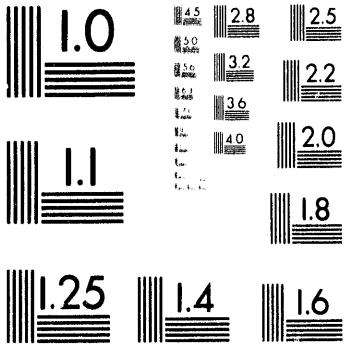
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Electron microscopy of AlN-SiC interfaces and solid solutions

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ABSTRACT: In a 2H AlN-SiC solid solution grown by MBE on α(6H)-SiC (3° from [0001]), the epilayer contained a high density of basal faults related to ~5 nm steps on the growth surface; no compositional inhomogeneity was detected by PEELS. In diffusion couples of polycrystalline, sintered AlN on SiC annealed at 1600 and 1700°C, 8H sialon [nominally $(AlN)_2Al_2O_3$] formed at the interface of SiC and recrystallized epitactic AlN grains, and Si_3N_4-rich β′ sialon particles formed in the SiC. No interdiffusion was detected by PEELS in diffusion couples of MBE-grown AlN on SiC annealed at 1700 and 1850°C. Irregular epilayer thickness explains companion Auger depth profile results.

1. INTRODUCTION

Aluminum nitride and silicon carbide are structural ceramics and wide bandgap semiconductors which have similar hexagonal structures and a lattice mismatch in the basal plane of only 0.9%. AlN-SiC materials are of interest as ceramic semiconductors (high power and ultraviolet optoelectronic devices, use in severe environments) and as high temperature structural materials. The tentative AlN-SiC phase diagram of Zangvil and Ruh (1988) shows a flat miscibility gap at ~1950°C between ~20 and 80 % AlN with a 2H solid solution above this temperature. The existence of the miscibility gap has been confirmed by several investigators (see Kern et al. 1993), notably Chen et al. (1992). Interdiffusion data exist for 1950°C and higher.

2. EXPERIMENTAL

Three AlN-SiC materials, each having α(6H)-SiC single crystal substrates (Cree Research) oriented 3-4° off (0001) toward [11$\bar{2}$0], have been examined in cross section by analytical and high-resolution electron microscopy (AEM and HREM): (1) an AlN-SiC solid solution grown at 1050°C by plasma-assisted, gas-source molecular beam epitaxy (MBE) (Kern et al. 1993); (2) diffusion couples of polycrystalline sintered AlN (Dow Chemical) on the above specified SiC substrate, annealed at 1600 and 1700°C; and (3) diffusion couples of MBE-grown AlN on SiC annealed for 70 h at 1700°C or 25 h at 1850°C. High-spatial-resolution parallel-detection electron energy-loss spectrometry (PEELS) was performed at 100 kV with a Philips EM400T equipped with a field emission gun and Gatan 666 spectrometer. Specimens were usually cooled to -130°C in Gatan cooling holders and spectra acquired with probes of 2-nm diameter (FWTM), ~1 nA, and incident and collection half-angles of 8 and 19 (or 30) mrad. Conventional diffraction contrast and HREM was performed with Topcon 002B (NCSU) and Philips CM12 and CM30 instruments.

3. RESULTS

Examination of the AlN-SiC solid solution epilayer grown by MBE at 1050°C revealed a high defect density (Fig. 1a). The predominant polytype was 2H determined by HREM and

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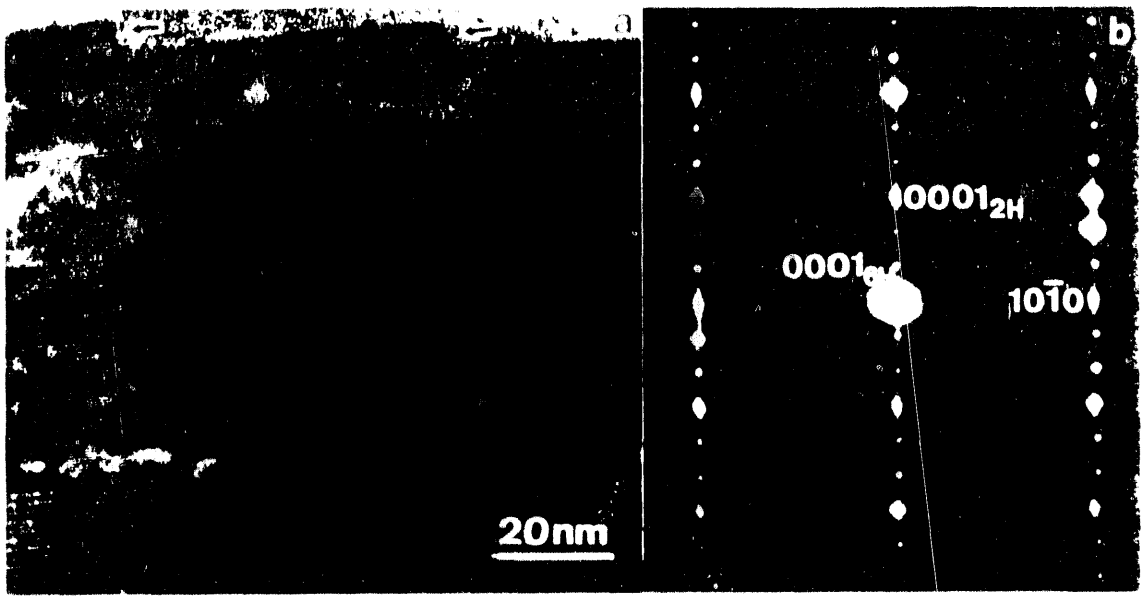

