Letters

Determining the Composition of InP-GaP Alloys using Vegard's Law

The mixed crystal system $In_xGa_{1-x}P$ is of interest because of its possible application to electroluminescent devices. At a composition near $x = 0.5$ the system exhibits the largest direct energy gap of any of the III-V zinc blende semiconductors [1]. Since the magnitude of the direct gap is composition-dependent a convenient method of measuring the alloy composition would greatly facilitate detailed studies on the system. One such method is the X-ray diffraction technique of determining lattice constants, but this requires a calibration curve for the alloy system. Earlier work [2] indicated that the InP-GaP system might not conform with Vegard's Law but the results reported here show that the system does conform (cf. GaP-GaAs system [3]).

The alloys used were crystallised by slowly cooling a gallium-indium melt in which InP had been dissolved. A seed crystal of either InP or GaP was used, depending on the composition of the deposited alloy. The grown alloys were separated mechanically from their substrates and ground to a fine powder. Lattice parameters were determined from their Debye-Scherrer powder patterns to within a precision of ± 0.002 Å using filtered CuKα radiation.

Alloy compositions were found using X-ray fluorescence (Philips 1212 spectrometer) by comparison with standards made from ground powder mixtures of InP and GaP in appropriate proportions. The precision of such an analysis was ± 2% and was limited by counting statistics on the small size of sample used (ca. 50 mg). The results are shown in fig. 1 which show that within experimental error Vegard's Law is obeyed for the InP-GaP system.

Having established that the variation in lattice constant is a linear function of alloy composition, the diffraction method has been used to measure the composition of epitaxial films of (In/Ga)P on single crystal substrates of InP using an X-ray diffractometer. In order to use the method in a standard Philips diffractometer, the substrate orientation must be on a lattice plane to within ± 1°, but with this proviso, the method enables the composition of an epitaxial film to be determined non-destructively.

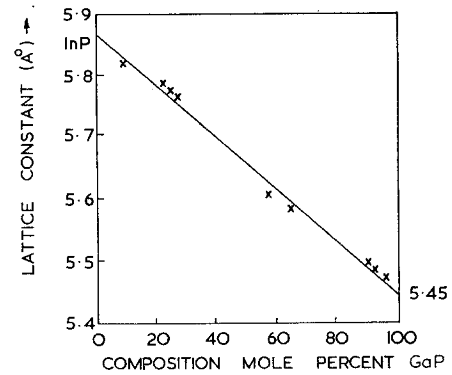

Figure 1 Lattice constant versus concentration.

In practice the substrate is mounted on the powder support table of the goniometer; the crystal angle is adjusted to the Bragg angle for the particular substrate orientation; the sample is rotated in its own plane until a maximum X-ray count rate is recorded and then a scan over a limited range of diffraction angles is run. A typical pattern of peaks is shown in fig. 2: two lines ($K\alpha_1$, $K\alpha_2$ doublets) are given for both film and substrate. To enable the substrate to be used as a reference in the measurement of diffraction angles the X-ray wavelength chosen should be energetic enough to penetrate through the film and give a measurable diffraction peak from the substrate as well as from the film itself, e.g. FeKα was found suitable for thin epitaxial film work (say 10 μm) but MoKα was needed to penetrate thicker (30 to 40 μm) films.

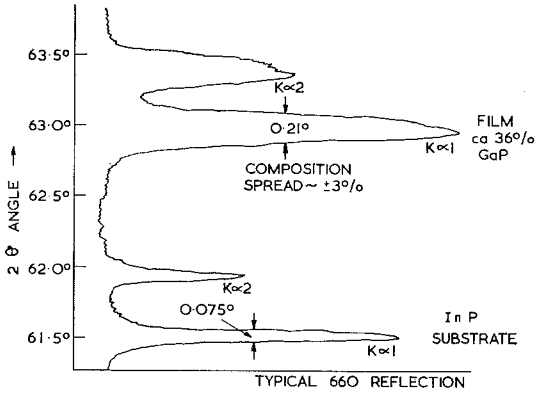

Figure 2 X-ray diffraction trace.

Under these conditions the substrate becomes the standard, and small errors caused by misalignment, absorption and eccentricity are minimised, providing the substrate and layer are of identical orientation [3].

The separation between the diffracted lines from the substrate and those from the film is then a measure of the composition of the alloy film; the relative line breadths of the diffracted peaks give a measure of either composition spread or of strain in the epitaxial film. Since the difference in angle (2θ) for 6th order 110 reflections between InP and GaP is 4.1°, and a measurement of 0.02° in 2θ is achievable using a chart recording, the precision with which the layer composition can be determined is little worse than that of the calibration standards.

We have shown that the InP-GaP alloy system obeys Vegard's Law and that the diffractometer provides a non-destructive method of determining the composition of epitaxial layers of the alloy on single crystal substrates of known orientation.

Acknowledgements

The authors wish to thank Dr C. Hilsum and Dr A. M. White for their overall guidance and consultation. This note is published by permission of the Director, RRE, Crown Copyright reserved.

References

1. C. HILSUM and P. PORTEOUS. IXth International Conference on the Physics of Semiconductors, Proceedings Vol. II, Moscow (1968).
2. N. N. SIROTA and V. V. ROSOV, *AN BSSR Doklady* (Russian) 7 (1963) 446.
3. E. W. WILLIAMS, R. H. COX, R. D. DOBROTT, and C. E. JONES. *Electrochem. Tech.* 4 (1966) 479.

Received 6 March and accepted 11 May 1970.

R. J. HERITAGE
P. PORTEOUS
B. J. SHEPPARD
RRE, Malvern
Worcs, UK

Kirkendall Effect and Diffusion in the Aluminium-Silver System

Diffusion in the aluminium-silver system has already been studied by Heumann and Dittrich [1], and by Heumann and Böhmer [2]. They have looked at both the interdiffusion coefficient and the partial diffusion coefficients in the aluminium-rich region.

In this work the diffusion coefficients have been determined, in the polycrystalline system, for the concentration in the marker interface, which was found to stay in the phase Ag_3Al. The interdiffusion coefficients were also calculated in the aluminium-rich phase and the calculated values were compared with the results of Heumann and Dittrich [1].

The interdiffusion coefficient has been calculated from the concentration penetration curves according to the graphical method of Matano by means of equation [3] of his paper

$$\tilde{D} = -\frac{1}{2t_a}\frac{dx}{dN_{Ag}}\int_{N_{Ag1}}^{N_{Ag2}} x \, dN_{Ag}\,, \qquad (1)$$

where N_{Ag} is the atomic fraction and t_a is the diffusion time.

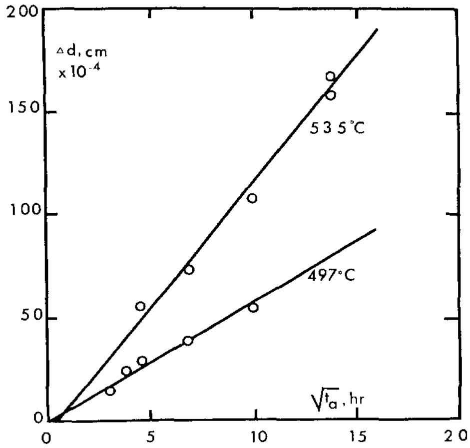

Figure 1 The marker displacement *Δd* as a function of the square root of the diffusion time t_a.