EBSD analysis of Grain Boundary Characteristics of Abnormally Grain Grown Alumina

Dong-Ik Kim¹, Je-Hun Lee¹,², Young-Woon Kim¹,
Kyu Hwan Oh¹ and Hu-Chul Lee¹

¹ School of Materials Science and Engineering, Seoul National University, Shinrim-dong 56-1,
Kwanak-ku, Seoul 151-742, Korea
² Center for Microstructure Science of Materials and School of Materials Science & Engineering
Seoul National University, San 56-1, Shinrim-dong, Kwanak-ku, Seoul 151-744, Korea

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Abstract. Electron Back Scattered Diffraction(EBSD) analysis was carried out to investigate boundary characteristics of Abnormally Grain Grown(AGG) Alumina. To obtain whole orientation information from AGG Alumina, separately measured BSD mapping data was combined together to be satisfied with condition of minimum misorientation, which is called Montage technique. The orientation information from 0.5 x 3.5mm and 0.35 x 1.00 mm sizes of AGG alumina was obtained. Misorientation distribution in Normally Grain Grown(NGG) Alumina was very close to the random misorientation boundary. Misorientation distribution between AGG and NGG alumina showed random boundary characteristics in 0.5 x 3.5 mm AGG alumina. Large part of captured grain inside AGG grain has high angle boundary of 90º.

Introduction

Microstructures of polycrystalline alumina and their related properties often depend critically on both the presence of dopants and of residual impurities. It is well known that small amounts of liquid forming additives such as CaO or SiO₂ can induce AGG during sintering of alumina.[1-2] On the other hand, the addition of MgO can prevent AGG[3], however, there exist critical concentration for the prevention of AGG in alumina.[4]

During AGG, large grains develop and grow fairly rapidly at the expense of the fine matrix materials. Abnormally large alumina grains are usually elongated with long basal planes. An amorphous(liquid, glass) phase was observed to be present as a thin film at the flat basal planes.[5,6]

AGG in the presence of a liquid is a phenomenon observed only when the grains are angular with flat interface such as Al₂O₃[7] TaC-TiC-Ni[8], and BaTiO₃.[9] In order to understand the growth mechanism of AGG, the investigation of orientation relationships between AGG and matrix grain boundaries are essential including identification of solid/liquid interface structures.

Recently orientation of crystalline material and misorientation between adjacent grains can be easily obtained EBSD equipment installed in SEM. Observation of AGG grain by EBSD was restricted due to its large geometrical features of AGG grains, typically mm in size. In this study, to get whole orientation data from AGG alumina, montage technique was used, which can join separately measured fairly small size map into whole area map data. To get optimum montage condition, a concept of minimum misorientation was used. The misorientation between AGG and NGG alumina was analyzed and compared with random misorientation.

Random Misorientation of Hexagonal Crystal
Among the reported crystal structure of alumina, hexagonal crystal information was used to indexing Kikuchi pattern during EBSD mapping. The misorientation distribution in randomly distributed hexagonal crystal was obtained by using quaternion representation of orientation and integrated over fundamental zone. The obtained results are as follows. $P(\phi)$ is probability and $\phi$ is misorientation. The calculated results shown in following figures comparing with measured data. Average value of misorientation is theoretically 60.07°. The maximum misorientation angle is 93.84°.

1) $0^\circ \leq \phi \leq 2 \tan^{-1}(2 - \sqrt{3}) = 30^\circ$ : $p(\phi) = \frac{1}{15}(1 - \cos \phi)

2) $30^\circ \leq \phi \leq 2 \tan^{-1}(1) = 90^\circ$ : $p(\phi) = \frac{(2 - \sqrt{3})}{15}\sin \phi

3) $90^\circ \leq \phi \leq 2 \tan^{-1}(\sqrt{2}(\sqrt{3} - 1)) = 91.98^\circ$ : $p(\phi) = \frac{1}{15}[(8 - \sqrt{3})\sin \phi - 6(1 - \cos \phi)]

4) $91.98^\circ \leq \phi \leq 2 \tan^{-1}(\sqrt{15 - 8\sqrt{3}}) = 93.84^\circ$

$$p(\phi) = \frac{1}{15}[(8 - \sqrt{3})\sin \phi + \{24S(a,b,\frac{\pi}{2}) + 12S(b,b,\frac{\pi}{6}) - 6\}(1 - \cos \phi)]$$

$$S(\alpha, \beta, \gamma) = \frac{1}{2\pi}\cos^{-1}\{(\frac{\cos \alpha \cos \beta - \cos \gamma}{\sin \alpha \sin \beta}) - \cos \alpha \cos^{-1}\{(\frac{\cos \beta - \cos \beta \cos \alpha}{\sin \gamma})\} - \cos \beta \cos^{-1}\{(\frac{\cos \alpha - \cos \beta \cos \gamma}{\sin \beta \sin \gamma})\]\}

$$a = \cos^{-1}((2 - \sqrt{3})\cot \frac{\pi}{2} \phi), \quad b = \cos^{-1}(\cot \frac{\pi}{2} \phi)$$

Montage Formulation

Observation of very large AGG grain by EBSD is restricted due to its large geometrical features typically mm in size and its high aspect ratio. To get whole orientation data from AGG alumina, required mapping area is divided into many subregions and separate mapping is carried out in the subregion. To join the separately measured mapping data, the minimum misorientation per unit overlapped area is used to determine overlapping location of mapping data as follows.

$$\text{Min}\{\frac{1}{A}\int_A k(x)w(g_a(x), g_b(x))dA(x)\} \quad (2)$$

where $A(x)$ is scanning area, $w(g_a(x), g_b(x))$ is misorientation between two measured data from regions a and b at same mapping position x and $k(x)$ is overlapping parameter where overlapped position has value of 1 and non-overlapped position has zero. During EBSD measurement, montage along 70° tilt axis is preferred.

