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Improvement in Quantitative Analysis of Defects and Microstructures in Si Multicrystals Using X-ray Diffraction

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1. Introduction

Over the past decade, Si multicrystals have emerged as the substrate material for commercial solar cells. To accelerate the adoption of solar cells to the world, ongoing research targets further reductions in energy costs by improving material performance, while reducing manufacturing costs and raw material costs. We have approached this issue from studies of crystal growth and defects characterization based on deep understanding of crystal physics of Si multicrystals. The former study leads us to proposal of the dendritic casting method [1] to control the structure and reduce crystal defects. This article presents the latter study, namely defects characterization in Si multicrystals.

Among the defects in Si multicrystals, we focused on sub-grain boundaries (sub-GBs) because they are known to perform as serious carrier recombination sites for solar cells [2]. We have proposed a technique for quantitative analysis of the spatial distribution of sub-GB density and angular difference between sub-grains in a crystal grain using rocking curve measurement of x-ray diffraction (XRD). Sub-GBs with relative angle (0.1~10°) were observed in a part of Si multicrystal sample ingots, and they tend to be dense in narrow area which spread in the growth direction of the ingot. Furthermore, we applied two-dimensional (2D) detector for the characterization, which provide us fast characterization as well as the information of residual strain distribution.

2. Defects characterization using XRD

The XRD measurements were performed on the as-cut surface that is parallel to the growth direction of the Si multicrystal ingot as shown in Fig. 1. We chose the rotation axis as the growth direction so that rocking curve measurements, i.e. 2θ-fixed and ω-scan, of XRD can separate the diffraction from each sub-grains with misorientation in rotation along the growth direction. Furthermore, in order to analyze a large area at once over the different crystal grains, we utilized structure-controlled Si multicrystal ingot with almost the same crystal orientation in the growth direction realized by “dendritic casting method”. Because of the same crystal orientation in the growth direction, we can use the same diffraction plane, i.e. the same 2θ angle, for the ω-scans in different crystal grains.

Figure 2 shows a typical profile of the ω-scan of XRD from the spot with one sub-GB. For the characterization of sub-GBs, the number of peaks in the profile is considered to be the number of sub-grains in the x-ray spot. Therefore,

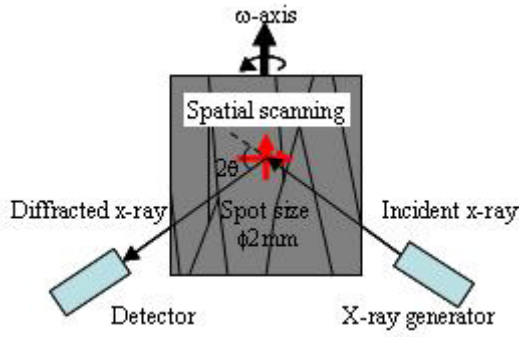

Fig. 1 Illustration of optical configurations of spatially resolved XRD measurement: The angle resolution is smaller than 0.01° in ω-scan. Note that such a resolution cannot be obtained by other conventional techniques for characterization of crystal orientation such as SEM-EBSP analysis.

dividing the number of peaks by the spot size. In addition, the angle between the peaks corresponds to the misorientation between the sub-grains. Performing the above analyses at each position on the cross-section of the ingot provides spatial information of sub-GBs. In this study, the ω-scans were performed every 1mm in x- and y-axis on the sample surface using the 2 mm x-ray spot in diameter.

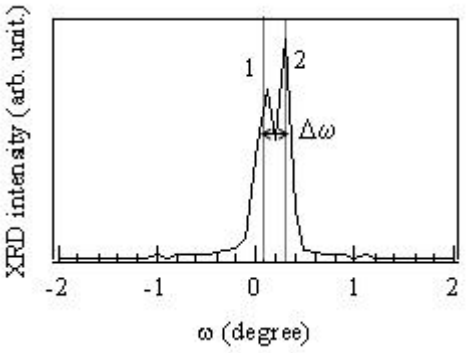

Fig. 2 Typical ω-scan profile of XRD from the spot with one sub-GB. For the characterization of the sub-GBs, the number of the peaks is considered to be the number of the sub-grains i.e. sub-GB density, and the angle between the peaks, Δω, corresponds to the misorientation between the sub-grains.

