

## Observation of the domain structure near $\beta$ -Incommensurate- $\alpha$ phase transition by X-ray topography

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### Introduction

Quartz crystal was an attractive material for industrial and scientific interests and has been studied for many years. In 1980's, the incommensurate (INC) phase with the modulation wave-vector  $q (=0.03a^*$ :  $a^*$  is a reciprocal lattice vector of hexagonal system) between  $\alpha$ - $\beta$  phases was established[1-3]. In INC phase near the phase boundary  $\pm\phi$  domain were observed by the electron microscope[3]. In the  $+\phi$  domain the  $\text{SiO}_4$  tetrahedron rotates counterclockwise around X-axis. If the laser light is input along Z-axis, the ring-shaped scattering pattern due to the  $\pm\phi$  domain wall was observed. While the laser light is input parallel to X-Y plane, the strong elastic scattering is observed at  $\alpha$ -phase close to the phase boundary in X-Y plane. In this report, in order to make clear the origin of this scattering, we examine carefully the vicinity of the phase boundary by x-ray diffraction topography and optical microscope.

### Experimental

For the purpos of the optical microscopic and x-ray topographic investigations, we made a furnace with the large window, which enables to measure up to  $2\theta = 50$  deg. for the forward scattering. Its temperature stability is less than 0.5 K within 10 hrs near the  $\alpha$ -INC- $\beta$  transition temperature ( $\sim 850$ K). There is the temperature gradient  $\Delta T \sim 0.5$  K between the top and bottom along vertical direction to enable to observe the  $\alpha$ -INC and the phase boundary at the center of sample. The weight of this furnace is very light (8 kg) enough to be mounted on the topographic goniometer of 15B beam line.

The samples are Z-cut synthetic quartz plate (TOYOCOM) with  $8 \times 8 \times 0.6$  mm<sup>3</sup>. The white and monochromatic sources were used. Topographic images were exposed on ordinary films for x-ray and x-ray CCD camera, which were put in the position 30 cms distant from the sample. To avoid the heating of sample by x-ray illumination, the lights with a low energy were attenuated by an aluminium plate.

### Results and Discussion

Figure 1(a) and (b) show the optical microscopic image and (1-10) topograph by a white x-ray, respectively, near the phase boundary between  $\alpha$  and INC phases. The fine structures are observed only at INC phase near the phase boundary in both images. These structure are responsible

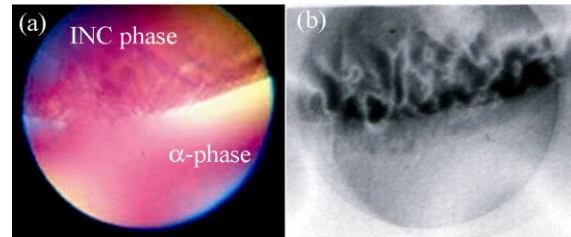


Fig. 1 (a) a optical microscopic and (b) (1-10) topographic images on a film for x-ray near phase boundary.

for the contrast due to the strain of  $\pm\phi$  domain boundaries. At the  $\alpha$ -phase side, no such structure was observed.

Comparing the Fig.1(b) with Fig.2(b), it is clear that the resolution of CCD camera is sufficient in quality for our experiment. In Fig.2(b), also in (1-10) topograph the fine structure is observed only just above phase boundary, while in (3-31) topograph the marble like domain is first found below the phase is boundary, i.e.,  $\alpha$ -phase. Since this contrast comes from the difference between the structure factors of  $\alpha_+$  and  $\alpha_-$  domains, these domains can be identified as the Dauphine twin. There are many fine domains, which have the walls parallel to Z-axis, near phase boundary as shown in Fig.2(c). These walls of the marble like domains could be the origin of the above mentioned anomalous elastic scattering.

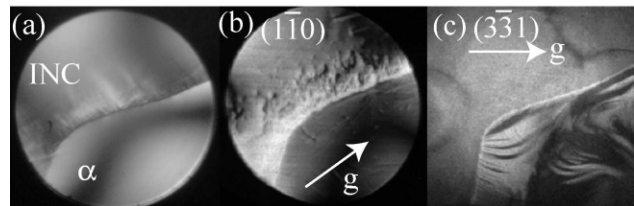


Fig.2 (a) microscopic image (b) (1-10) topograph and (c) (3-31) topograph by x-ray CCD camera.

### References

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