

# Application News

SPM-Nanoa™ Scanning Probe Microscope (Atomic Force Microscope)

## Visualization of Piezoelectric Response in Extremely Small Region of Piezoelectric Material by SPM

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### User Benefits

- ◆ SPM enables observation of the piezoelectric response of individual polarization domains, which is a key factor determining the characteristics of piezoelectric materials.
- ◆ Evaluation based on an atmosphere (air, inert gas, low vacuum) close to the actual use environment is possible.
- ◆ Detailed searches for targets can be conducted with a high resolution optical microscope, which is integrated with the SPM in a single system.

### Introduction

Piezoelectric materials have the piezoelectric property that application of a voltage causes deformation. Utilizing this characteristic, these materials play an important role as sensors, buzzers, and filters in various industrial products. Recently, particularly with ongoing downsizing of electronic equipment and communication devices, even higher performance has been required in piezoelectric materials, and development is being promoted energetically.

Piezoelectric materials are composed of aligned spontaneous polarization regions (domains). These domains are polarized in different directions, as shown in Fig.1 and display different responses to applied voltage. The response of the individual domains is a key factor that determines the characteristics of a piezoelectric material. However, high sensitivity is required in order to evaluate the domain response because deformation on the nanometer order under an applied voltage is not uncommon. Here, we introduce an example in which the minute response of a piezoelectric material to an applied voltage was captured by using a scanning probe microscope (SPM (AFM)), which is capable of detecting the piezoelectric response at the sub-nanometer order.



Fig. 1 Image of Polarization Domains

### SPM-Nanoa

The scanning probe microscope (SPM) is a type of microscope which enables high magnification observation and measurement of the 3-dimensional topography and local physical properties of samples by scanning the sample surface with a tiny probe called a cantilever. Shimadzu SPM-Nanoa is a new SPM which is equipped with an advanced, high sensitivity detection system and automatic observation function as standard features, and satisfies the user's "desire to see" more simply, in greater detail, and more quickly, providing powerful assistance for work ranging from topographical observation of minute regions to the measurement of physical properties. Fig. 2 shows the appearance of the SPM-Nanoa. The advantages of the SPM-Nanoa can be summarized in the following 3 points.

- ① Automatic observation: Adjusts the laser beam axis, adjusts the parameter settings during observation, and performs image processing automatically.
- ② Extensive functionality: Observes localized physical properties with high resolution.
- ③ Saves time: Various support functions realize fast observation.

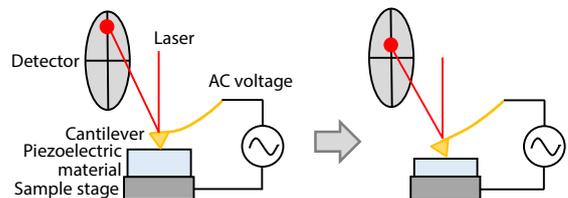
This article introduces an example in which localized physical properties (piezoelectric response) were captured with high resolution, which is one of the functions in ② Extensive functionality.



Fig. 2 SPM-Nanoa™ Scanning Probe Microscope

### Polarization Domain Observation System

Fig.3 shows a schematic diagram of expansion/contraction detection of the piezoelectric material. The laser beam is irradiated on the back side of the cantilever, and the setting of the detection system is done so that the reflected laser beam is incident on the detector. The amount of cantilever deflection is detected by using the phenomenon in which the position of laser incidence on the detector shifts vertically as a result of deflection of the cantilever. An AC voltage is applied between the probe and the sample while the cantilever is in contact with the piezoelectric material, and the expansion or contraction of the material is detected from the amount of deflection of the cantilever.



When the piezoelectric material contracts, the position of laser beam incidence on the detector shifts downward.