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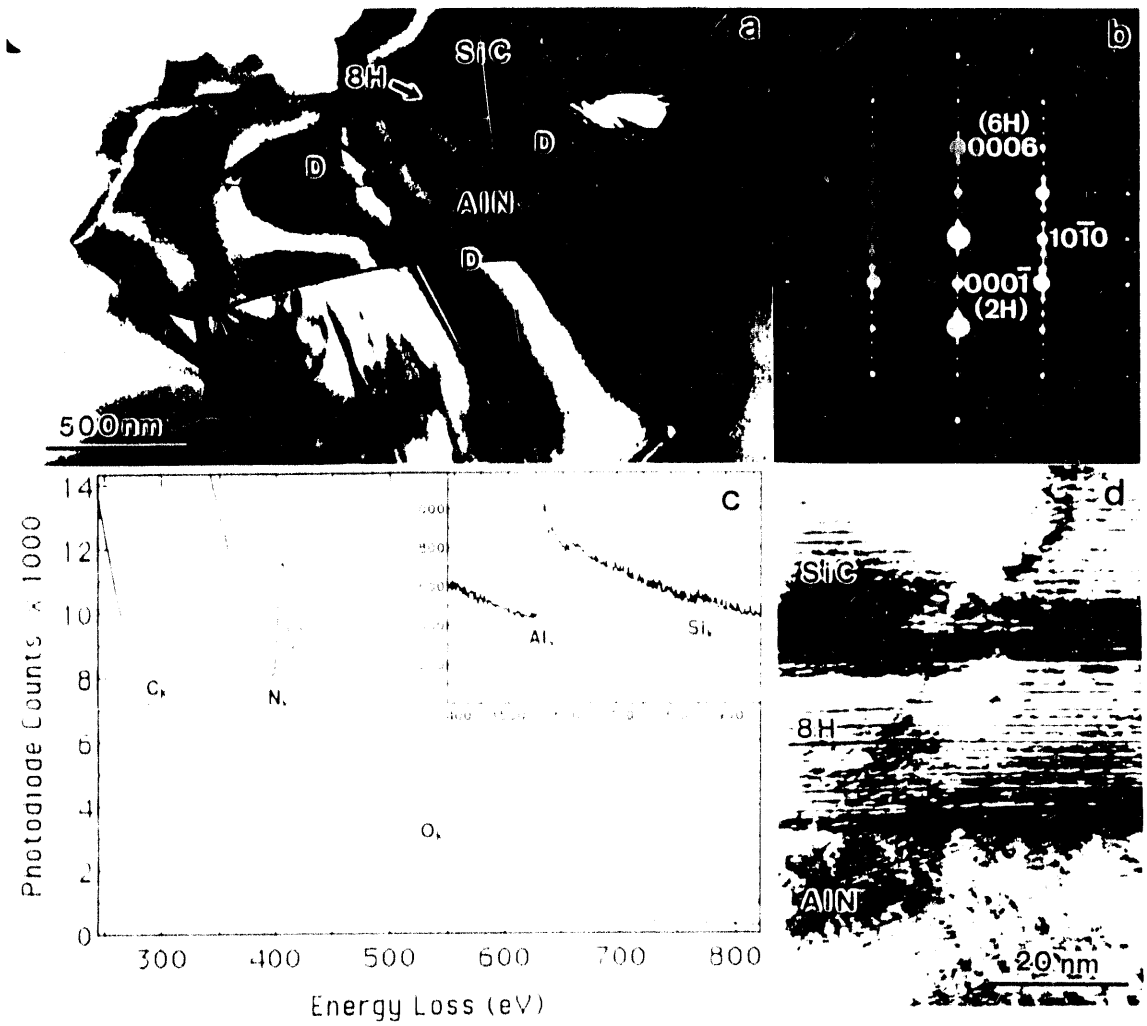

Fig. 2. Diffusion couple of polycrystalline sintered AlN and α(6H)-SiC annealed at 1700°C. (a) AlN grain containing dome defects (D) epitactic to SiC, and 8H sialon interface phase. (b) [11$\bar{2}$0] SAD pattern of the three epitactic phases. (c) PEELS analysis of interface phase. (d) Beam damage and 2.3-nm periodicity of 8H sialon interface phase.

