REMTES

TECHNOLOGY FOR REMOTE TEMPERATURE MEASUREMENTS IN MICROFLUIDIC DEVICES

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Fabrication of a microfluidic luminescence probe

Version Final

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INTRODUCTION

REMTES – “Technology for remote temperature measurements in microfluidic devices” is a Science Fund of the Republic of Serbia funded project (Program PRIZMA, Grant Contract No. 7017) coordinated and completely executed by "Vinča" Institute of Nuclear Sciences – National Institute of the Republic of Serbia, University of Belgrade (VINS). The project will run from December 1st 2023 to November 30th 2026.

REMTES is a highly ambitious and innovative project aimed at developing a breakthrough system for measuring sample temperatures on the nanoliter scale. The project will develop an optical self-referencing thermometer for use in micro- and nanofluidics in the 0–100 °C temperature range by exploiting temperature-induced changes in the luminescence of materials and nanomaterials; that is, by advancing luminescence (nano-) thermometry in a targeted manner. The project aims to go beyond the state of the art and implement a radically new technology that merges the fields of luminescence thermometry, photothermal spectroscopy, and microfluidics to develop new-generation luminescent thermometry probes using cutting-edge luminescent, temperature-sensitive, and chemically stable inorganic materials in bulk and nanomaterial forms. The probes will be embedded in microfluidic chip channels to enable self-referenced remote temperature measurements, and the technology will be validated by a portable microfluidic luminescent thermometer, as well as in-situ temperature measurements of fluid flow in nanoliter volume samples. Multiple conceptual breakthroughs can be further envisaged from the proposed technology credibly spreading its impact to multiple technological areas.

D3.3: Fabrication of a microfluidic luminescence probe is a deliverable of WP3 of the REMTES project. The report describes design and fabrication of a microfluidic luminescence probe based on the design and fabrication of a microfluidic chip and selected luminescence probes. This includes selecting two microfluidic luminescence probes for the portable microfluidic luminescent thermometer and chemical microanalysis system.

D3.3.: Report on the design and fabrication of a microfluidic luminescence probe

In this phase of the project, microfluidic luminescence probe – MLP was fabricated starting from optimized Tb-doped, Ce-codoped RbY_3F_{10} luminescent probe inserted into microfluidic chip. The procedure was as follows:

Tb-doped, Ce-co doped RbY3F10 luminescent probe

Synthesis: The RbY_3F_{10}: 10 mol%Ce, x mol%Tb nanoparticles were synthesized using a microwave-assisted solvothermal method. Initially, Y_2O_3 and Tb_4O_7 were dissolved in concentrated HNO_3 and evaporated to dryness, resulting in yttrium (III) nitrate and terbium nitrate. The obtained yttrium (III) nitrate and terbium nitrate were then mixed with in stoichiometric ratios with cerium (III) nitrate and EDTA and dissolved in 15 mL of a 1:1 (v/v) ethylene glycol and deionized water mixture. The solution was stirred for 15 minutes at room temperature. Subsequently, RbF and NH_4F were added and fully dissolved, after which the pH was adjusted to 9 with addition of NH_4OH. The mixture was stirred for an additional 15 minutes to ensure homogeneity and transferred to a 30 mL silicon carbide (SiC) vessel. The vessel was sealed and placed in an Anton Paar Monowave 400 microwave synthesis reactor (Anton Paar GmbH, Austria). The solvothermal reaction was carried out at 180 °C for 45 minutes under continuous stirring at 600 rpm. After cooling, the resulting nanoparticles were collected by centrifugation at 4000 rpm for 10 minutes, followed by washing with ethanol and an ethanol–water mixture (1:1 v/v). The final product was air-dried overnight at 40 °C. Samples were prepared with a constant concentration of 10 mol% Ce(III) and varying concentrations of Tb (x = 0, 1, 2, 5, 10, 20, 40 mol%). The corresponding precursor amounts are listed in Table 1.

Table 1 Precursor amount for synthesis of the RbY_3F_{10}: 10 mol%Ce, x mol%Tb nanoparticles.

