# Characterization of crystal grain shape and crystal axis orientation mapping on surface of HOPG using electron backscatter diffraction

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**Introduction**

Highly oriented pyrolytic graphite (HOPG) is a polycrystalline graphite, c-axes of the crystal grains aligning perpendicular to the surface. The a-axis, however, have different orientations between the crystal grains. Since the sizes of the crystal grains are in a range of several microns to several hundred microns, it is difficult to measure the grain size, shape, and distribution of the crystal grains by means of X-ray diffractometry and transmission electron microscopy. The orientation and shape of each crystal grain on the surface of HOPG and a highly oriented graphite film prepared from Kapton can be evaluate by electron channeling pattern (ECP) and electron channeling contrast (ECC) that is the special measurement mode attached to scanning electron microscope (SEM)[1-4]. It is difficult to make the map of crystal information obtained by a lot of ECPs. Because time to measure one ECP is a few minutes. And, the number of data to make the map of crystal information will become 10,000 points or more. Moreover, the PC system for the analysis of ECP is not marketed

As the resolutions of ECP and ECC are about several microns, ECP and ECC techniques cannot analyze the shape and crystal orientation of the grain with several microns or less in size. On the other hand, an orientation imaging microscopy (OIM) using electron backscattered diffraction (EBSD) system goes into practical use. OIM is able to analyze the grain orientation at high speed due to the recent development of high sensitivity charge-coupling devices (CCD) camera and also high-speed PC.

High spatial resolution of OIM was obtained by installing the OIM system in high-resolution field emission scanning electron microscope (FESEM). The OIM system came to be able to analyze the spatial resolution of 0.02 microns in 0.1 seconds or less. The OIM system can analyze the shape, crystal orientation, and grain orientation distribution, and orientation difference between the crystal grains for a polycrystalline material with already-known crystal structure by mapping using the information of the EBSD pattern. In the present study, the analysis was made for a high quality HOPG for calibration of scanning tunneling microscopy.

**Experimental**

HOPG with clean, flat and fresh surfaces was obtained by stripping off the original surface layers by an adhesive tape. The EBSP measurement, the mapping processing, and the crystal orientation analysis were carried out by FESEM (S4100, Hitachi Ltd.) in which the OIM system developed by TSL was installed. The specimen is placed on a stage inclined usually 70 degrees from the horizontal line in the FESEM and faces the EBSD detector shown in Figure 1. EBSP was imaged on the phosphor screen by irradiating the electron beam to the sample, viewed using a CCD camera transferred to personal computer.

EBSPs were measured 60000 points in three-micron step in the region of 300 microns x600 microns on the HOPG surface, and the mapping was carried out.

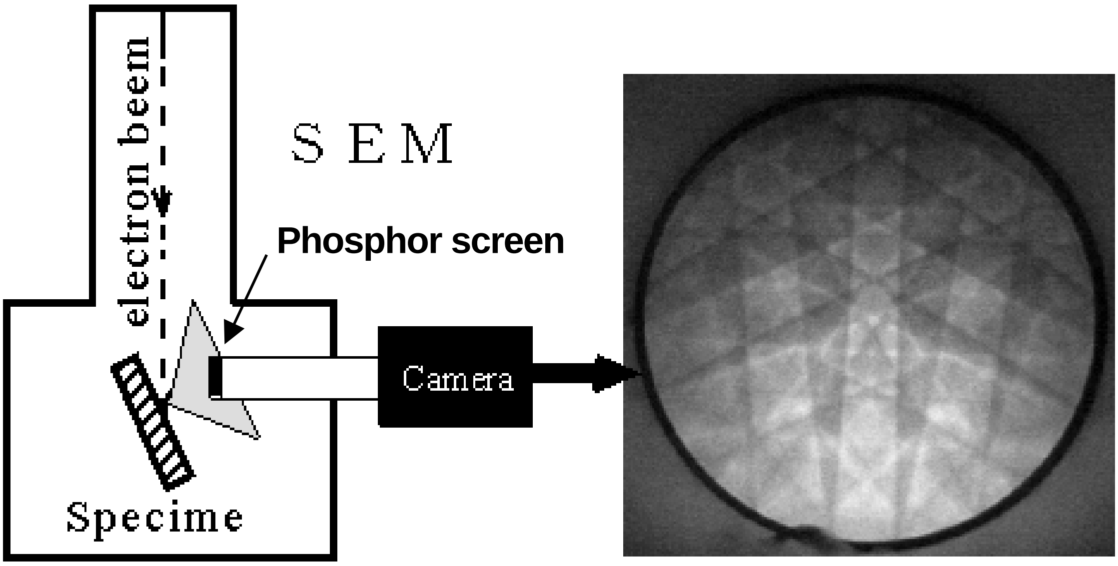



Fig.1 Schematic of EBSD system and EBSD pattern.

