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Observations of Al segregation around dislocations in AlGaN

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Transmission electron microscopy has been used to observe Al segregation around the threading dislocations in Al 0.1 Ga 0.9 N and Al 0.3 Ga 0.7 N grown by metalorganic chemical vapor deposition on 6H–SiC. Dislocation lines were found to have up to 70% more Al concentration than those regions free of dislocations in the matrix. The Al-depleted regions around the dislocations are shown to be within a few nanometers from the dislocation lines. The results also show that more Al segregate to edge dislocations than to screw ones. © 2001 American Institute of Physics.

GaN is a wide band gap material and has many excellent properties for optical and electronic applications, Al x Ga 1−x N (x = 0.1–0.3) has been often used as the cladding layer or quantum well layer in GaN-related devices of blue light-emitting diodes, laser diodes, and power transistors. The quantum well layer in GaN-related devices of blue light-emitting diodes, laser diodes, and power transistors.1,2 The materials used were triethylaluminium, triethylgallium, and NH3. The cross sectional TEM specimens were prepared by mechanical thinning, and ion milling by Ar ion beam to perforation. TEM observations were carried out in a JEOL JEM 2010F microscope with a field-emission gun which can form an electron probe in a 0.5 nm diameter size. The operating voltage was set at 200 kV. The compositions were obtained by EDX from an Oxford Instrument EDX detector with an ultrathin window and EELS from a Gatan energy filter. All necessary precautions for quantitative analysis of compositions had been taken before the acquisition of the spectra. The compositional measurements were taken from regions with thickness less than 20 nm where high-resolution TEM images could be revealed, so that the thin-film criterion for quantitative analysis is satisfied without the need for correction of the absorption effect. The beam broadening size is estimated to be less than 2 nm. The orientations of the specimens during the EDX acquisition were far from the zone axes and two-beam directions to avoid any significant channeling and absorption effect. The concentrations were determined by assuming that the nitrogen fraction is constant, and the sum of Ga and Al are 100% in total. The intensities of Al Kα and Ga Kα lines, and the calculated k factor provided by the program of quantitative analysis of EDX are used for composition determination. The statistic error of each averaged composition is determined at the 99% confidence level by using t distribution.

Typical bright-field TEM images of Al 0.1 Ga 0.9 N and Al 0.3 Ga 0.7 N samples with the incident beam near a [1 1 00] zone axis are shown in Fig. 1. The dislocations grown from

(a)

(b)

FIG. 1. Bright-field TEM micrographs showing dislocations in (a) Al 0.1 Ga 0.9 N and (b) Al 0.3 Ga 0.7 N are shown.

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the interface between AlGaN and SiC are clearly seen. The dislocation density of Al$_{0.3}$Ga$_{0.7}$N is estimated in the range of $10^9$ cm$^{-2}$, and the mean dislocation spacing is about 300 nm. For the Al$_{0.1}$Ga$_{0.9}$N samples, the dislocation density is of the order of $10^{10}$ cm$^{-2}$ with the mean dislocation spacing of about 40 nm. These threading dislocations grown in the AlGaN layer are originated from the interface with SiC during MOCVD deposition due to the lattice misfit between AlGaN and SiC. The conventional invisibility criterion (where $\mathbf{g} \cdot \mathbf{b} = 0$) was used to determine the dislocation type and the Burgers’ vectors. The edge type dislocations have the Burger’s vector $1/3 \langle 1120 \rangle$, the screw $[0001]$, and the mixed $1/3 \langle 11\bar{2}3 \rangle$. Detailed examination of the diffraction patterns confirms that no precipitate and/or phase separation exist in the AlGaN layer. A high-resolution TEM image in Fig. 2 also shows partial dislocations without the presence of second phase. Thus, it is certain that the dark line of contrast in the TEM images is mainly due to the strain field of a dislocation in the AlGaN layer. Dark-field imaging by the weak beam technique also confirms this point.

EDX from areas probed by a micron-size electron beam on Al$_{0.3}$Ga$_{0.7}$N samples shows that the averaged Al concentration is 29.0 $\pm$ 2.0 at% close to the nominal value of 30%. However, nanobeam measurements show that the matrix free of dislocations has only 21.0 $\pm$ 1.2 at% Al. The EDX spectra from an edge dislocation and a region in the matrix free of dislocations in Al$_{0.3}$Ga$_{0.7}$N, acquired at the same electron beam condition, are superimposed as shown in Fig. 3(a). These spectra were obtained in the Al$_{0.3}$Ga$_{0.7}$N layer at a distance of more than 60 nm away from the interface between AlGaN and SiC. With normalization of the intensities in these two spectra with respect to the Ga $K$ lines which have been adjusted to the same height, it is obvious that the Al intensity is much higher at the dislocation. The Al concentration is 36.0 $\pm$ 1.9 at% in average at the edge dislocation lines. Compared with the matrix, the dislocation core in Al$_{0.3}$Ga$_{0.7}$N can be enriched with more than 70% increase of Al. The Si peak is believed to come from the so-called Si internal fluorescence peak due to the dead layer of the Si(Li) crystal in the EDX detector, and partly from the doping of Si in deposition. Since the distribution of Si intensity is uniform over the AlGaN layer, the effect on the results can be ignored. For the Al$_{0.1}$Ga$_{0.9}$N case, Fig. 3(b) also shows the Al enrichment at an edge dislocation in comparison with the matrix. The matrix contains 9.6 $\pm$ 0.3 at% Al, while there exists about 15.6 $\pm$ 1.0 at% Al at the edge dislocations. The Al composition profiles across the edge dislocations in both alloys are demonstrated in Fig. 4. It is apparent that Al is enriched around the dislocation cores, but depleted at both sides in 2–3 nm regions. The depletion of Al to 4–6 at% is very significant in Al$_{0.1}$Ga$_{0.9}$N, whereas it is about 15 at% in Al$_{0.3}$Ga$_{0.7}$N. It is likely that the Al concentration at the dislocation cores may be much higher than the measured values if the beam broadening effect could be corrected. More than five dislocations characterized in each alloy show the similar profiles. The EELS maps in higher spatial resolution also confirm Al segregation at the dislocations. Measurements at the screw and mixed dislocations indicate that the enriched Al concentration is about 31 and 34 at% in average, respectively, in Al$_{0.3}$Ga$_{0.7}$N, whereas it is 12.2 and 13 at% in Al$_{0.1}$Ga$_{0.9}$N. The Al segregation at the screw type dislocations is less severe than at the edge type. For the mixed dislocations, it lies between the edge and the screw types. These results show similar behaviors of Al segregation around dislocations in both Al$_{0.1}$Ga$_{0.9}$N and Al$_{0.3}$Ga$_{0.7}$N alloys.

Solute segregation to dislocations is often observed in metallic alloys, for example, carbon atoms in α-iron can...
alloy. The presence of Al segregation around dislocations may pin the dislocation lines, resulting in difficulty of movement. As a consequence, the dislocation density may not be easily reduced. Thus, the process modification is necessary if a low dislocation density is desired for the AlGaN layer.

In summary, Al segregation around the dislocations in AlGaN alloys grown on 6H–SiC has been observed by TEM. The Al concentration enriched at the dislocation core can be more than 1.6 times as high as that in the matrix. The Al-enriched level depends on the dislocation type, which the strongest is around the edge type, the next is around the screw type and the weakest is around the screw type. Also, Al-depleted regions surrounded the enriched dislocation cores have been revealed to be a few nanometers away.

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