Quantitative analyses of Brazil twin defects in chalcedony by X-ray powder diffraction method

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Introduction

Chalcedony and agate have been recognized as aggregates of submicron-sized quartz crystals. However, recent transmission electron microscopy (TEM) observations[1] reveal that chalcedony commonly includes new silica polymorph, "moganite", which structure is similar to that of modified quartz by Brazil twinning[2-3]. The quartz crystal precipitated from low-temperature solution is known to be characterized by intergrowth with moganite and/or development of Brazil twin lamellae.

In this study, XRD measurements on synthetic submicron-sized quartz crystals and natural chalcedony samples were carried out to clarify the growth process of moganite and genesis of Brazil twin lamellae.

Experimental

Experimental samples are listed in Table 1. The synthetic samples were obtained by re-crystallization from silica gel (Wako Q-22) in 0.1N KOH solution at 175-225°C. Natural chalcedony (agate) samples were collected from Brazil, and Hosaka, Fukushima Prefecture, Japan. SR resonant powder diffraction data collections were carried out with multiple-detector system (MDS) installed at the BL-4B2 station. The step scan widths were 0.004° or 0.005° in 2θ and fixed counting times were 1.5 or 2 sec. The wave length of the incident X-ray was 1.2 Å.

Table 1. List of measured samples

Sample No.	Occurrences	Growth temp. (°C)	Mature times (hours)
1	Natural chalcedony (Hosaka, Fukushima)	-	-
2	Natural chalcedony (Hosaka, Fukushima)	-	-
3	Natural agate (Brazil)	-	-
4	Synthetic	175	363
5	Synthetic	175	576
6	Synthetic	200	171
7	Synthetic	200	250
8	Synthetic	225	100

Results and Discussion

The results of cell parameter refinement of quartz crystals in the samples are shown in Figure 1. The unit cell dimensions show slightly different trends between the synthetic and the natural samples. In the synthetic samples, the unit cell dimensions are reduced with increasing temperature and mature time of growth. Peak fitting analyses also shows different crystallographic directions of strain in the synthetic and natural crystals.

Our TEM observations show that the synthetic samples are composed of quartz and moganite that includes numerous planar defects. However, a characteristic reflection of moganite could not be detected in the synthetic samples by the XRD analyses using SR. This result suggested that an amount of moganite crystal is too little in the synthetic sample to be detected and/or that the highly disordered structure with planar defects reduce intensity of the reflection. Although moganite nucleates only at the initial stage of the crystallization of quartz crystals, moganite do not grow at the following growth stage. A kinetic process causes the moganite crystal at the nucleation stage of the silica precipitation.

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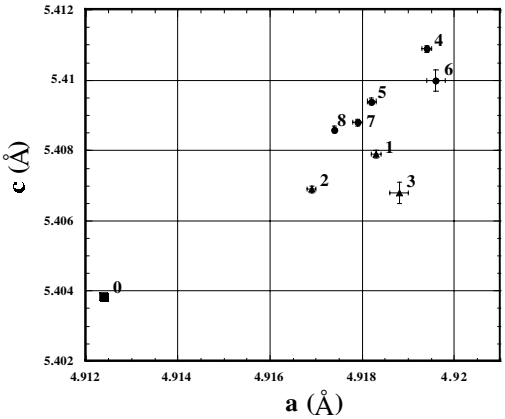

Figure 1. Variation of unit cell dimensions of measured samples. The number in the figure corresponds to the sample number in Table 1. No. 0: the bulk quartz crystal measured by Will et al.[4]

References

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