## Results and discussion

Figure 2 shows an Image Quality (IQ) map of the HOPG surface. In figure 2, the pattern of clear EBSP is obtained from a bright part in the IQ map. The analysis of the EBSP pattern obtained from a dark or black part is difficult. The dark or black part is attributed to the grain boundary, dislocation, other defects and impurities. Therefore, the region enclosed with dark or black parts in the IQ map can be considered to be crystal grain.

Figure 3 shows the crystal-direction map (IPF mapping image). The crystal-direction map is almost the same brightness in one crystal grain. The crystal orientation is shown by the brightness in the inverse pole figure (figure 4).

Figure 5 shows the crystal-direction map overlaid with boundary-lines artificially. The line thickness in Fig. 5 is defined by the misorientation angle between crystal grains on the surface of the HOPG. (Misorientation of 3-5 degrees; , 6-15 degrees; , 16-30 degrees; )

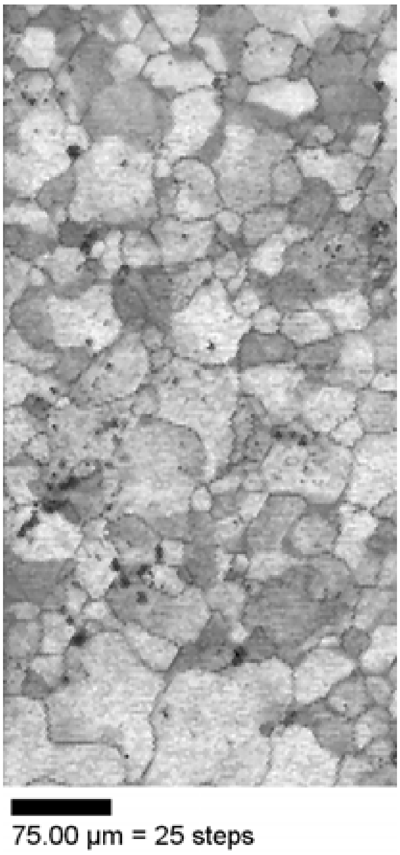



Fig.2 Image Quality map from the HOPG

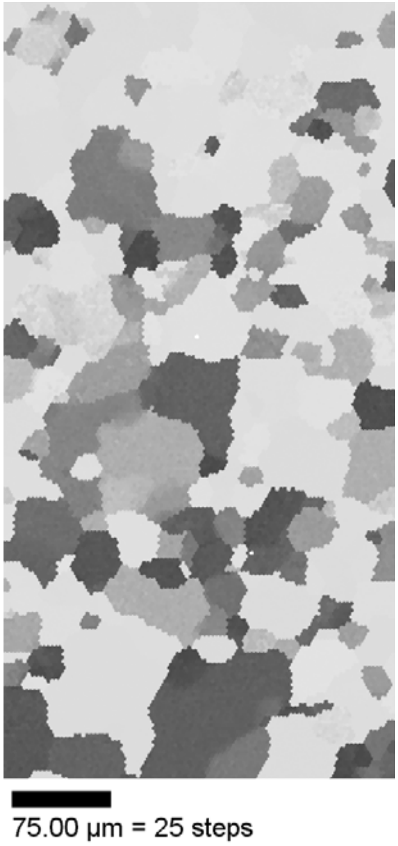



Fig.3 Crystal-direction map from the HOPG.

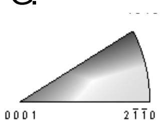
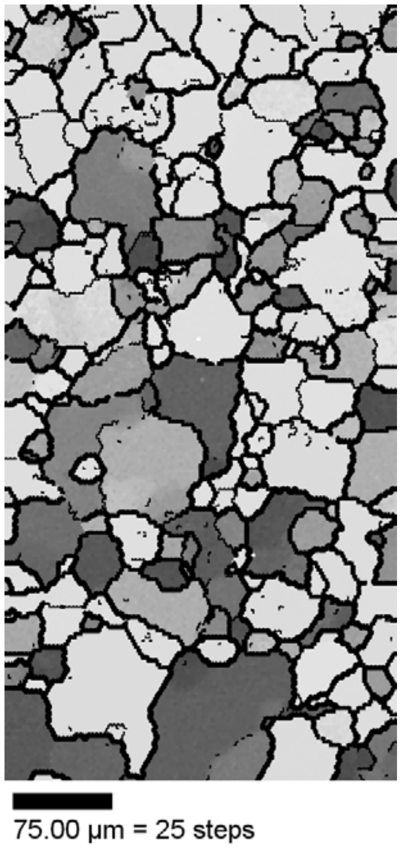



Fig.4 Inverse pole figure




Boundaries: Rotation Angle

| | Min | Max | Fraction |
|---|---|---|---|
| (thick line) | 16° | 30° | 0.476 |
| (medium line) | 5° | 15° | 0.330 |
| (thin line) | 2° | 5° | 0.170 |

Fig.5. Crystal-direction map overlaid with boundaries lined according to misorientation angle from the HOPG.