Fig. 3 Schematic Diagram of Expansion/Contraction Detection of Piezoelectric Material

The vertical response of local domains to the applied voltage is measured by lock-in detection by using the expansion/contraction of the piezoelectric material detected by the method described above as the input signal and the applied AC voltage as the reference signal. The direction and magnitude of expansion/contraction are evaluated by the phase signal and the amplitude signal, respectively. The illustrations in Fig. 4(a) and (b) show domains ① and ②, which displayed different responses, extracting the instants (a)-1 and (b)-1 when a minus voltage is applied to the cantilever side and a plus voltage is applied to the sample stage side, and the instants (a)-2 and (b)-2 when a plus voltage is applied to the cantilever side and a minus voltage is applied to the sample stage side. In this case, the parts shown in red (Fig. 4) are detected as amplitude signals.

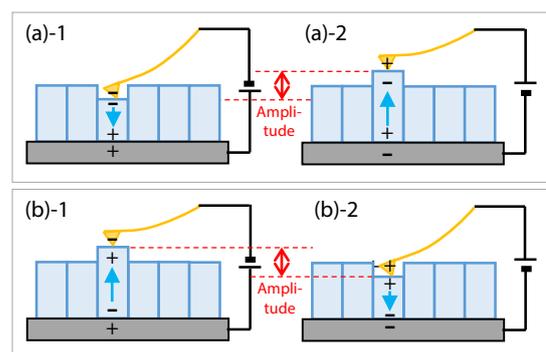
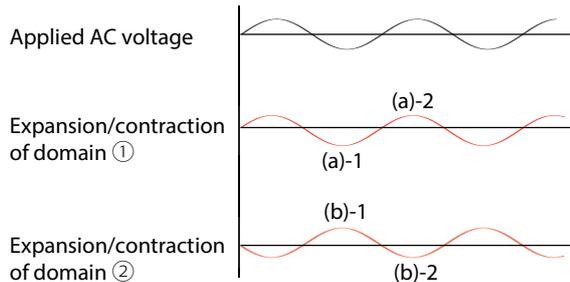


Fig. 4 Relationship of Expansion/Contraction of Piezoelectric Material in Response to Applied Voltage and Amplitude Signal  
(a) Domain ① (b) Domain ②

In domain ① and domain ②, the phase signals are detected as values which differ by 180°, as shown in Fig. 5.



In domain ① and domain ②, the response has opposite phases (differs by 180°).

Fig. 5 Relationship of Expansion/Contraction of Piezoelectric Material in Response to Applied Voltage and Phase Signal

### ■ Piezoelectric Response Measurement of LiNbO<sub>3</sub> Single Crystal

The piezoelectric response in the vertical direction was measured in a LiNbO<sub>3</sub> single crystal (Fig. 6), in which domains with different polarization characteristics are arranged adjacently at a 10 μm pitch.

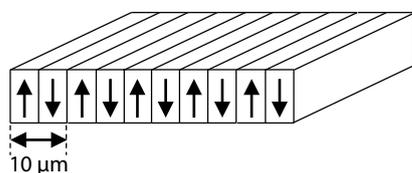


Fig. 6 Polarization Domain Structure of LiNbO<sub>3</sub> Single Crystal

Table 1 Observation Conditions

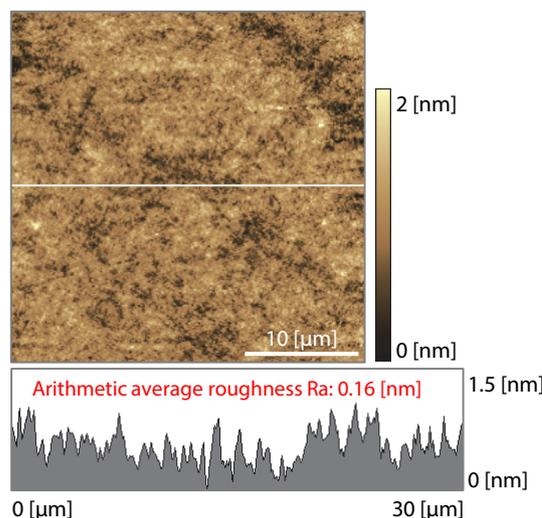
Instrument	: SPM-Nanoa scanning probe microscope
Scanner	: Wide range scanner (125 μm)
Observation mode	: PFM mode
Observation field	: 30 μm × 30 μm

