The Effect of Atomic Mass on the Physical Spatial Resolution in EBSD

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Abstract: In this study, bicrystals of silver (Ag) and aluminum (Al) were used to investigate the physical spatial resolution of the electron backscatter diffraction system combining a digital image correlation method. Furthermore, the effect of the accelerating voltage and probe current was investigated on the physical spatial resolution of the lateral and longitudinal resolutions for Ag and Al, respectively. The lateral and longitudinal resolutions show high dependency on the accelerating voltage for a low atomic mass material of Al. In addition, these are almost independent of the accelerating voltage for a high atomic mass material of Ag. Moreover, the probe current does not play any role on both the lateral and longitudinal resolutions. The best lateral resolutions for Al and Ag are 40.5 and 12.1 nm at 10 kV and 1 nA, respectively. The best longitudinal resolutions of 23.2 and 80 nm were obtained at 10 kV and 1 nA for Al and Ag, respectively.

Key words: EBSD, spatial resolution, Monte Carlo, bicrystals

INTRODUCTION

Recently, electron backscatter diffraction (EBSD) has been used to characterize crystalline materials with sizes that range from 50 to 100 nm size range. This has been made possible by the latest developments in nanotechnology. Transmission electron microscope is often used to analyze nanocrystalline materials because of their high angular and spatial resolutions. Although it has resolution limitation, EBSD has been widely used for analyzing crystalline materials with grain sizes ranging from centimeters to submicrons. However, EBSD has the limit of spatial resolution, which is determined by the nature of backscattering electrons in a 70° tilted condition (Humphreys, 2004). The nature of backscattering electrons is influenced by the backscattering coefficient, electron beam size (Isabell & Dravid, 1997; Dingley, 2004), and the excitation volume in the materials (Ren et al., 1998; Schwarzer et al., 2009).

Two kinds of spatial resolution are defined: the physical resolution (Zaefferer, 2007) of the distance from a high-angle grain boundary and the effective resolution (Humphreys et al., 1999) of the distance across a boundary, over which patterns cannot be indexed by software algorithms. Many techniques have been developed to improve the spatial resolution of EBSD. Compared with a tungsten scanning electron microscopy (SEM), the application of the field emission gun SEM significantly increases the effective spatial resolution by up to 30 nm (Humphreys, 2004). Another approach using hardware modification is to employ the energy filter in selecting the energy range of the backscattered electrons (Deal et al., 2008), which enhances the pattern contrast. Lowering accelerating voltage is an alternative to improve the spatial resolution, which works by reducing the interaction volume; thus far, the best recorded

value of lateral resolution of 10 nm has been obtained at 7.5 kV for TWIP steel (Steinmetz & Zaefferer, 2010).

The purpose of the current study is to understand the effect of the atomic mass on the spatial resolution of EBSD. A cross-correlation method was combined with the EBSD technique to determine the physical spatial resolution, under operational conditions of accelerating the voltage and probe current for silver (Ag) and aluminum (Al).

EXPERIMENTS

In order to obtain crystals for resolution determination, 99.99% pure Ag and Al were annealed at 985 and 680°C, respectively, for 6 h. Both bicrystals were slowly cooled down to room temperature. Ag and Al bicrystals were mechanically polished to 0.3 μm Al_2O_3 and then electro-polished afterwards. The Al bicrystal was electro-polished using an electrolyte consisting of 100 mL H_3ClO_4 and 400 mL $\text{C}_2\text{H}_5\text{OH}$ (ASTM, 1999) at 20 V for 60 s. Meanwhile, the Ag bicrystal was electro-polished using a cyanide-free electrolyte (Lyles et al., 1978) consisting of 115 mL glacial acetic acid, 43 mL H_2SO_4 , 350 mL anhydrous methyl alcohol, and 77 g thiourea at 9 V for 90 s.

EBSD measurements were conducted on a JOEL 7001F SEM with Schottky field-emission gun equipped with EDAX/TSL EBSD system and a digital camera. All samples were tilted to 70° and observed at a working distance of 15 mm. Camera exposure time was set to 0.5 s at a resolution of 910×910 pixels to ensure the least noise. The accelerating voltage was varied from 5 to 30 kV in increments of 5 kV. Probe currents of 1 nA (aperture diameter = 30 μm), 10 nA (30 μm), and 40 nA (50 μm) were selected. The beam was moved in steps of 3 nm for all EBSD measurements.

Spatial resolution may be defined as the lateral resolution (parallel to the tilt axis) and the longitudinal resolution (perpendicular to the tilt axis). Lateral scanning means that

Figure 1. a: Normalized correlation coefficient of $C_L(x)$, $C_R(x)$, and overlap coefficient (x) as a function of distance. b: The Kikuchi patterns at positions of A_1 , A_2 , and A_3 .

the grain boundary on the bicrystal is parallel to the incident electron beam, whereas the longitudinal scanning means that the grain boundary is perpendicular to the incident beam. By scanning across the grain boundary on the bicrystals, Kikuchi patterns were recorded on the disk for further analysis. The lateral and longitudinal resolutions were then calculated from these patterns with the above-mentioned overlap coefficient.

