Characterization of Defects in LiNbO₃ Using IR Thermal Imaging Camera and PPE Method

赤外線カメラと PPE 法を用いた LiNb0₃の欠陥評価

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1. Introduction

Recently, lithium niobate (LiNbO₃; LN) is in the great attention as ferroelectric materials which are useful in wide fields of industry due to its outstanding piezoelectric effect, pyroelectric effect, and electro-optical effect. However, a large quantity of defects produced during the crystal growth, essential cause decline in the materials characteristic. In recent years, the infrared thermal imaging camera (IR-CAMERA) has been applied in various fields of security, construction and medicine because of convenience and precision. In this study, we use the IR-CAMERA to study thermal property. In particular, we focus on the new method that can evaluate the defects quickly and conveniently. We have detected the defects in the LN substrates, while comparing with the results of the defects observation by the scanning laser microscope (SLM) and the light scattering tomography (LST), and evaluated the behavior of the heat as the macroscopic viewpoint. Moreover, we compared with the results of photopyroelectric (PPE) method to estimate thermal effects of the defects in the LN substrates.

2. Experimental

We prepared samples with different amount of defects, sample A (low defect density) and sample B (high defect density). The samples were mirror-polished. First, we observed the shape of defects on the surface of each sample using the SLM, and calculated the density and occupation ratio of defects from the SLM images. Next, we investigated the depth distribution of the defects using the LST, with a well-shaped beam of an Nd-YAG laser (λ = 1064 nm) to illuminate the inside of substrates [1]. When the infrared light enters from the cleaved surface, the defects areas

glisten as scatterers. The scattering light is visualized by using a near-infrared charge coupled device (CCD) camera. The laser beam was adjusted to 25 μ m in diameter to obtain clear tomograms. Finally, we observed the defects classified through the process of each sample by IR-CAMERA, and compared with the PPE method for estimating thermal effects in substrates [2].

The IR-CAMERA detects and visualizes infrared light radiated from the materials. If the substrates have defects or distortions, the thermal properties (thermal capacity, thermal diffusivity, thermal conductivity, etc.) of these areas will be changed compared with the periphery. Therefore, these areas show a relatively different temperature. Using the amount of infrared radiation can detect defects and distortions in substrate conveniently and accurately. The Amount of infrared radiation can be described as follows:

$$W = \sigma T^4 \tag{1}$$

where, W is the amount of infrared radiation per unit area (W/cm² · μ m), σ is the Stefan-Boltzmann constant (5.6705 × 10⁻¹² W/cm² · K⁴) and, T is the temperature (K) [3].



Fig. 1. Schematic diagram of the experiment.

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In this study, we used a long wavelength IR-CAMERA, with a measurement range of -20 ~ 250 °C. Thermal sensitivity and spectral range of this camera were 0.02 K at 303 K and 8 ~ 14 μ m, respectively. A 25 μ m close up lens was used, where minimum detectable size was 25 × 25 μ m. The samples were heated by an electric heating plate and observed on a flat copper plate. To decrease the background noise, the sample and camera were shielded by box (**Fig. 1**).

3. Results and Discussion

We observed the shape of defects on the LN surface and structural defects identified by using the SLM. **Table 1** shows the results of the density and occupation ratio of defects from the SLM observation.

Table 1. The results of SLM observation.

Properties	Sample A	Sample B
Defect density (cm ⁻²)	3.3×10^{3}	3.6×10^4
Defect area-ratio (%)	1.59	2.62



(low defect density) (high defect density) Fig. 2. SLM images of the sample A and B. The defects

are observed as the black points in the substrates.



(low defect density)

(high defect density)

Fig. 3. LST images of the sample A and B. The white parts are scatterers of the reflected light.

Next, the LST was applied to observe the depth distribution of the defects. From the observation, we confirmed that the defects located not only on the substrate surface but also in the substrate. **Figures 2** and **3** show the SLM and LST photograph, respectively. Finally, the thermal behavior of defects is observed by using the IR-CAMERA. **Figure 4** shows the IR images of the each sample.



Fig. 4. IR-CAMERA images of the sample A and B. The defects are observed as different temperature compared with the periphery in the substrates.

The black spots correspond to defects in the substrate. The defects detected by the SLM and LST were also observed. The defects showed the lower temperature compared with that of the periphery, which the IR images are due to the difference of infrared radiation depending on the heat capacity. In general, the heat capacity of defects areas were changed compared with that of the periphery. As a result, we assume that the temperature variations are caused by the difference in heat capacity. The result is equivalent to the thermal diffusivity and thermal conductivity estimated by PPE method. Though the observable size of defects is larger than the resolution of the IR-CAMERA (25 µm), the thermally detectable size of defects in this study is approximately 1 µm. The IR-CAMERA is effective to detect line and point defects such as through-defects, impurities and vacancy. We conform that the IR-CAMERA is more effective technique to evaluate the defects in LN than conventional ultrasonic microscope, because it can observe defects easily, immediately and precisely without any damages in the substrates.

4. Conclusion

For the first time, we applied the IR-CAMERA to characterize the defects in LiNbO₃. Compared with the SLM, LST and PPE, IR-CAMERA was effective to evaluate the defects such as through-defects and impurities in LiNbO₃. And also, we confirmed visually that the defects in the substrate affected the temperature distribution of the entire substrate.

References

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