Optimization Study of High Spatial Thermal Imaging for High-Pressure Heat Transfer Experiment using Thermographic Phosphor

Ahyeong Cho, Jonghwi Choi, Hyungdae Kim*
Department of Nuclear Engineering, Kyung Hee University, Republic of Korea
Corresponding author: hdkims@khu.ac.kr

***Keywords :** high-pressure boiling, surface temperature measurement method, thermographic phosphor

1. Introduction

Supercooled flow boiling occurs when the local heat flux in a sub-channel of the pressurized water reactor is high or in the emergency core cooling situation. In order to understand and predict the boiling phenomenon under high-pressure conditions, thermal-hydraulic experiments have been conducted. Infrared (IR) thermometry has been mainly used as the experimental technique for measuring boiling surface temperature[1,2]. Since the size of the boiling bubble at the high-pressure of about 9.8 MPa is 10 μm, it is necessary to use the measurement technique with a smaller spatial resolution[3]. However, the spatial resolution achieved in previous studies using IR thermometry is shown in table I, and it was difficult to measure surface temperature changes due to boiling. Technical limitations exist due to the absence of the high-magnification lens for infrared light. Therefore, there have been attempts to visualize the surface temperature using the thermographic phosphor that emits visible light according to temperature[4,5]. Depending on the selection of phosphor, it can be used in a wide and various temperature range. In order to measure the surface temperature using phosphor, deposition on the visible window is required. Previously, it was mainly attached using the chemical binder and plasma spraying. If the chemical binder was used, it was deformed at high temperature. In the case of plasma spraying, it cannot be used as the boiling surface because of its poor durability. Therefore, it is necessary to use other deposition methods to fabricate phosphor thin film. The objective of this study is to optimize the optical setup and thin film to measure the surface temperature at high spatial resolution using thermographic phosphor.

Table I: Spatial resolution of IR thermometry achieved in previous studies

Reference	Spatial resolution (μm)
Buffone and Sefiane, [1]	30
Teyssieux et al., [2]	17

2. Method

2.1. Fluorescence intensity ratio (FIR) method

In the process of phosphor being excited by external energy and emitting energy returning to the ground state, various types of transitions occur, and some transitions emit visible light. One of these transitions occurs more frequently as the temperature increases, resulting in an increase in fluorescence intensity. On the other hand, other transitions occur more often as the temperature decreases, increasing fluorescence intensity. Each transition has the unique wavelength due to the difference in energy level, and two wavelengths with opposite intensity tendencies according to temperature are called characteristic wavelengths. The intensity ratio of the two characteristic wavelengths according to temperature follows the Boltzmann distribution and can be expressed as equation (1).

$$(1)\ R_{FIR} = \frac{I_1}{I_2} = B\, e^{-\frac{\Delta E}{kT}}$$

Where I_1 and I_2 are the intensities of each characteristic wavelength, B is the constant, and ΔE is the effective energy difference between the two excitation levels. By taking the natural logarithm of both sides of equation (1), equation (2) can be expressed.

$$(2)\ \ln(R_{FIR}) = -\frac{\left(\frac{\Delta E}{k}\right)}{T} + \ln(\mathrm{B})$$

Therefore, if the emission intensity of the characteristic wavelength is measured at each temperature and the ratio is taken, the linear equation for the reciprocal of the temperature can be obtained. By measuring the fluorescence intensity of the two characteristic wavelengths of phosphor and taking the ratio, the temperature can be calculated inversely through equation (2).

2.2. Electrophoretic deposition (EPD)

EPD is the deposition method using the electrophoretic phenomenon in which colloidal particles in the solution move by the uniform electric field. (Fig. 1) The thickness of the thin film is determined in proportion to the deposition time.

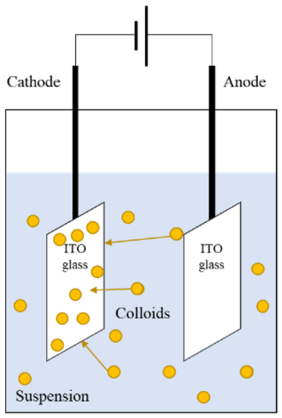

Fig. 1. Schematic diagram of EPD

2.3. Sputtering method

The Sputtering method is the deposition method using the sputtering phenomenon in which ions with high energy collide with the target and the target particles break the bond and fall apart. (Fig. 2) When the electric field is applied to the substrate and the target material, the gas in the chamber is ionized to generate plasma. Ions collide with the target material due to the potential difference inside the chamber, and the target is sputtered and deposited on the substrate. During the sputtering process, the film quality and thickness of the film vary depending on the degree of vacuum inside the chamber, the flow rate of atmosphere gas, the temperature of the substrate, and the strength of the electric field.