In a part of Si multicrystal sample ingots, sub-GBs with relative angle (0.1~10°) were observed. As an example of the sample contains many sub-GBs, (a) photo image of the cross-section of the ingot, (b) spatial distributions of sub-GB density and (c) spatial distribution of maximum relative angle between sub-grains are shown in Fig.3. In

Fig.3 (b), the areas with zero density are assigned as areas where there is no sub-GB and the areas with –1 density are assigned as areas where crystal orientation dose not satisfy the x-ray diffraction condition, in other words, where the crystal orientation in the growth direction differs from that of the main area. It is found that spatial distribution of sub-GB density and maximum relative angle between sub-grains were successfully imaged over some GBs. As a result of the analyses of several sample ingots, we found that the spatial distribution of sub-GBs is not homogeneous and sub-GBs tend to be dense in the narrow area and spread in the growth direction of the ingots.

3. Application of 2D detector for defects characterization

Figure 4 shows schematic illustration of optical configuration of the XRD measurements using 2D detector. In this case, the information of crystal orientation is reflected in χ direction on the 2D image plate and can be taken without mechanical moving scan. This provides faster characterization of sub-GBs comparing with the process using normal detector. Furthermore, the information of crystal lattice size that is residual strain is reflected in 2θ direction on the 2D image plate. Therefore, informations of crystal orientation and residual strain can be taken in one trigger of the image, simultaneously.

In order to confirm the validity of 2D detector, we performed sub-GB analysis using normal detector and 2D detector on the same points with several sub-GBs. The same profile with that taken using normal detector can be taken in 2D detector in tenth shorter time. Furthermore, fluctuations of the spot position in 2θ direction were observed. This might indicate the existence of residual strain in the Si multicrystals. The quantification of the residual strain is still under considerations.

4. Summary

We have demonstrated that spatially resolved XRD is a powerful tool to clarify the spatial distribution of the sub-GB density and angular difference between sub-grains. Sub-GBs found to be dense in narrow area and spread in the growth direction of the ingot. We also attempted to apply 2D detector for the characterization and showed faster process time and potential to measure the spatial distribution of the residual strain.

Acknowledgements

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References

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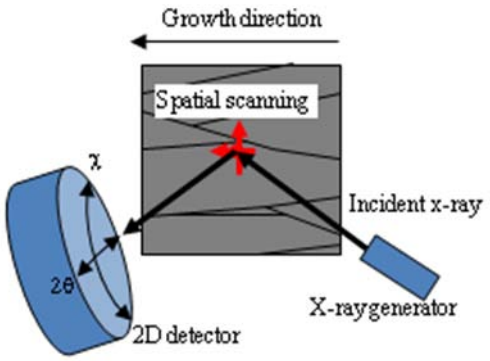

Fig.4 Illustration of optical configurations of spatially resolved XRD measurement using 2D detector.

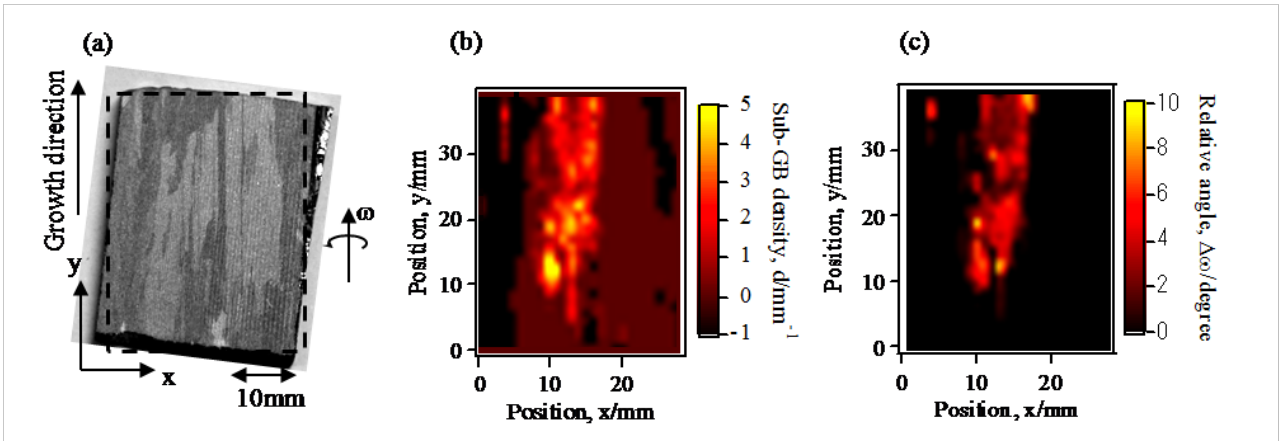

Fig.3 (a) Photo image of the sample surface. Spatial distributions of (b) sub-GB density and (c) relative angle between sub-grains in broken square in Fig. 3 (a). In the Fig. 3 (b), the areas with -1 sub-GB density show unknown area where crystal orientation dose not satisfy the diffraction condition. The areas with 0 sub-GB density show the areas without sub-GBs.