Tree-like features formed on photoelectrochemically etched n-GaN surfaces
——— Revelation of threading dislocations in GaN ———

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Dislocation density is one of the important factors in the evaluation of crystalline quality of semiconductor materials. In order to reveal dislocations as etch pits, wet chemical etching is widely used for Si and conventional III-V semiconductors. In contrast with those semiconductors, chemical stability for III nitrides makes it difficult to use wet chemical etching for etch pit formation. Therefore, a simple and rapid method to reveal dislocations is highly desired for III nitrides. It has been demonstrated that dislocations in n-type semiconductor crystals can also be detected as protrusions or hillocks by photoelectrochemical etching [1,2]. This method was successfully applies to n-InP [2], showing that the method has a higher resolution and a higher sensitivity for defect delineation than the conventional wet chemical etching. The mechanism for the dislocation detection as a protrusion is based on recombination of photo-generated holes at dislocation sites. Since the electrochemical dissolution reaction needs holes, reduced concentration of holes at a dislocation site results in a reduced dissolution rate and, therefore, makes a protrusion there. Such a revelation of defects by the photoelectrochemical etching can be done under the condition of a lower current density and a higher light intensity [1]. Provided that dislocations in III nitrides act as nonradiative recombination centers for holes, it is possible to apply photoelectrochemical etching to reveal dislocations in III nitrides. Recently, dislocations in III nitrides have been thought to act as nonradiative recombination centers [3], although, in the early stage of research, they were reported not to be nonradiative recombination centers because of the weak dependence of luminescence efficiency on dislocation density in GaN. In this paper, we report photoelectrochemical etching of n-GaN. Tree-like features are formed on photoelectrochemically etched n-GaN surfaces. The features are attributed to threading dislocations. This means that dislocations in GaN act as recombination centers of holes. Thus, the photoelectrochemical etching is found to become a convenient method to detect dislocations in III nitrides.

Photoelectrochemical etching of n-type GaN was performed using a 0.1 mol/l NaOH solution at 20°C. N-type GaN films of 2 μm thick were grown on α-Al_2O_3(0001) with MOVPE method using TEGa and NH_3 as source gases and N_2 carrier gas. Grown GaN had a carrier concentration of $2x10^{17}$-$2x10^{18}$ cm^{-3}. Special procedures have not been done to reduce dislocation density in grown GaN films. The etching was made galvanostatically with a current density of 0.5 mA/cm^2. A He-Cd laser with 325 nm wavelength and 100 mW output power was irradiated to the film surface through the solution.

Figure 1 shows SEM images of etched GaN surfaces. After the etching for 30 min (Fig. 1(a)), “grass-like” features are formed on the etched surface. This non-uniform etching shows some kind of inhomogeneity involved in the GaN layer. By magnifying the image of Fig. 1(a), one can see that the grass-like features consist of many trees standing perpendicularly to the film surface (Fig.1(b)). The density of trees is counted to be about $10^{10}/cm^2$. The shape and the density ($\sim 10^{10}/cm^2$) of trees strongly suggest that they are due to threading dislocations involved in n-GaN. Adesida et al. [4] observed whiskers very similar to the trees obtained here. They also confirmed all whiskers to contain threading dislocations using TEM observation. The very fine structures of the trees are seen in Fig. 1(b). As reported previously [5], fineness of formed protrusions should be governed by hole diffusion length; A shorter diffusion length gives a finer shape of protrusion. The existence of a thin needle with a diameter less than 0.1 μm in Fig. 1(b) suggests a very short diffusion length of holes in the sample. With increasing etching time, i.e., etching depth, etched surface becomes smooth as shown in Fig.1(c) and (d).

Especially, a very smooth surface is obtained when the etching proceeds to dissolve almost all of the film. How did the trees formed at the early stage of the etching disappear? The trees are removed by breaking away when the part of GaN layer near the substrate, which is easily dissolved electro-chemically without UV irradiation [6], is dissolved. Some wrecks of “trees” are found on the etched surface shown in Fig. 1(c).

In summary, we have studied photoelectrochemical etching of n-GaN. Tree-like features are formed on etched n-GaN surfaces. The trees with a density of about 10^{10} cm^{-2} are attributed to threading dislocations. This fact shows that dislocations in GaN act as recombination centers of holes. Thus, the photoelectrochemical etching is found to be a convenient method to detect dislocations in III nitrides.

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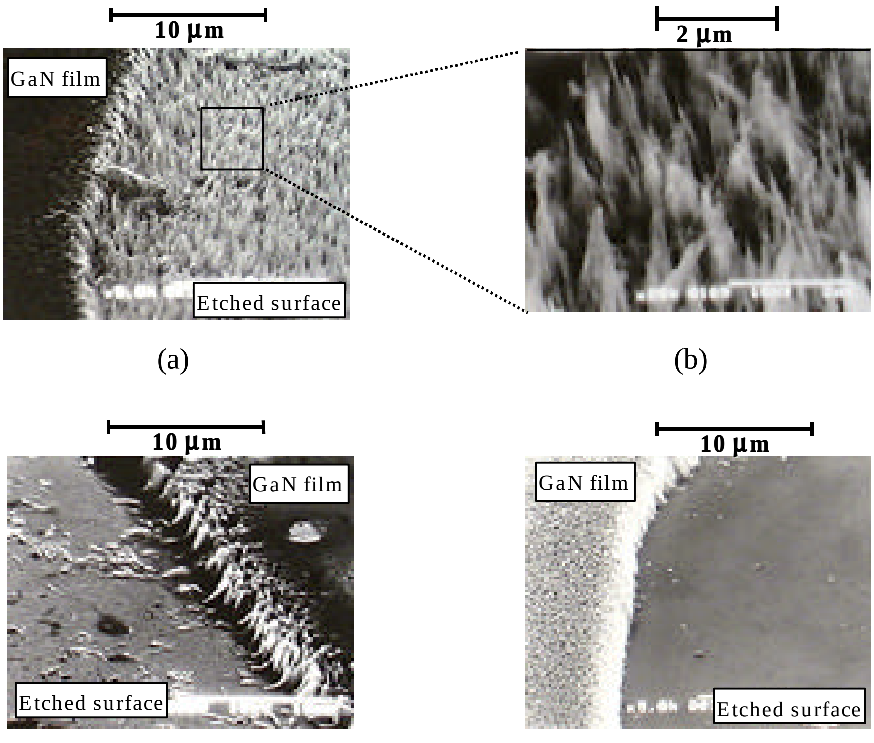

(a) (b)

(c) (d)

Fig. 1. SEM images of photoelectrochemically etched n-GaN for a different time.
(a) 30min, (b) 30min (magnification of (a)), (c) 105 min, (d) 120 min.