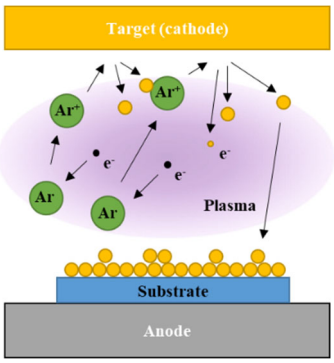

Fig. 2. Schematic diagram of sputtering method

3. Sample Preparation

3.1. Phosphor synthesis

In order to be used for thermal hydraulic experiments at high-pressure condition, it must be usable at 250~350℃. Therefore, $SrB_4O_7:Sm^{2+}$ was selected as phosphor. It was synthesized by the solid phase synthesis, which is the method of combining and mixing solid powders and reacting through diffusion of particles at the high temperature. $SrCO_3$, H_3BO_3 and Sm_2O_3 were used as the precursor materials.

3.2. Thin film deposition

The EPD sample was deposited with the phosphor thin film after depositing the ITO thin film on the borosilicate glass substrate. The substrate was connected to the cathode and deposited for 30 seconds, followed by annealing at 500℃ for 30 minutes. The thickness of the thin film was measured in the cross section through the scanning electron microscope and was approximately 7~15 μm.

The sputtering sample was deposited by selecting annealing as the process variable. It was deposited on the sapphire substrate at the voltage of 70 W and the temperature of 100℃ for 24 hours. Three samples were manufactured under the same conditions. Thereafter, one sample was annealed at 600℃ for 1 hour in a reducing atmosphere, the other sample was annealed at 600℃ for 1 hour in air, and the remaining sample was not annealed. The thickness of the deposited thin film was measured with the surface scan profiler and was approximately 10~12 μm.

4. Experiment

4.1. Experimental setup

The optical system was designed to excite the sample with the laser and measure the emitted light, and the experimental setup is shown in Fig. 3. The laser with the wavelength of 355 nm, and the diameter of 1 mm was used. To measure the surface temperature, the laser was irradiated to the sample by expanding the beam diameter to 6 mm using the 2× and 3× beam expander. The laser had the frequency of 1,000 Hz and the power of 2.5 W. The laser was aligned using three ultraviolet (UV) mirrors. The expanded laser was reflected by the UV mirror at the bottom of the sample and passed through the dichroic mirror with the cut-off wavelength of 405 nm to irradiate the sample. The heating block was heated by the mounted cartridge heater, and the sample was heated through conduction from the heating block. In the case of the spectroscopy experiment, the hole was made in the sample cover to measure the light emission on the sample. (Fig. 3(a)) In the spatial resolution measurement experiment using the high-speed camera, the fluorescence emitted from the sample was reflected by the dichroic mirror below the sample and measured by the camera. (Fig. 3(b)) The long-distance microscope was used to measure with high spatial resolution. In order to obtain by dividing the wavelength range of the two characteristic wavelength regimes, the fluorescence intensity image was obtained by dividing the wavelength based on 605 nm using the image splitter.

