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ION BEAM SPUTTERED AlO_xN_y ENCAPSULATING FILMS

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Technical Report: April 1979
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by

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The encapsulating properties of 800-1500 Å aluminum oxynitride films, deposited on GaAs by low energy ion beam sputtering, were characterized by optical microscopy, electrical conductivity, Auger profiling, and ellipsometry. The better films were found to withstand annealing to above 900°C with minimal physical deterioration.

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ABSTRACT

The encapsulating properties of 800-1500 Å aluminum oxynitride (AlO_xN_y) films, deposited on GaAs by low energy ion beam sputtering, were studied over a range of y from 0.1 to 0.8. Particular attention was given to chemical and sputter cleaning procedures. The structures were characterized by optical microscopy, electrical conductivity, Auger profiling, and ellipsometry. The better films were found to withstand annealing to above 900°C with minimal physical deterioration. The films with a higher proportion of oxygen allowed some oxygen diffusion; those made with inferior cleaning procedures an out-diffusion of arsenic.

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I. INTRODUCTION

Dielectric encapsulating films play an important role in the processing of ion implanted GaAs devices,[1-3] such as field effect transistors, light emitting diodes and radiation detectors. After implantation, it is necessary to anneal the semiconductor to remove lattice damage introduced by the high energy ions. Since GaAs and other III-V materials tend to dissociate, in this case by arsenic effusion, at temperatures (~700°C) below useful annealing temperatures,[4] it is impossible to anneal bare samples without significant surface degradation. Thus, some procedure, such as encapsulation, is necessary.

There have been numerous studies of implantation and annealing of GaAs using encapsulants such as SiO_2,[5] Si_3N_4,[6-10] and AlN.[11-14] It has been demonstrated that silicon dioxide and silicon oxynitride layers allow gallium to out-diffuse from a GaAs surface.[10] Better surface protection is provided by Si_3N_4 films. These have been deposited by rf sputtering,[9] pyrolytic deposition,[15] rf plasma deposition,[8-10] and neutralized ion beam sputtering.[16] Pyrolytic nitride layers require high substrate temperatures (>700°C) during deposition,[15] plasma depositions considerably less (~350°C),[10] and sputtering only the order of 200°C. The ion beam technique further allows a large degree of versatility and process control.[16,18,19]

One rather fundamental difficulty with Si_3N_4 encapsulants is the diffusion of Si into the GaAs during annealing.[16] For this reason, we have chosen to investigate the more stably bonded aluminum nitride and aluminum oxynitride dielectrics. A further motivation for investigating AlN

is that rf sputter layers have been shown[17] to have a thermal expansion coefficient ($6.63 \times 10^{-6} \cdot C^{-1}$) reasonably close to that of GaAs.

The current study involves the identification of composition dependent encapsulation properties of films in the AlO_xN_y system and some comparison with SiO_xN_y films. All films are prepared by low energy neutralized ion beam sputter deposition onto GaAs, with the principle control on the composition being the nitrogen partial pressure in the vaccuum chamber. The primary tests for successful encapsulation are the observation of deterioration during annealing using optical microscopy and the profiling of diffusion using Auger analysis.

II. EXPERIMENTAL PROCEDURES

Aluminum oxynitride films were deposited using low energy neutralized ion beam sputtering.[18-19] A pure Al sheet (99.99%) was used as a target. The sputter beam consisted of argon and nitrogen ions and was injected with an equal number of electrons emitted from a hot tungsten filament. The sputter beam diameter was 5 cm. The films were simultaneously deposited onto GaAs substrates and precleaned Corning 7059 microscope slides which were partially covered with a thermally evaporated gold electrode. Typical deposition rates were 35-50 Å/minute, and final thicknesses were generally in the 1000 Å range. The films on the glass substrates were used to obtain transmissivity spectra and interferometric thickness measurements. Electrical resistivities and dielectric constants were obtained from the Au-aluminum oxynitride-Au sandwiches on the glass substrates. The thermally evaporated Au top electrodes on these sandwiches were circular, 1.67×10^{-6} m^2 in effective area, and ~1000 Å thick. The films on the GaAs substrates were

used for ellipsometric investigations and Auger electron spectroscopy studies before and after heat treatment.