RESULTS AND DISCUSSION

A quantitative method was adopted to investigate the effect of accelerating voltage and probe current on the physical resolution. This method is based on the similarity or the correlation coefficient of two Kikuchi patterns using a digital image correlation (DIC) technique. An overlapping Kikuchi pattern at A_2 (Fig. 1b) occurred when an incident beam came close to a grain boundary, and the correlation coefficient was reduced. Figure 1a shows the process of determining the spatial resolution using DIC technique for Ag at 20 kV and 10 nA. Two reference patterns were selected at the left and right end of a scanning line, respectively. The correlation coefficients $C_L(x)$ and $C_R(x)$ across the boundary were obtained with respect to the reference pattern at the left and right ends, respectively (Fig. 1a). The correlation coefficient $C(x)$ is given by

$$C(x) = \frac{\sum g_{ij} \tilde{g}_{ij}}{\sqrt{\sum g_{ij}^2 \sum \tilde{g}_{ij}^2}}, \quad (1)$$

Figure 2. (a) Lateral and (b) longitudinal resolutions that are dependent on accelerating voltages of 1, 10, and 40 nA for Al and Ag, respectively. The dotted lines indicate the results calculated from Monte Carlo simulation.

where g_{ij} and $\tilde{g}_{ij}$ stands for gray scales in the reference and sampling images at each position of i and j , respectively.

Once the $C_L(x)$ and $C_R(x)$ have been normalized, their coefficient and multiplication are defined as an overlap coefficient $C_{\text{overlap}}(x)$ in Figure 1a:

$$C_{\text{overlap}}(x) = C_L(x) \times C_R(x). \quad (2)$$

Three Kikuchi patterns at positions A_1 , A_2 , and A_3 , respectively, are shown in Figure 1b. The overlap coefficient at A_2 is the highest peak in this profile, and the coefficients at A_1 and A_3 are zero, without overlap. Finally, the Gaussian fitting function was used to calculate the full-width at half-maximum (FWHM) of the overlap coefficient curve. In the current study, the spatial or longitudinal resolution is defined as the FWHM of each peak.

In addition to experimental measurement, Monte Carlo simulation using CASINO software (Drouin et al., 2007) was employed to determine the spatial resolution, in which 5,000 electrons with 10 nm spot size were used. Given that the backscattered electron with 80% energy of the incident beam still contributed to the patterns (Deal et al., 2008), 75% cutoff energy of the incident beam was used to determine the spatial resolution.

Figure 3. Image quality maps of deposited copper at (a) 10 kV and (c) 30 kV. Inverse pole figure maps in terms of the normal direction at (b) 10 kV and (d) 30 kV. All measurements were conducted at a probe current of 10 nA.

The results of the lateral resolution are presented as functions of accelerating voltage for probe currents of 1, 10, and 40 nA (Fig. 2a). Considering the case of Al, the lateral resolution stayed constant below 10 kV and decreased when the accelerating voltage decreased from 30 to 10 kV. The best lateral resolution for Al was 40.5 nm, which was obtained at 10 kV and 1 nA. For Ag, the lateral resolution was almost the same, and the optimal lateral resolution of 12.1 nm was also obtained at 10 kV and 1 nA. The results of the Monte Carlo simulation are consistent with those of the experiments: the spatial resolution decreased with the decrease of the accelerating voltage in the range of 10–30 kV and is nearly independent of the probe current. However, the lateral resolution for experiments and simulations exhibited a large difference when the accelerating voltage is below 10 kV. The difference may be explained by the effect of the spherical aberration, because this problem is not considered for Monte Carlo simulation. Decreasing the acceleration voltage increases the electron beam diameter, indicating that the spatial resolution becomes worse (Babin et al., 2009).

The results of the longitudinal resolution are presented as functions of accelerating voltage for probe currents of 1, 10, and 40 nA (Fig. 2b). In the case of Al, the longitudinal resolution decreased as the accelerating voltage decreased from 30 to 5 kV; moreover, it was almost independent of the probe current. The best lateral resolution for Al was 80 nm, which was obtained at 10 kV and 1 nA. The lateral resolution for Ag was almost the same, and the optimal lateral resolution of 23.2 nm was also obtained at 10 kV and 1 nA.

Two operating conditions at 10 kV and 10 nA as well as at 30 kV and 10 nA were performed on an electroplated copper to display the effect of the resolution on the orientation image map. Figure 3 shows the image quality (IQ) map and inverse pole figure (IPF) map in terms of the normal direction. Compared with the marked area I_1 at 10 kV, a major difference was observed at the marked area I_2 in the IQ map obtained at 30 kV in that the two grains were not easily distinguished from one another (Figs. 3a, 3c). Compared with the marked area Q_1 at 10 kV, an elongated grain at the marked area Q_2 in the IPF map was obtained at 30 kV (Figs. 3b, 3d). This can be attributed to the increase of accelerating voltage, which caused the size of the interaction region to increase in a particular longitudinal direction. This effect is more pronounced in a material with a low atomic mass, such as Al. Therefore, the interaction zone can be considered as a probe tip of EBSD. Generally, the dimensions of the interaction zone are defined as the ratio of the longitudinal to lateral resolution corresponding to the height to width ratio (Fig. 4). In our results, the ratio was larger than the theoretical value of 2.9, because the accelerating

Figure 4. Y/X ratio of longitudinal (Y) to lateral (X) resolution, which is dependent on the accelerating voltage at 10 nA. The longitudinal resolution is Y , and the lateral resolution is X .

voltage was larger than 10 and 30 kV for Al and Ag, respectively. This means that the interaction zone extended more in the longitudinal direction as the accelerating voltage increased. This is consistent with the results discussed above.

CONCLUSIONS

The best lateral resolutions of 40.5 and 12.1 nm were obtained at 10 kV and 1 nA for Al and Ag, respectively. In addition, the best longitudinal resolutions of 23.2 and 80 nm were obtained at 10 kV and 1 nA for aluminum and silver, respectively. In decreasing the accelerating voltage, the lateral and longitudinal resolutions significantly improved for a material with low atomic mass, while it is independent of the accelerating voltage for a material with high atomic mass. Finally, the probe current does not have any significant influence on the lateral and longitudinal resolution in both cases.

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