Figure 6 shows the histogram of the crystal gain size when each shape of the grains is approximately oval. The grain size is distributed within the range of about 8-120 microns. The peak in the histogram is about 20 micron diameter.

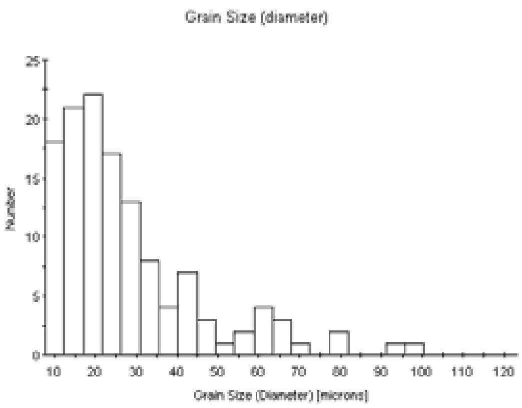



Fig.6 Histogram of the crystal gain size for HOPG

Figure 7 shows the histogram of the boundary where the misorientation angle of the adjoining crystal grain is 3-30 degrees in the HOPG. The number of the boundaries is somewhat higher around low and high misorientation angle sides than around middle.

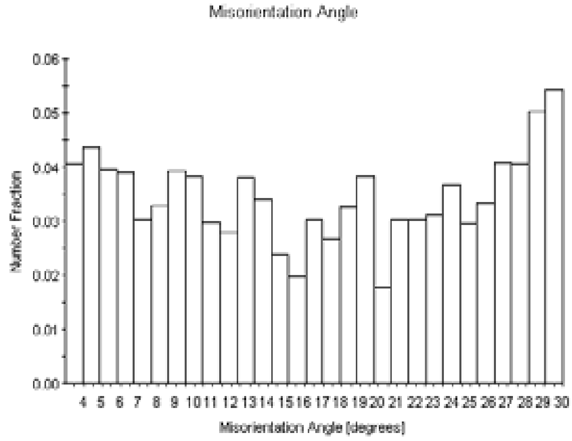



Fig.7 Histogram of the boundary where the misorientation angle

The pole figure and pole plot are shown in Figs. 8 and 9. The c-axes distribution concentrates almost on the center, and c-axes of the crystal grains in HOPG are considered to be vertically distributed to the surface in the difference within the range of 2-3 degrees. It was realized that the IOM system using EBSD is very useful to examine the grain size, and orientation distribution, crystal grain distribution for the highly oriented graphite such as HOPG.

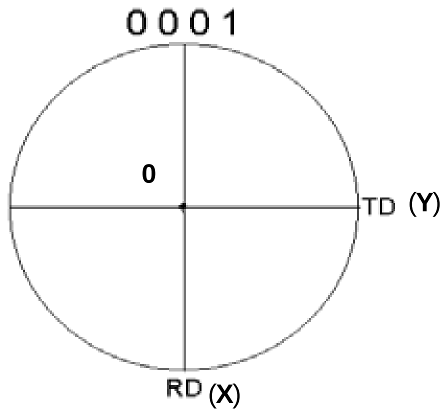



Fig.8 Pole figure for HOPG

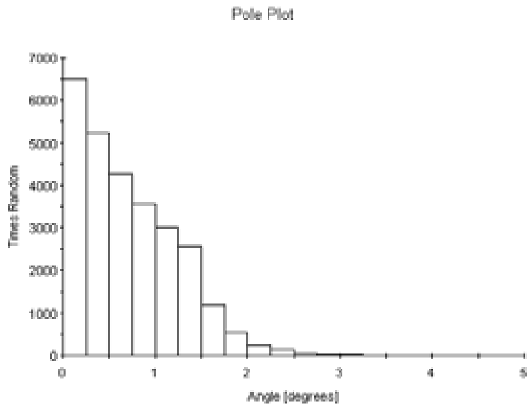



Fig.9 Pole plot for HOPG

## References

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