Letters

AlN film containing Si

Metal nitride films have been studied because they are useful in solid-state devices. Of these nitride films (BN, AlN, Si_3N_4, etc), AlN has attracted most attention and several investigators have reported success in epitaxial growth [1–4]. It is difficult to dope impurities into such nitrides of wide band-gap, and this note reports on the preparation of AlN film containing Si atoms, referred to as δ-AlN, because the lattice constants change from AlN, while the structure remains as the wurtzite type.

It was reported [5] that AlN film was prepared by nitriding evaporated Al film with d.c. glow discharge in a pure N_2 atmosphere using an Al plate as cathode; the δ-AlN film was obtained by the same method using an Si cathode. Structure and composition of the film were observed by the transmission electron diffraction and by electron probe microanalyser (EPMA). The film was prepared in a bakeable vacuum chamber made of stainless steel. NaCl single crystals (cleaved in air) were used as the substrates, onto which Al was evaporated at 200 to 300° C and about 5×10^{-9} torr (the pressure rose up to about 5×10^{-8} torr during evaporation). The thickness of the Al film was ranged between 200 and 600 Å. N_2 was admitted through a variable-leak valve and discharge was started at about 6×10^{-2} torr. The discharge current and voltage were 2 to 4 mA and 1 to 2 kV respectively. The distance of cathode from substrate was about 15 mm, and the substrate temperature was 200 to 400° C. The Al film was exposed to glow discharge under these conditions for 30 to 60 min. The structure of the film was greatly affected by these parameters, the thickness of nitride films obtained being estimated to be 100 to 300 Å.

A typical diffraction pattern taken from the film obtained under the above-mentioned conditions is shown in Fig. 1. The diffraction pattern indicates that the Al film grows with a definite orientation to the NaCl substrate. The orientation of AlN and δ-AlN grown on the Al film are affected by that of the Al film [6], and it is easily seen that the δ-AlN film had the same structure and orientation as that of the AlN film. Fig. 2 shows an electron micrograph of the film. Moiré fringes observed near the bend contours of Al grain are composed of Al (2 2 0) and δ-AlN (1 1 0) diffraction lines, and their spacing agrees with that calculated from *d*-values of Al (2 2 0) and δ-AlN (1 1 0).

The existence of Si in the film was confirmed by EPMA, and showed that Si existed only in the film prepared on the Si cathode and that the amount of Si increased with the discharging current. Because the film was very thin the accuracy of EPMA measurement was not good, and so far the ratio of Si/Al in δ-AlN film is not confirmed.

According to a recent report [7], AlN with an expanded *c*-dimension was obtained as a diffuse phase in Si–Al–O–N system (called sialon). The

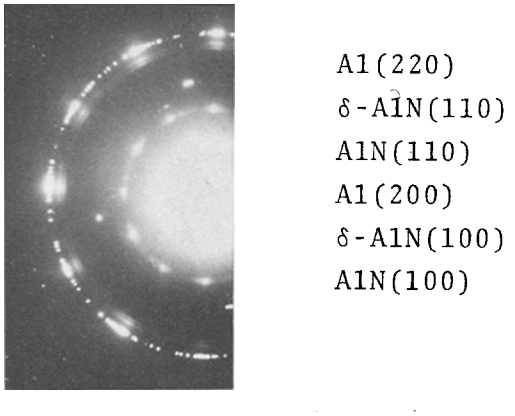

Figure 1 An electron diffraction pattern taken from AlN and δ-AlN films on Al film.

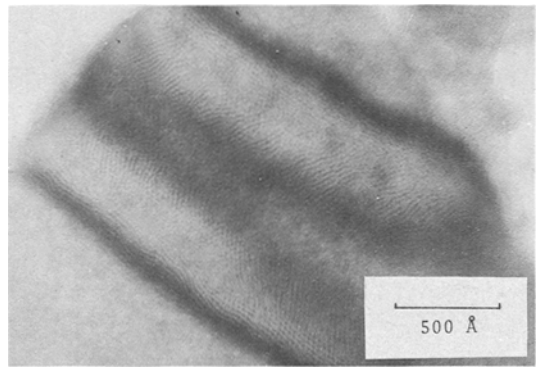

Figure 2 Electron microscope photograph of the same film as Fig. 1.

TABLE I Lattice constants of AlN, AlN^{δ} and δ-AlN

	a-axis (Å)	*c*-axis (Å)
AlN [8]	3.114	4.986
AlN^{δ} [7]	3.079	5.30
δ-AlN (present)	3.07 ± 0.01	5.2 ± 0.1

diffuse phase, named AlN^{δ}, has a wurtzite structure like AlN, the lattice constants being elongated in the *c*-direction and shrunk in the *a*-directions compared with AlN crystal. The lattice constants obtained for δ-AlN are tabulated with those of AlN^{δ} and AlN in Table I. These results suggest that δ-AlN (the present film) may be the same as AlN^{δ} (Jack's result). The existence of oxygen in δ-AlN film could not be confirmed. Considering the chemical activities of Si and Al to oxygen, however, it is plausible to consider that oxygen was taken into the film from residual gas and that δ-AlN film was constituted with the Al–Si–N–O system.

The definite splitting of the diffraction spots of AlN and δ-AlN in Fig. 1 suggests that Si atoms occupy the substitutional site rather than the intersitial site in AlN. It is naturally considered from their ionic radii that the O atom substitutes for N. It seems difficult, however, to decide whether Si substitutes for Al or N.

In conclusion, we have confirmed that Si can be doped rather easily into AlN crystal by using a d.c. glow discharge method. The formation mechanism of nitride film by this method is considered to be as follows; the surface of AlN film reacts with active nitrogen created by the glow discharge while the cathode materials (Si) are sputtered onto substrate. Then the AlN film including Si grows on the Al film. This suggests that another metal impurity may be doped into AlN by this method, which would be appropriate for searching the materials dopable into AlN film. The δ-AlN film is an electrically good insulator like AlN film, and detailed experiments on electrical properties are now being carried out with metal–insulator–metal type diodes.

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YOICHIRO UEMURA
MINORU IWATA
National Institute for Researches in Inorganic Materials, Kurakake, Sakura-Mura, Ibaraki-Ken, Japan

On carbide cracking as a source of acoustic emission in steel

Although Acoustic Emission (AE) detection is being widely used as a non-destructive-testing technique, especially in flaw location, the microscopic sources of emission are not generally well understood. For example, in steels of a ferrite/cementite microstructure the role of carbide cracking in generating detectable emission is unclear [1–4]. The present study was carried out to help clarify this particular problem.

Uniaxial tensile tests, using specimens of dimensions shown in Fig. 1a, were carried out on a spherodized steel of composition:

C	Mn	Si	P	S
1.10	0.17	0.16	0.009	0.009
Cr	Cu	Ni	Sn	Al(tot)
0.05	0.15	0.06	0.02	0.03

After machining, the specimens were vacuum heat-treated at 680° C for 3 h followed by furnace cooling. The mechanical testing was carried out at room temperature in an Instron floor model tensile-testing machine at a cross-head displacement rate of 2 mm min^{-1}. Simultaneous AE measurements were made on all specimens using a