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In situ observation of twin boundary formation at grain-boundary groove during directional solidification of Si

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ABSTRACT

Twin boundary formation at grain boundaries in multicrystalline Si during directional solidification was investigated by in situ observation of the crystal/melt interface. It was clearly shown that a twin boundary was formed on the {111} facet of grain-boundary groove at the crystal/melt interface. The large amount of undercooling in the melt at grain-boundary grooves promoted rapid crystallization and twin boundary formation.

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Multicrystalline Si (mc-Si) ingots grown by directional solidification for use in solar cells always exhibit twin boundaries [1–12]. Structural analysis of mc-Si ingots and wafers thereof has revealed that Σ3 twins with a {111} boundary plane were often generated at grain boundaries. However, it is not well understood when or why this occurs. It has been argued that one reason might be to reduce the free energy at the crystal/melt interface [2]. If the grain boundary energy is large, defects such as dislocations, twins, or a new crystal grain may be generated to reduce the energy. On the other hand, Duffar and Nadri argued that, theoretically, twins would be expected to appear on facets at grain boundary-solid-liquid triple junctions (that is, at grain-boundary grooves) [13]. Recently, Tsoutsouva et al. directly observed twin boundary generation at a grain-boundary groove at the crystal/melt interface during directional solidification from a Si{110} seed crystal by using in situ X-ray imaging [14]. They showed that the twin boundary was generated when the melt at the grain boundary groove crystallized. However, such direct evidence for the formation of twin boundaries remains very limited, owing to the difficulties in observing the Si crystal/melt interface due to the high melting temperature (1687 K). Therefore, the details of this process have not been clarified yet. We previously reported the formation of grain-boundary grooves [15] and impurity accumulation [16] at the Si crystal/melt interface using in situ observations. Reflection optical digital microscopy could be performed in our system, allowing the surface structure of the sample to be clearly observed.

In the present study, we attempted to obtain experimental evidence for the generation of {111} Σ3 twin boundaries at grain boundaries during the directional growth of mc-Si. In particular, we focused on twin boundary formation at grain-boundary grooves, which has been reported by Tsoutsouva et al. [14].

The crystal/melt interface of a mc-Si sample with grain boundaries was observed during the directional solidification process by using an in situ observation system that consisted of a furnace and a microscope [15,16]. The Si raw materials, placed in a silica crucible, were melted completely in the furnace. Then, directional growth was promoted by cooling one side of the crucible. A high-speed camera with a light source was used to obtain a reflected image of the sample surface, allowing us to clearly identify grain boundaries and twin boundaries in the growing crystal. The crystal growth process was recorded as a video at 250 frames/s. After crystallization, a crystallographic orientation analysis was performed to determine the Σ values of the observed grain boundaries by the electron backscattering diffraction pattern (EBSP) method.

Fig. 1 shows a mc-Si crystal/melt interface in motion. The recording was started at $t = 0$ s, at which time we could observe two grain boundaries that formed grooves at the crystal/melt interface. It is known that {111} facets appear on the growth surface at the grain boundary. Thus, the groove was formed by two {111} facets [13,15]. Another groove started to appear at the crystal/melt interface at $t = 5.7$ s in the area indicated by the red circle (clearer in the magnified image in the red square). Let us focus on the morphological changes occurring in the vicinity of this groove. The depth of the groove increased with crystal growth until around $t = 30$ s. Then, the melt in this deep groove rapidly crystallized, filling the groove, as shown in the images for $t = 35$–60 s. Fig. 2 shows a frame taken at $t = 60$ s and its schematic. The remnant of a deep groove is clearly visible in the crystal. We note that a new line, indicating the formation of a new boundary, extends from under the {111} facet that formed a deep groove, while there is no boundary extending from the upper {111} plane. These results strongly indicate