The gallium arsenide substrates used were squares 1 cm on a side with <100> orientation. They were initially cleaned mechanically with detergent, rinsed through flowing deionized water, and blown dry with N_2. The samples were then chemically cleaned with acetone, methanol, xylene, deionized water, and finally Chemsol Z (registered trademark of Burns and Towne, Inc., 662 Cross St., Malden, MA 02148). Cleaned substrates were mounted into the vacuum system, which was typically evacuated to 3×10^{-7} Torr.

Early samples deposited on GaAs substrates were chemically cleaned only (i.e., no subsequent sputter cleaning of the substrate or the target was performed). The deposition proceeded after N_2 was introduced into the vacuum system and the deposition times for this group of samples were 15 and 30 minutes. Auger electron spectroscopy (AES) studies performed on the early samples revealed a large amount of oxygen in the films. Therefore, several modifications were made in the predeposition procedure. The target was sputter cleaned prior to the deposition for 35 minutes using a 1000 eV beam energy in an argon atmosphere of 5×10^{-5} Torr. The GaAs substrates were shielded during the target cleaning step. After sputter cleaning of the target, the substrate was then sputter cleaned for 5 minutes using a 300 eV argon beam in the same 5×10^{-5} Torr background pressure. The vacuum system was then pumped down to a pressure of less than 3×10^{-7} Torr before the sputtering gases were introduced. The sputter deposition was performed using an 800 eV beam energy and a current density of 2 mA/cm^2, while varying the argon-nitrogen mixture ratio. Deposition times were typically 20 or 30 minutes.

Films on the GaAs substrate were examined ellipsometrically in order to determine the refractive index, thickness, and surface homogeneity using a Gaertner Model L117 ellipsometer with 6328 Å radiation from a He-Ne gas laser. The complex index of refraction of the GaAs substrate was assumed to be 3.7 - 0.14 i when interpreting the ellipsometric data. The transmissivity spectra of the films on the glass substrates were examined by means of a Beckman DK-2A spectrophotometer, sensitive in the 1700 Å - 35,000 Å spectral region. Dielectric constants were obtained from capacitance measurements at 1 MHz using a Boonton Model 72B capacitance meter. Interferometric thicknesses of the films were determined using a Sloan Model M-100 interferometer. The Auger analysis was performed with a Physical Electronics, Inc. 548 ESCA/Auger system using a 3 KeV, 10 μA electron beam. Depth profiles were conducted in 5×10^{-5} Torr argon using a 2 KeV sputter beam to sputter at a rate of approximately 40 - 50 Å/minute. AES characterization and optical measurements of the later films indicate that there was always a measurable amount of oxygen present, later traced to a small leak in the argon gas line. Therefore, these films will henceforth be referred to as aluminum oxynitride (AlO_xN_y) rather than aluminum nitride.

Post deposition annealing of the AlO_xN_y encapsulants was performed in a flowing hydrogen atmosphere at temperatures between 400°C and 1000°C. The typical annealing time was 15 minutes. The samples were maintained in the flowing hydrogen atmosphere until they reached the ambient temperature. Thermal deterioration was monitored by optical microscopy and AES, comparing the initial samples to the heat treated samples.

III. RESULTS

The sputtered aluminum oxynitride films exhibit good adhesion, good optical homogeneity and an average transmission near 90% over the 2000-30,000 Å spectral range. The sample surfaces appear smooth under an optical microscope with 400x magnification. The dielectric constant, measured at 1 MHz and at room temperature using an Au-AlO_xN_y-Au sandwich, is the order of 4.5. The low electric field dc resistivity is in the 10^8-10^{10} Ω-cm range at room temperature and is relatively uniform across an individual film.

The refractive index of the sputtered films was found to be a strong function of the nitrogen to argon gas ratio during sputtering. This data is shown in Figure 1. The values for the poorer quality early depositions fall at somewhat lower values, just as they did in our studies of Si_3N_4.[16] Also shown in Figure 1 is the atomic fraction of AlN in the deposited film (the y in the above formula). These values were calculated assuming the index of refraction varies linearly with the fraction of AlN,[20,21] in the total volume and that the end point indices are 1.55[22] and 2.18[23] for Al_2O_3 and AlN respectively. The better films had a monotonic dependence of refractive index on nitrogen fractional pressure, explained with hindsight by the small oxygen contamination in the argon gas.

