adequate for this purpose. We, first, expose the experimental device and then proceed to the temperature measurement by the two techniques followed by comparison of results.

2. Experimental procedure

The experimental setup is presented in Fig. 1. It consists of a thermal probe on which a fluorescent microcrystal is fixed. This probe is placed on an optical excitation device, mainly, composed by a laser excitation source, a monochromator and an optical detection system. The thermal probe is a Wollaston thermo-resistive probe usually used in scanning thermal microscopy [9].

The optical excitation part is performed using a tunable dye laser able to operate at different powers and different wavelengths. The exciting beam is set to a wavelength of 652 nm (red) and is modulated at 7 Hz frequency. The fluorescence from the active particle is focused onto the entrance slit of a Jobin–Yvon M25 monochromator by a set of lens inserting a filter which cuts the scattered photons of the laser source. This monochromator is equipped on its exit slit by a Hamamatsu R928 photomultiplier of which output signal is sent to a PAR 121 lock-in amplifier and a synchronous recorder.

The luminescent phosphor is a microcrystal of $Sr_{0.3}Cd_{0.7}F_2$ co-doped with Er^{3+} and Yb^{3+} ions glued at the probe apex on Pt/Rh thermo-resistive filament. When excited in the red range, this microcrystal exhibits a green emission spectrum with sharp lines whose intensities are sensitive to small changes in temperature. The Yb ions are usually used to excite the Er^{3+} ions because the $2F_{5/2}$ excited multiplet corresponds to $4I_{11/2}$ multiplet allowing an energy transfer from the Yb ion to the Er ion. One can also obtain the green emission by laser excitation at a wavelength of 974.4 nm of Yb ion. In our work, the Yb ions don't participate in the process of green emission due to excitation laser at 652 nm. This laser wavelength excites directly Er^{3+} ions. We used this co-doped crystal ($Sr_{0.3}Cd_{0.7}F_2$: Er^{3+}(4%)–Yb^{3+}(6%)) in order to give an opportunity in the future to use an IR laser at 974.4 nm.

The probe is powered by a DC electric current of varying intensity leading to Joule heating. The probe temperature can then be deduced both from the optical sensor characteristics and from the electric circuit's elements. It should be noted also that the temperature sensor with the fluorescent microcrystal is positioned on a turret with four degrees of freedom. Fine adjustments on the position of the microcrystal and the excitation wavelength are also performed.

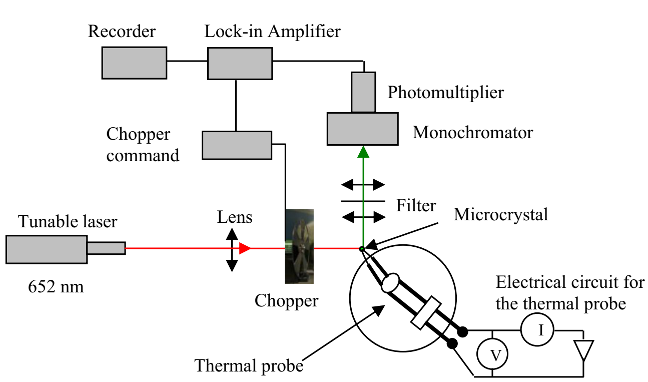

Fig. 1. Experimental set-up.

3. Results and discussion

3.1. Temperature measurement by photoluminescence of doped microcrystal

We have used fluorescent microcrystals of about 10 and 25 μm obtained from $Sr_{0.3}Cd_{0.7}F_2$transparent bulk single crystals co-doped with Er^{3+}(mol.4%) and Yb^{3+}(mol.6%) ions which are mechanically ground into fine powder. The bulk crystals were synthesized in our laboratory by the Bridgman method [10]. First, we have studied the emission spectrum of the bulk crystal to verify that the Boltzmann law can be applied. For this, we use a rectangular crystal plate 0.3 mm thick of this material. To ensure that the Boltzmann law can be applied to determine the material temperature, we place this crystal plate in a heater whose temperature is measured by a thermocouple. This heater is equipped with a small aperture through which the laser beam reaches the crystal [11]. The thermal contact is such that the crystal is at the temperature of the heater.

The green fluorescence spectrum emitted by the erbium ions of the bulk crystal (Fig. 2) and of microcrystal (Fig. 3) excited in the anti-Stokes mode (Fig. 4) by the dye laser operating at a wavelength of 652 nm consists of three distinct sharp lines located at 522, 540 and 549 nm. The emitting levels of these three lines are respectively 19,130, 18,500 and 18,380 cm^{-1} above the ground level of Er^{3+} ions. The first line (522 nm) corresponds to the $^2H_{11/2}$ (1) → $^4I_{15/2}$ (1) transition, the second line (540 nm) to the $^4S_{3/2}$ (2) → $^4I_{15/2}$ (1) and the last one (549 nm) to the $^4S_{3/2}$ (1) → $^4I_{15/2}$ (4) transition. Numbers in parentheses indicate

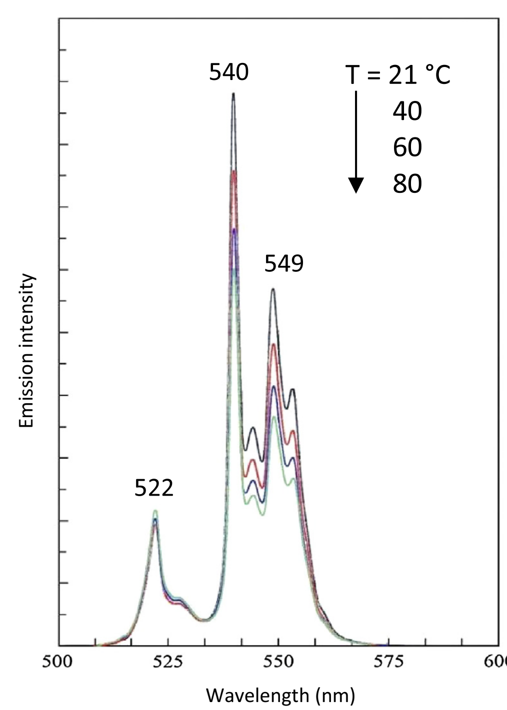

Fig. 2. Emission spectra from Er^{3+} ions of bulk crystal for different values of its temperature.

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