Fig. 7 shows the measurement results. In Fig. 7(a), the roughness of the LiNbO<sub>3</sub> surface is observed. In the Topographic image, a topography suggesting polarization domains is not observed, and it is not possible to determine even the domain distribution, much less the piezoelectric characteristics of the LiNbO<sub>3</sub>. In Fig. 7(b), the magnitude of expansion/contraction of the LiNbO<sub>3</sub> under an applied AC voltage is measured. Here, it can be understood that the expansion/contraction of domain A is larger than that of domain B at the same applied voltage. A tiny amplitude difference of only 0.4 nm could be captured clearly by the advanced and high sensitivity detection system of the SPM-Nanoa. In Fig. 7(c), the response of the LiNbO<sub>3</sub> to the applied AC voltage is measured. The phases of domain A and domain B differ by 180°, indicating that these domains have polarization characteristics in opposite directions.

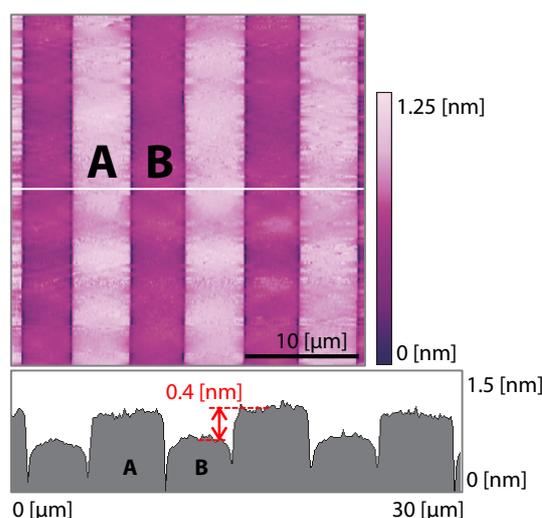
### ■ Conclusion

By using a Shimadzu SPM, it was possible to capture the distribution of polarization domains in an ultra-small region of a LiNbO<sub>3</sub> single crystal and the responses of the respective domains to an applied voltage. Although this article introduced an example in which the piezoelectric response in the vertical direction was captured, similar measurements in the horizontal direction (torsion) are also possible. A detailed evaluation of the piezoelectric response of sample materials is possible by an examination combining the measurement results in the vertical and horizontal directions.

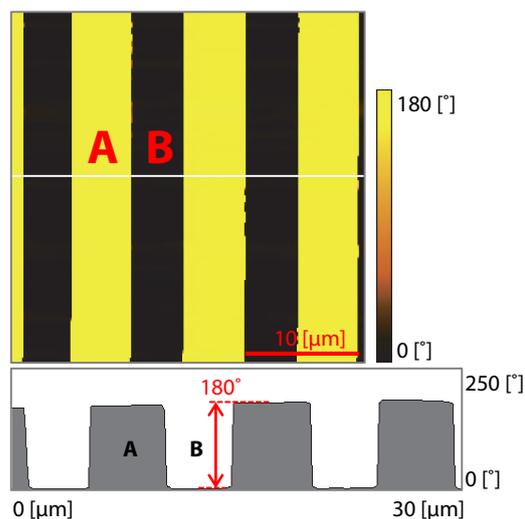
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(a) Topographic image (top) and profile at position of analysis line (bottom)



(b) Amplitude image (top) and profile at position of analysis line (bottom)



(c) Phase image (top) and profile at position of analysis line (bottom)

Fig. 7 Results of Piezoelectric Response Measurements of LiNbO<sub>3</sub> Single Crystal