The films made with relatively little nitrogen were a mixture of free aluminum and Al_2O_3 and had a metallic appearance. The identification of free aluminum is also confirmed more directly from the Auger spectrum, where it is seen (Figure 2) that there are large differences in energy of the aluminum transition depending on whether the aluminum is free (68 eV), bound to oxygen (52 eV), or bound to nitrogen (57 eV). The relative

electronegativities of the three elements (Al - 1.18, N - 3.0, O - 3.5) suggest that the Al peak due to AlN bonding would indeed be intermediate between those associated with Al and Al_2O_3. A similar situation exists with silicon where the peak characteristic of Si-N bonding is found at about 89 eV[20] while the Si-O peak falls in the vicinity of 76 eV.

The approximate bulk composition of several films were determined from the Auger spectra by using atomic fractions of components as well as from the index of refraction (see Table I). The measured peak heights were corrected by the proper sensitivity factors to give the fraction of AlN as defined above. The general relationship between Al_2O_3 to AlN was also observed qualitatively in the relative peak heights of the Al lines at 52 and 57 eV.

Figure 3 shows the Auger depth profiles for several samples, picturing the important elements. The profile in part (a) shows an early sample where the fraction of oxygen is high and not uniform. There is some evidence that at the interface it may be bonded to the GaAs substrate. We attribute this possibility to the lack of proper predeposition removal of the substrate oxide.

In contrast, Figure 3b shows a relatively flat profile, taken from an intermediate quality sample (#7 in Table I). This sample had a relatively high oxygen content, and there was only light sputter cleaning of the target before deposition. A small oxygen excess is seen at the outermost surface, which we attribute to oxidation upon exposure to the atmosphere. There may also be a very small oxygen pile-up at the substrate interface, although in this case, the extra oxygen is bonded, at least primarily, to aluminum. The same sample after heat treatment is shown in Figure 3c. Here the outer surface shows further oxidation, and the interfacial oxygen pile-up has

become significantly more pronounced, suggesting some transport of oxygen through the dielectric film. The interface is also homogeneously broadened indicating that mutual inter-diffusion is occurring. It is seen, in fact (Figure 3c), that the arsenic outdiffusion is somewhat more pronounced than that of the gallium. Another sample (#4) with high oxygen content, but better predeposition cleaning, shows the oxygen pile-up at the interface, but no out-diffusion of arsenic. The final Auger spectrum (Figure 3d) shows a good quality sample with relatively high fraction of AlN after it has been annealed. In contrast to Figure 3c, we do not observe the out-diffusion of arsenic, or appreciable oxygen movement.

Effects of heat treatment are also seen in Fig. 4, where representative photographs (400x) show the aluminum oxynitride layers after annealing. The early samples deteriorate very quickly as illustrated in Figure 4a, taken after a 15 minute 500°C anneal. This obvious deterioration is probably due to poor adhesion resulting from the absence of in situ cleaning. If we proceed to Figure 4b where the sample substrate was sputter etched before deposition, but the target only lightly, we find some improvement in the degree of deterioration. Finally, in Figure 4c, we illustrate a high quality sample which was deposited after both the substrate and the target were well sputter cleaned. In this and similar samples there is no visible evidence of deterioration at an annealing temperature of 900°C.

IV. CONCLUSIONS

Aluminum oxynitride films can be deposited in a relatively straightforward manner using low energy ion beam sputtering, assuming that both target and substrate are given a thorough pre-deposition sputter etch. With reasonable care, films can be deposited that withstand temperatures to at least

900°C with no observable visual deterioration and minimal cross diffusion with the gallium arsenide substrate. This ability to serve as an effective encapsulant appears to improve as films closer to pure AlN are deposited.