RbY_3F_{10}:10 mol%Ce, xmol% Tb	**RbF**	**Y_2O_3**	**$Ce(NO_3)_3x6H_{20}$**	**Tb_4O_7**	**NH_4F**	**EDTA**
x= 0	0.5 mmol (0.0522g)	0.675 mmol (0.1525 g)	0.15 mmol (0.0651 g)	/	5 mmol (0.1850 g)	0.25 mmol (0.0730 g)
x= 1		0.6675 mmol (0.1508 g)		0.00375 mmol (0.0028 g)		
x= 2		0.66 mmol (0.1492 g)		0.0075 mmol (0.0056 g)		
x= 5		0.6375 mmol (0.1441 g)		0.01875 mmol (0.0140 g)		
x= 10		0.6 mmol (0.1356 g)		0.0375 mmol (0.0280 g)		
x= 20		0.525 mmol (0.1186 g)		0.075 mmol (0.0561 g)		
x= 40		0.375 mmol (0.0847 g)		0.15 mmol (0.1122 g)		

Structural analysis: RbY_3F_{10}:10 mol% Ce crystallizes in a cubic structure (space group $Fm\bar{3}m$, 226) similar to KY_3F_{10} (ICDD 01-071-2757). As shown in Figure 1, the experimental diffraction pattern of

RbY_3F_{10}:10 mol% Ce closely matches reference pattern of KY_3F_{10}, confirming the formation of the cubic phase. The most intense reflections are observed at 2θ = 26.46° (3.4 Å), 44.02° (2.1 Å), and 52.12° (1.7 Å), which correspond to the (222), (440), and (622) planes, respectively. Compared with KY_3F_{10}, the diffraction peaks shift toward lower angles, indicating lattice expansion caused by the incorporation of larger ions: Rb^+ (1.72 Å, CN = 12) relative to K^+ (1.64 Å, CN = 12), and Ce^{3+} (1.143 Å, CN = 8) relative to Y^{3+} (1.019 Å, CN = 8).

Structural analysis of the RbY_3F_{10}:10 mol% Ce, x mol% Tb samples (x = 1, 5, 10, 20, 40) was performed by comparing their X-ray diffraction (XRD) patterns with the reference pattern of KY_3F_{10} (ICDD 01-071-2757), as presented in Figure 1. The small difference in ionic radii between Tb^{3+} (1.040 Å, 8-fold coordination), Ce^{3+} (1.143 Å, 8-fold coordination) and Y^{3+} (1.019 Å, 8-fold coordination) enables isoionic substitution without phase segregation, even up to 10 mol% Ce and 40 mol% Tb doping.

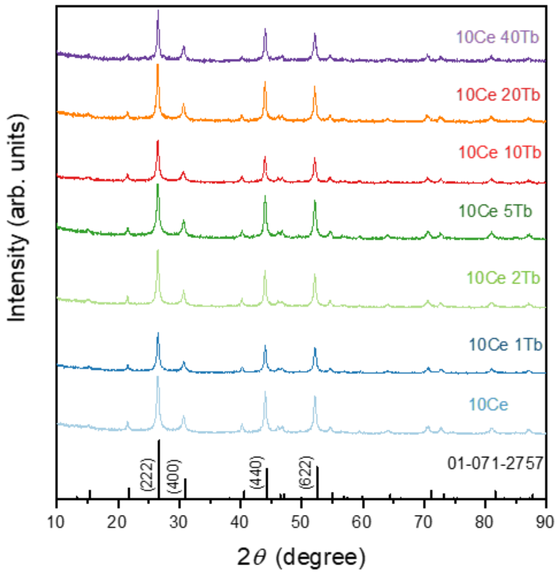

Figure 1 X-ray diffraction (XRD) patterns of RbY_3F_{10}: Ce 10mol%, x mol% Tb Eu samples (x = 0, 1, 5, 10, 20, 40), compared with the reference pattern of KY_3F_{10} (ICDD 01-071-2757).

Morphology: RbY_3F_{10}: 10 mol%Ce, 20 mol%Tb powder sample was analyzed by Transmission electron microscopy (TEM). In the TEM image given in Figure 2, clusters of nanoparticles are already visible at low magnifications. The particles form flower-like aggregates that further agglomerate into larger assemblies. The primary clusters range from 100 to 350 nm in diameter and are composed of uniformly sized nanoparticles with an average size of about 40 nm.

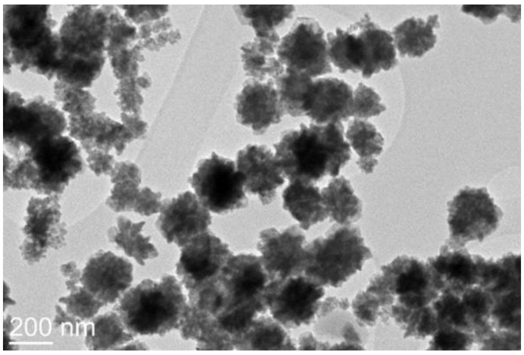

Figure 2 Transmission electron microscopy image of RbY_3F_{10}: 10 mol%Ce, 20 mol%Tb powder sample

Photoluminescence properties: Herein, Ce is used as a co dopant to enhance the emission of the Tb dopant. Figure 3a shows excitation spectrum of the representative RbY_3F_{10}: 10 mol%Ce, 20 mol%Tb sample with marked peaks corresponding to both Tb-dopant and Ce-co dopant. Figure 3b shows emission spectra of all synthesized samples showing that RbY_3F_{10}: 10 mol%Ce, 20 mol%Tb sample shows maximal emission intensity when measured under the same experimental conditions. Therefore, this sample is chosen for further integration with microfluidic chip into microfluidic luminescent probe.