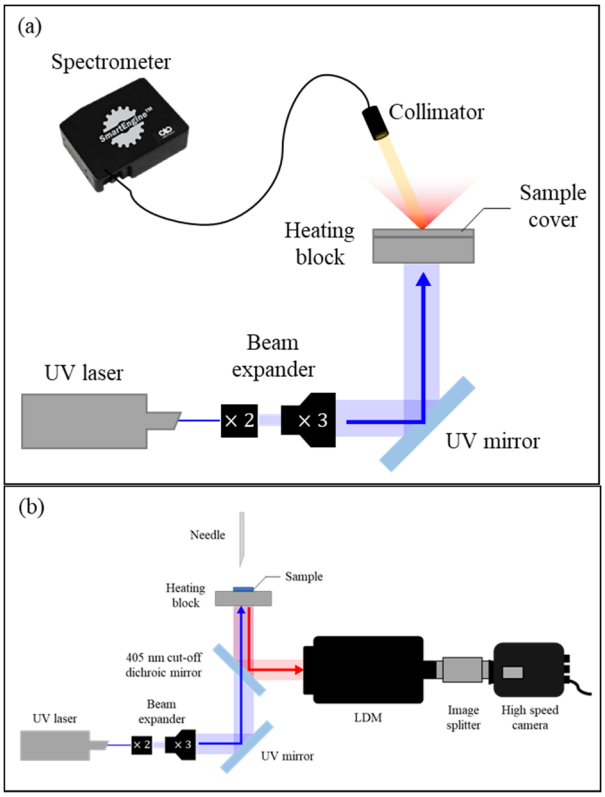

Fig. 3. Experimental setup: (a) spectroscopy experiment, (b) spatial resolution measurement experiment

4.2. Spectroscopy

4.2.1. EPD sample

The fluorescence spectrum according to the temperature of the EPD sample was measured, and the ratio of the characteristic wavelength was obtained. (Fig. 4) The linear equation of $\ln(R_{FIR})$ for the reciprocal of temperature was calculated, and the relative temperature measurement sensitivity was calculated. As a result, it was 0.3%K^{-1} to 0.5%K^{-1} in the temperature range of 200 to 350℃, and 0.3314%K^{-1} at 327℃.

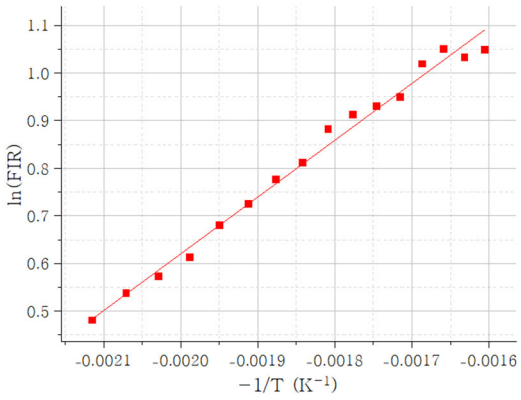

Fig. 4. $\ln(R_{FIR})$ value according to the reciprocal of the temperature of EPD sample

4.2.2. Sputtering sample

The fluorescence spectrum according to the temperature of the sputtering sample was measured. $\ln(R_{FIR})$ for the reciprocal of temperature was shown in Fig. 5. In the case of sample annealed in air, the temperature did not increase linearly as the temperature increased. The relative temperature measurement sensitivity of the non-annealed sample was approximately 0.2%K^{-1} higher than that of the sample annealed in the reducing atmosphere. The relative temperature measurement sensitivity at 327℃ of each sample is shown in table II.

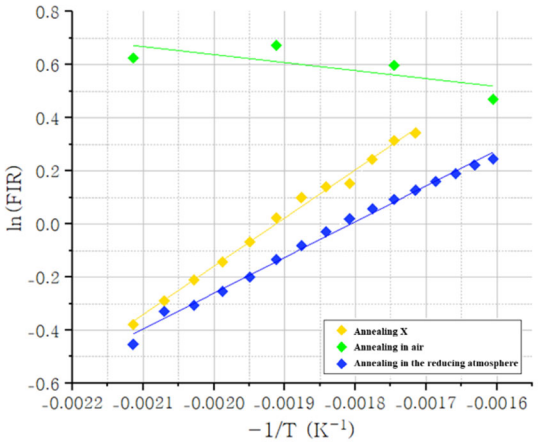

Fig. 5. $\ln(R_{FIR})$ value according to the reciprocal of the temperature of sputtering sample

Table II: Relative temperature measurement sensitivity at 327℃

Sample	Relative temperature measurement sensitivity (%K^{-1})
Annealing X	0.5049
Annealing in air	-0.08
Annealing in the reducing atmosphere	0.3741

4.2.3. Intensity of fluorescence

Fluorescence intensity could be relatively compared using the exposure time of the spectrometer and the measured intensity. The measured fluorescence intensity was integrated and divided by the exposure time for comparison. The relative fluorescence intensity is shown in Fig. 6. The fluorescence intensity of EPD sample was approximately 100 times stronger than that of sputtering sample.