If the small Si signal in Fig. 2c is intrinsic, rather than due to a surface sputtered film (from the SiC), the phase would be consistent with the 8H sialon phase (Jack 1976). The oxygen presumably originates as a surface impurity on the starting materials. It is not clear whether the epitactic orientation is controlling the interface phase formation or vice versa. However, it seems highly unlikely that several AlN grains in a TEM specimen would be oriented for exact epitaxy with the SiC, suggesting recrystallization of AlN (2H) from the 8H sialon. Interestingly, this would imply that the dome defects, prevalent in the epitactically oriented grains, nucleate at the dome top, increase in diameter as the grain boundary advances, and eventually pinch off at the oxygen-rich basal fault.

No aluminum was detected in the SiC, even adjacent to the interface, but particles typically 250 nm in diameter (Fig. 3a), and occasionally up to a micrometer long, form within the SiC at distances up to several micrometers from the SiC-AlN interface. Quantitative analysis of typical PEELS data (Fig. 3c) yields Si_3N_4 with small levels of O and Al. Diffraction patterns (Fig. 3b) confirm the Si_3N_4-rich β′ sialon identification and show $[0001]_{\beta'}$ $[11\bar{2}0]_{6H}$ and $[10\bar{1}0]_{\beta'}$ $[0006]_{6H}$.

High spatial resolution PEELS failed to detect any interdiffusion in the diffusion couples of AlN grown by MBE on α(6H)-SiC and annealed at 1700 or 1850°C, either near the substrate interface

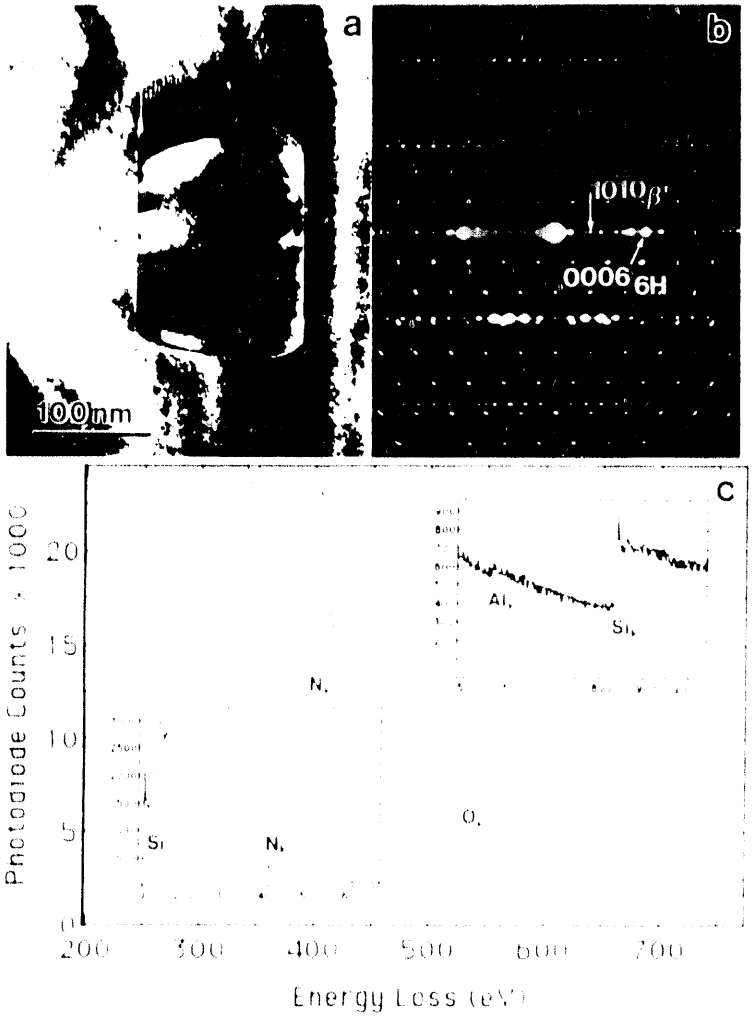

Fig. 3 (a) Si_3N_4 rich β' sialon particle in SiC matrix (b) SAD pattern (c) PEELS analysis of β' sialon

Fig. 4 Diffusion couple of MBE-grown AlN on α(6H)-SiC annealed at 1850°C for 25 h showing growth-surface steps, large variations in epilayer thickness, threading dislocations, and absence of faults (cf. Fig. 1a)

or at threading dislocations in the epilayer (Fig. 4). Earlier Auger depth profiles had indicated apparent substantial interdiffusion. However, the large growth-surface steps and concomitantly large variations in epilayer thickness, shown in Fig. 4, explains the misleading Auger results.

ACKNOWLEDGEMENT

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