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TABLE I. Summary of Data

Sample	Target Cleaning	Substrate Cleaning	N_2 Gas Fraction	Film Thickness (Å)	Refractive Index	Fraction AlN (index of refraction)	Fraction AlN (AES)	Obvious Deterioration
1	No	No	0.75	1080	1.65	0.17	0.15	410°C
2	No	No	0.90	1080	1.78	0.38	-	550
3	Yes	Yes	0.30	950	metallic	-	-	600
4	Yes	Yes	0.55	920	1.72	0.30	0.27	>900
5	Yes	Yes	0.70	910	1.73	0.31	-	900
6	Yes	Yes	0.89	1080	1.95	0.65	0.65	900
7	lightly	Yes	0.90	870	1.86	0.51	0.56	500
8	Yes	Yes	0.92	1010	1.90	0.57	-	-
9	Yes	only sputter no chemical	0.98	900	2.01	0.73	-	700
10	Yes	Yes	1.00	960	2.03	0.77	0.76	>900

FIGURE CAPTIONS

Figure 1. Index of refraction of aluminum oxynitride film vs. fraction of nitrogen gas in sputter beam. Deduced fraction of nitrogen in film is shown on second vertical axis. Triangles represent early runs without proper cleaning.

Figure 2. Auger spectrum of aluminum line from samples that are primarily (a) free aluminum, (b) Al_2O_3, (c) AlN.

Figure 3. Auger depth profiles: (a) poor quality early sample (#1), (b) intermediate quality smaple (#7), (c) same sample after annealing, (d) high index of refraction sample after annealing (#10).

Figure 4. Visual observation of annealed samples: (a) early sample (#2) with no sputter cleaning, 500°C anneal, (b) intermediate quality sample (#7), light sputter etch before deposition, 500°C anneal, (c) good original sample (#10), normal sputter etch, 900°C anneal.

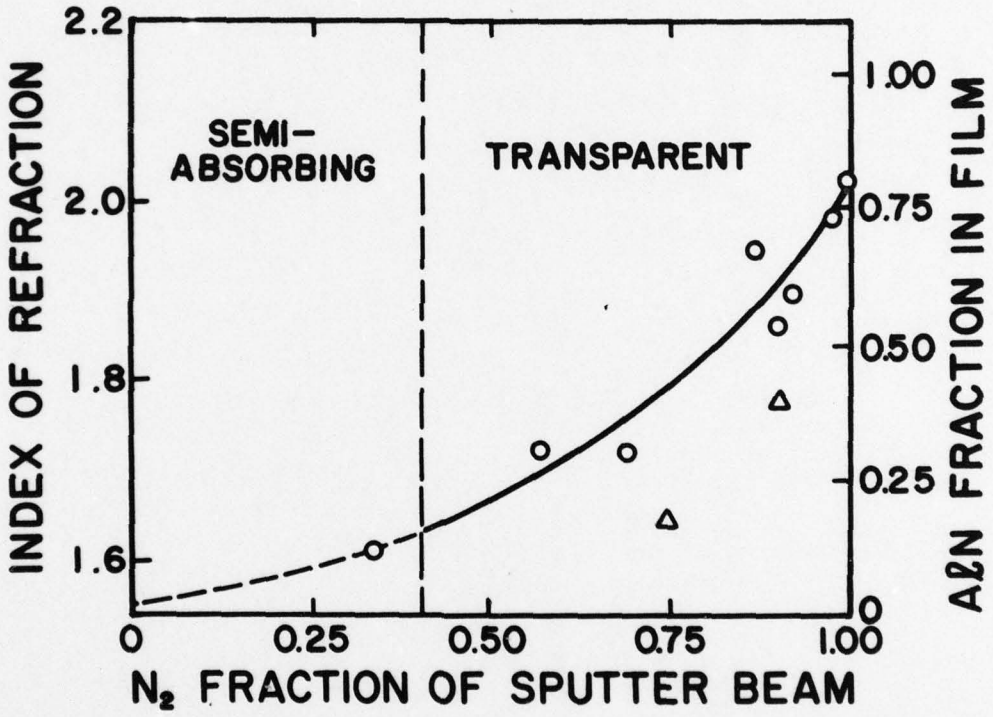

INDEX OF REFRACTION
2.2
2.0
1.8
1.6
SEMI-
ABSORBING
TRANSPARENT
1.00
0.75
0.50
0.25
0
AlN FRACTION IN FILM
0
0.25
0.50
0.75
1.00
N2 FRACTION OF SPUTTER BEAM