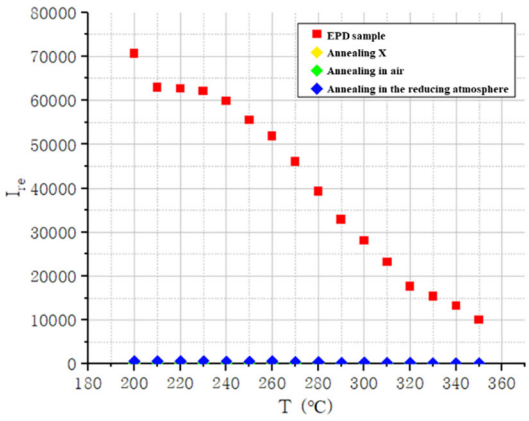

Fig. 6. Fluorescence intensity according to sample

4.3. Spatial resolution analysis

Because high-speed imaging was performed using the high-magnification long-distance microscope, the EPD sample with high fluorescence intensity was used. The temperature of the sample was maintained at 150℃, and the temperature change was measured by contacting the surface with the room temperature needle with the outer diameter of 260 μm. (Fig. 7(a)) The spatial resolution of FIR thermometry was measured to be 3.295 μm/pixel. For comparison, the surface temperature was measured using IR thermometry under the same conditions. (Fig. 7(b)) The measured spatial resolution was 106.156 μm/pixel. Therefore, it was confirmed that the surface temperature can be measured with improved spatial resolution through phosphor. However, there was the cooled temperature difference between IR thermometry and FIR thermometry, which was judged to be due to the uneven phosphor surface and image alignment error.

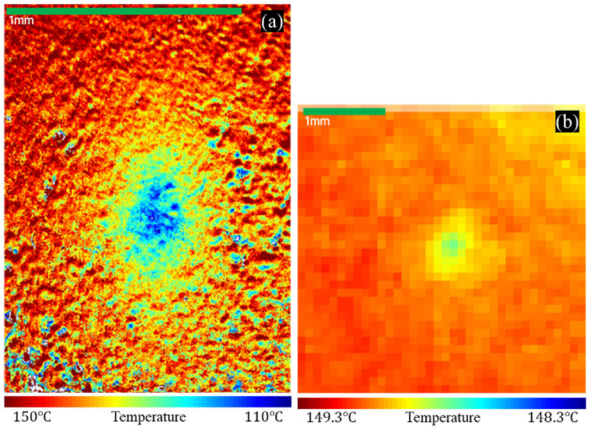

Fig. 7. Surface temperature distribution, when the needle was in contact: (a) FIR thermometry, (b) IR thermometry

5. Conclusion

To utilize the surface temperature measurement method using thermographic phosphor under high temperature and high-pressure condition, thin film fabrication and optical system optimization studies were conducted. Samples were prepared by depositing phosphor through the EPD and sputtering method, and the relative temperature measurement sensitivity and fluorescence intensity were analyzed through spectroscopy. As a result, the sputtering sample without annealing had the highest relative temperature measurement sensitivity, and the fluorescence intensity of the EPD sample was 100 times stronger than that of the sputtering sample. The surface temperature was measured using the EPD sample with strong fluorescence intensity and compared with IR thermometry. In FIR thermometry, the spatial resolution of 3.295 μm/pixel was reached, which is the value that can measure the surface temperature change due to boiling at high-pressure condition. Therefore, by complementing for the error in image alignment and depositing the phosphor thin film with the uniform surface, it will be possible to measure the surface temperature in the high-pressure boiling thermal-hydraulic experiment.

ACKNOWLEDGMENTS

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT: Ministry of Science and ICT) (2019M2D2A1A02059364)

REFERENCES

[1] C. Buffone, K. Sefiane, IR measurements of interfacial temperature during phase change in a confined environment, Experimental Thermal and Fluid Science, Vol. 29, no. 1, pp. 65-74, 2004.
[2] D. Teyssieux et al., Near-infrared thermography using a chargecoupled device camera: Application to microsystems, Review of Scientific Instruments, Vol. 78, no. 3, sp. 034902, 2007.
[3] R. Semeria, High-speed cinematography and pool boiling under high pressure, La Houlle Blanche, Vol. 6, pp. 679–687, 1963.
[4] J. Brubach, C. Pflitsch, A. Dreizler, B. Atakan, On surface temperature measurements with thermographic phosphors: A review, Progress in Energy and Combustion Science, Vol. 39, no. 1, pp. 37-60, 2013.
[5] J. G. Odhiambo, W. Li, Y. Zhao, C. Li, Porosity and Its Significance in Plasma-Sprayed Coatings, Coatings, Vol. 9, no. 7, sp. 460, 2019.