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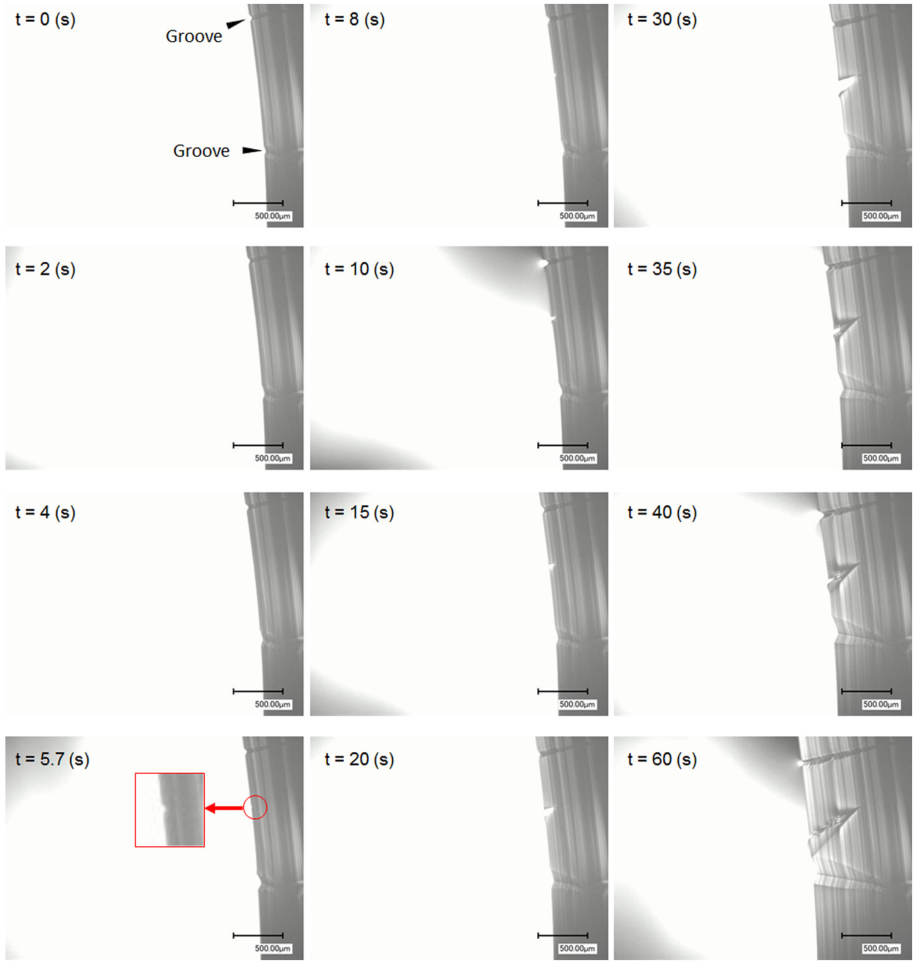

Fig. 1. Crystal/melt interface during unidirectional growth of mc-Si. The recording was started at time t = 0 s.

that a {111}Σ3 twin boundary was formed upon rapid growth at the deep groove.

EBSP analysis was performed to identify the grain boundary characteristics and grain orientations. Fig. 3 shows (a) a scanning electron microscopy (SEM) image, (b) an image quality (IQ) map with colored grain boundaries, and (c) an orientation map for the TD direction (crystal growth direction). We can easily identify the observation area shown in Fig. 1 because the remnant of the deep groove is visible in the SEM image, as shown in Fig. 3(a). Thus, the orientation analysis was performed around this area. Analysis of the grain boundary characteristics (Fig. 3(b)) confirmed that the new boundary, which was generated upon rapid crystallization at the deep groove, was a Σ3 twin

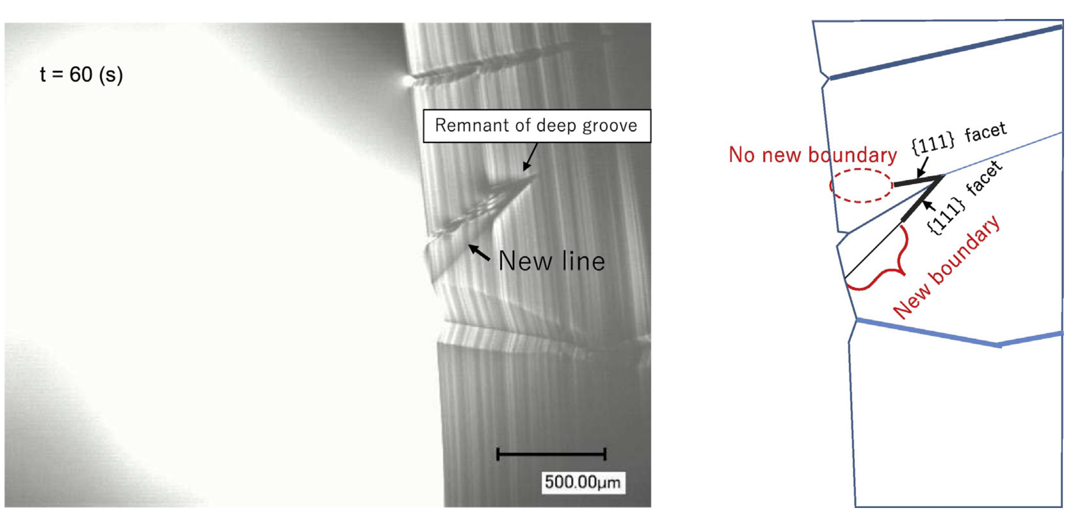

Fig. 2. Image of crystal/melt interface at t = 60 s and its schematic. The remnant of a deep groove is clearly visible in the crystal.

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