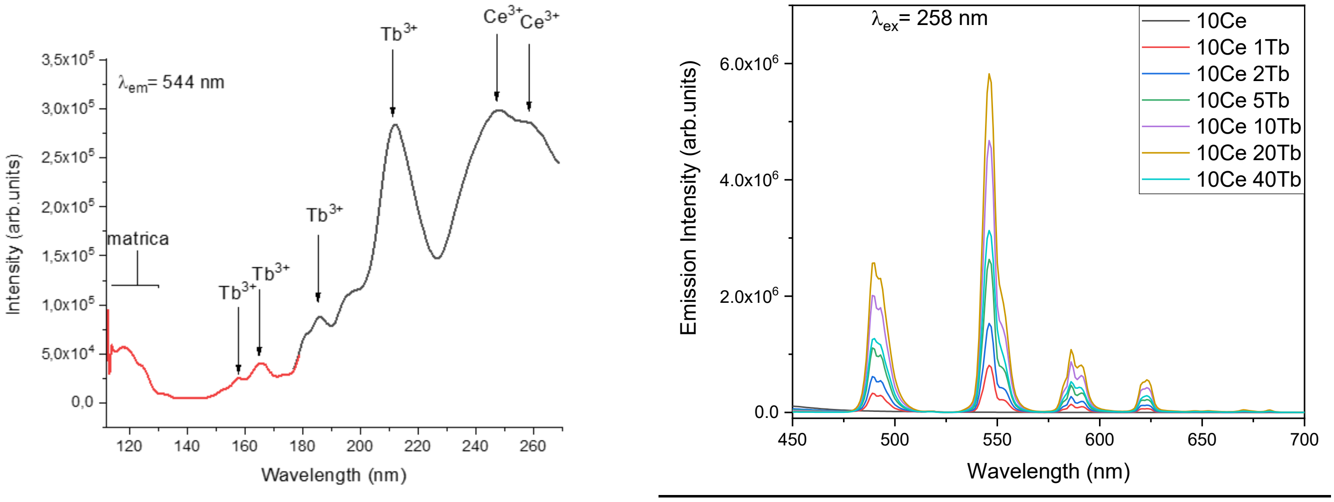

Figure 3 Photoluminescent a) excitation and b) emission spectra of synthesized samples.

Microfluidic luminescent probe

The best emitting sample, RbY_3F_{10}: 10 mol% Ce, 20 mol% Tb, was mixed with polyvinyl alcohol (PVA, 50 000–85 000 g/mol). The mixture consisted of 10 wt% PVA and 1 wt% RbY_3F_{10}: 10 mol% Ce, 20 mol% Tb in 5.0 mL of deionized water. Initially, 0.5560 g of PVA was dissolved in 5.0 mL of deionized water, after which 0.056 g of the nanomaterial was added. The mixture was stirred for 24 h to ensure complete homogeneity. After that, mixture was inserted into two types of fabricated microfluidic chips as shown in Figure 4 under daylight and 265 nm excitation.

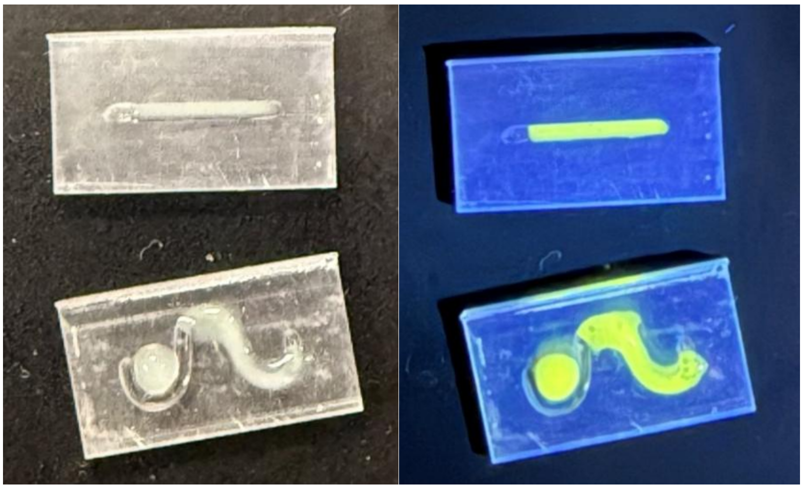

Figure 4 Microfluidic luminescence probe under a) daylight and b) 265 nm excitation.