Fig. 1 Schematic Diagram representing Montage between separately measured area.
Experiments

Composition of alumina used in this study is 100ppm CaO, 100ppm SiO2, and 50ppm MgO doped Alumina. Premixed powder was sintered at 1600°C for 16h. To obtain a good Kikuchi pattern during EBSD measurement, thermal etching technique was used after mechanical polishing. EBSD Mapping was carried in INCA CRYSTAL from Oxford Instrument Ltd., which is installed in JEOL 6500F SEM. To get the whole orientation information from AGG alumina and around AGG grain, nine mappings were carried out serially. Each mapping has 256x256 points with 300 magnification, where each point covers 2.548µm². After montage of nine serial mapping data, the whole measurement points are 2242 x 312 pts, equivalent to sizes of 3573x497µm. The solved mapping points are 569731 mapping points and AGG grain has 387102 points. The total number of grains of 5° grain boundary in whole mapping is 8869 with 1 AGG grain.

Result and Discussion

Fig. 2 shows the pattern quality map of montage area. Nine serial mapping was carried out and montaged with the condition of minimum misorientation. Fig. 3 shows geometry of AGG grain, which has flat cylinder shape and orientation of its axis is [0001]. Fig. 4 shows misorientation distribution from NGG grain shown in Fig.2 and from AGG/NGG boundary together with theoretically random boundary.

The misorientation average in NGG Grains is 58.20° and that in AGG/NGG boundary is 59.40°. Both misorientation averages are very close to random misorientation average of 60.27°. AGG/NGG boundary has 7282 mapping points equivalent to length of 11605µm. Along AGG/NGG boundary, 1007 NGG grains contact with an AGG grain, where each NGG grain contacts 34.63µm(7.231 points) in average with AGG grain. Misorientation average inside AGG Grain is 0.92°. As shown in Fig. 4, grain boundary characteristics between NGG grains and between AGG and NGG grains are statistically close to random boundary. Number of grains included in misorientation calculation seems to be enough to obtain statistics. Typical and reliable equipment obtaining orientation information is Transmission Electron Microscope(TEM), of which reasonably and practically measurable number of grains is below one hundred. EBSD mapping and Montage technique used in this study can increase the measurable grain number of statistical data about hundred thousand, which is thousand times larger than TEM.

Fig 2. Pattern Quality of Montaged mapping area

Fig. 3 Typical Geometry of AGG Grain
As shown in Fig. 3, AGG grain has flat type cylinder shape of which radius is about ten times larger than axis. This geometrically anisotropic feature shows that growth rate along radial direction seems to be about ten times faster than along axis direction and that possibly grain boundary characteristics has a deviation from random distribution. The misorientation distribution shown in Fig. 4 shows almost random characteristics. Fig. 5 shows the misorientation distribution on the disc and along wall side of AGG alumina. The misorientation distributions on disc side and on wall side show random distribution. This fact shows that orientation characteristics between AGG grain and its adjacent NGG grain may not affect the growth rate of AGG grain and geometrical features. These facts require an isotropic growth mechanism to explain randomness of AGG/NGG grain boundary.
Fig. 6 Montaged Map of 0.35 x 1.00 mm AGG alumina

Fig. 7 Selected Grains of High misorientation about 90° with AGG Grain

Fig. 8 Misorientation Distribution on the disc circle and wall of 0.35x1.0mm AGG alumina

Fig. 6 shows orientation map of 0.35x1.0mm AGG alumina. The shape of alumina is fairly irregular disc, of which axis direction is [0001]. AGG alumina contacts with NGG alumina and captures small alumina grain inside AGG grain. The captured grains seem to be slow moving AGG/NGG grain boundary. The misorientation distribution of AGG/NGG grain boundary is shown in Fig. 8 as “all AGG/NGG interface”, including misorientation data from the captured grain. The misorientation probability near 90° is two times larger than that of random boundary. Fig. 7 shows the grain with high angle AGG/NGG boundary. The selected range of misorientation is from 87.5° to 92.5°. About half of grain boundary is located at the captured grain inside AGG grain. The misorientation distribution without captured grain is shown in Fig. 8. The misorientation AGG/NGG boundary without captured grain shows random boundary as shown in Fig. 8. These facts show that ‘statistically’ slow growing AGG/NGG boundary is high angle boundary of misorientation near 90°. But the result may not be coincident with flat AGG/NGG boundary as shown in Fig. 2, which is 3 times larger than shown in Fig. 6.
Summary

EBSD analysis was carried out to investigate boundary characteristics of AGG Alumina. To obtain whole orientation information from AGG Alumina, separately measured EBSD mapping data was montaged with condition of minimum misorientation. The orientation information from 0.5 x 3.5 mm and 0.35 x 1.00 mm sizes of AGG alumina was obtained. Misorientation distribution in NGG alumina was very close to the random misorientation boundary. Misorientation distribution between AGG and NGG alumina showed random boundary characteristics in 0.5 x 3.5 mm AGG alumina. Large number of captured grain inside AGG grain has high angle boundary of 90°. Without captured grain, the misorientation shows random boundary in 0.5 x 3.5 mm AGG alumina.

References