Fig. 1

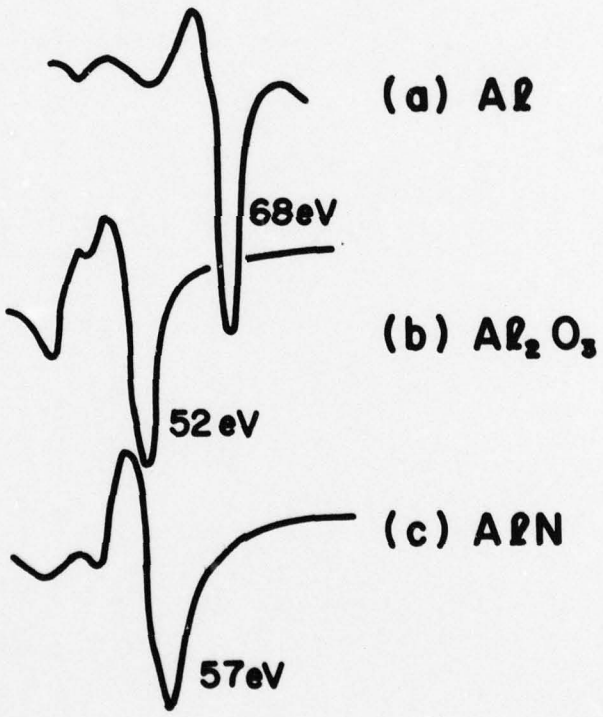


Fig. 2

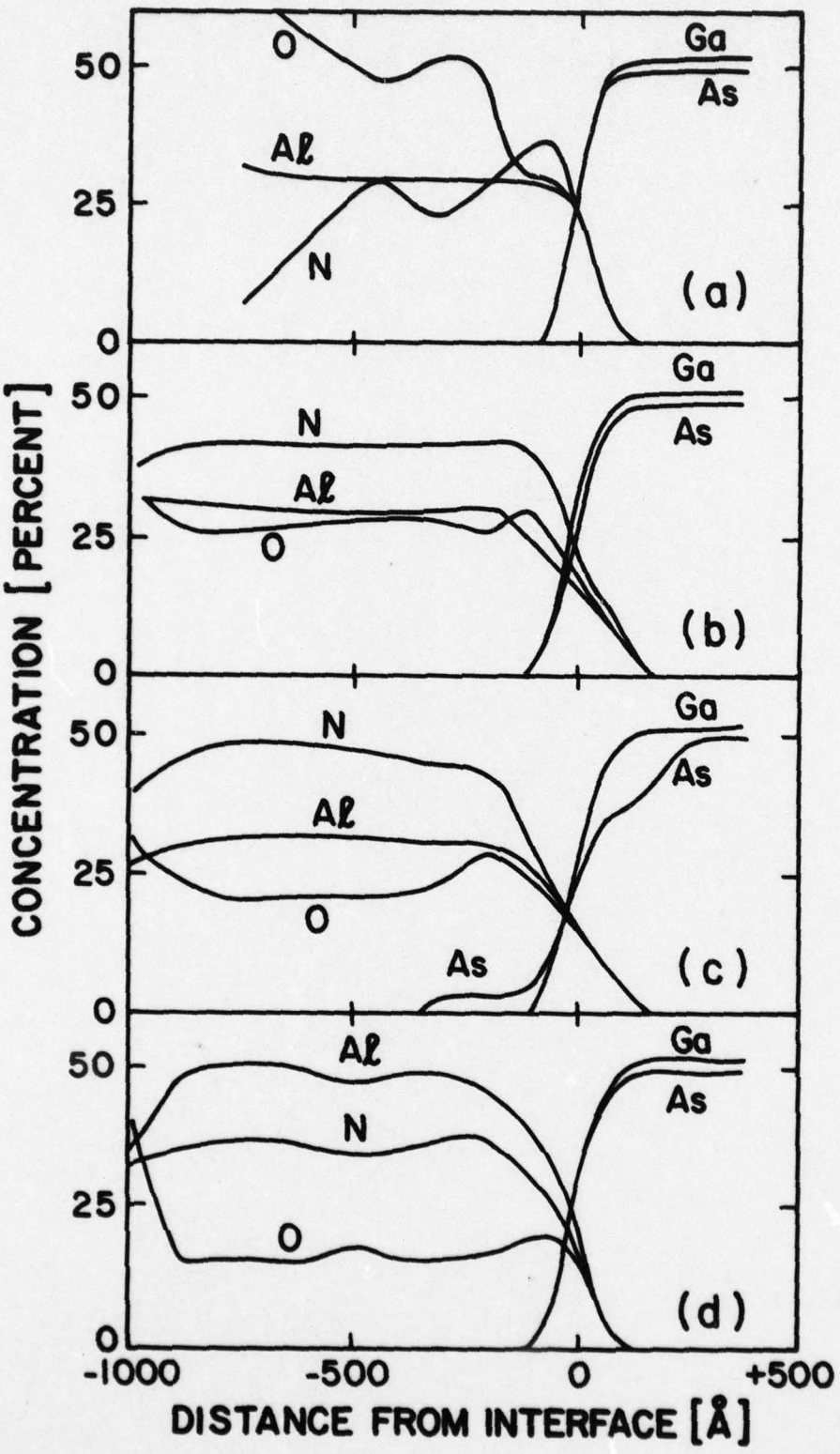
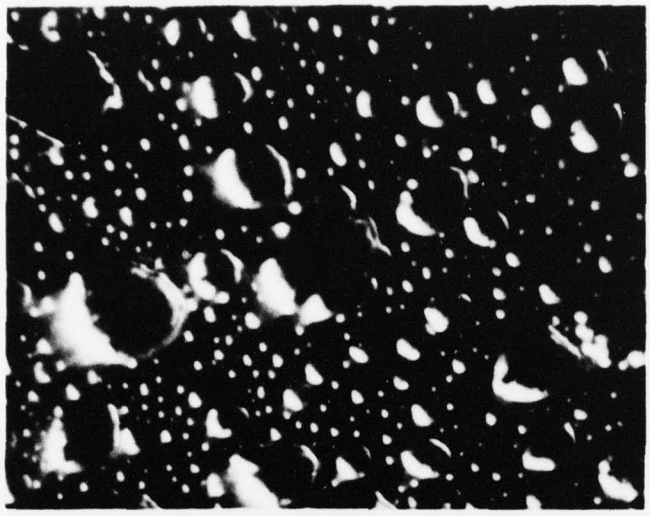
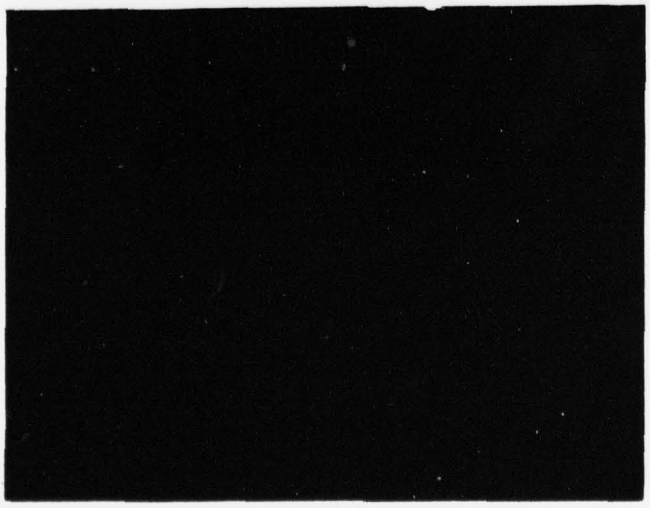

CONCENTRATION [PERCENT]
50
25
0
O
Al
N
Ga
As
(a)
(b)
(c)
(d)
-1000
-500
0
+500
DISTANCE FROM INTERFACE [Å]

Fig. 3



(a)

(b)



(c)

50μm

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