ion phenomena in polycrystalline tungsten carbide [8].

References

- 1. J. STEIGMAN, W. SHOCKLEY, and F. C. NIX, Phys. Rev. 56 (1939) 13.
- 2. C. LEYMONIE, "Radioactive Tracers in Physical Metallurgy". (J. Wiley and Sons, Inc., New York, 1963), pp. 54, 57.
- 3. T. J. RENOUF, Phil. Mag. 8 (1964) 781.
- 4. G. C. KUCZYNSKI and R. J. LANDAUER, J. Appl. Phys. 22 (1951) 952.
- 5. A. ANDREWS and S. DUSHMAN, J. Phys. Chem. 29 (1925) 462.

Growth of Epitaxial Silicon Layers by Ion Beam Sputtering

Sputtering as a method of growing semiconductor epitaxial layers has received very little attention compared with the more widely used methods of chemical transfer and vacuum-evaporation. Silicon layers showing preferred orientation have been grown by triode-sputtering [1], but there is no published account of good quality epitaxial silicon layers grown by any sputtering techniques. Only limited information is available on the growth of epitaxial layers of some of the other semiconducting materials such as Ge [2-6] and GaAs [7]. The twinning and high fault density present in these layers could be due to inherent limitations in the diode- and triodesputtering techniques used. These limitations are high operating pressures, low deposition rates and limited control of unwanted primary and secondary ions incident on the substrate surface. In the present work, high quality epitaxial silicon layers have been grown by a novel technique of sputtering by an argon ion beam of high current density in a vacuum in the range of 10⁻⁴ torr and at controlled rates of up to 400 Å per min. The bombardment of the substrate surface by positive ions during deposition, which has been shown to have an adverse effect on the quality of the layers, has been eliminated.

A schematic diagram of the sputtering arrangement is shown in fig. 1. Argon at a pressure of a few tenths of a torr was leaked into the ionisation chamber of an ion-beam source developed from the design of Nelson and Hill [8]. The electrons emitted from the heated tantalum filament ionised the argon atoms which were concentrated along the central axis of the chamber due to the magnetic field of the current

- 6. B. GOLDSTEIN, Rev. Sci. Instr. 28 (1957) 289.
- 7. C. E. GROUTHAMEL and C. E. JOHNSON, Analyt. Chem. 26 (1954) 1284.
- 8. C. P. BUHSMER, Ph.D. Thesis, State University of New York, College of Ceramics, Alfred, NY (1968).

Received 28 July and accepted

28 August 1970 C. P. BUHSMER State University of New York College of Ceramics New York, 14802 USA

through the filament. The extraction electrode at earth potential extracted a fraction of these ions from the ionisation chamber which was held at + 9 kV. A pressure in the range of 10⁻⁴ torr was maintained in the vacuum chamber by pumping argon at about 180 l/sec with a helium cryopump and by limiting the leakage of argon from the ionisation chamber only through the 2 mm aperture of the ionisation chamber. The ionsource was bakable to over 400° C and was water-cooled during the operation to reduce the contamination from it to a minimum. Contamination from the sputtering of the tantalum filament was minimised by maintaining the filament bias with respect to the ionisation chamber at 20 V, which is well below the threshold voltage for sputtering of tantalum of 26 V [9]. Another source of impurity was found to be due to the sputtering of the edge of the 3 mm aperture of the extraction electrode due to the tendency of the high-current-density ion-beams to diverge. To minimise this divergence, and hence the sputtering of the aperture, optimum values of angles for the electrodes were determined experimentally and were found to be $\phi_1 = 30^\circ$, $\phi_2 = 32^\circ$ and $\phi_3 = 42^\circ$. The emerging ion beam of between 3 and 8 mA bombarded the central region of a silicon target, a slice of silicon 32 mm in diameter and 3 mm thick, at energy of 12 keV and an angle of 60° to the target normal. The silicon sputtered from the target was deposited on a (111) surface of a single crystal silicon slice 32 mm in diameter and 0.25 mm thick, placed above the target. The slice was heated by focused radiation from outside the vacuum and was held at +9 kV to prevent it from being bombarded by positive ions. Before deposition, the slice was cleaned by flashing it at 1100° C for 2 min in a vacuum of 10^{-8} torr and the target was

© 1970 Chapman and Hall Ltd.



Figure 1 Schematic diagram of the sputtering arrangement for growing epitaxial silicon layers. The voltages and currents shown are typical.

cleaned by bombarding it for 10 min with the ion beam with the shutter closed.

Silicon layers were deposited at substrate temperatures of between 700 and 900° C at rates of up to 400 Å per min and were examined by transmission electron miroscopy. There was found to be a transition from polycrystalline to single crystal in the temperature range of 700 to 730° C. The appearance of the layer was indistinguishable from that of the chemically polished substrate. The single crystal layers were free from twinning, included grains and stacking faults. The layers, however, contained a number of small crystallographic defects the nature of which is not clearly understood. A more detailed investigation of these layers will be published in a later paper. Both p type layers on n type substrates and ntype layers on p type substrates have been deposited. The mesa section ($\sim 1 \text{ mm diameter}$) exhibited sharp diode characteristics, giving reverse breakdown voltages of between 30 and 150 V. The reverse current at voltages just below the breakdown voltages were about 10 μ A. As this reverse current varied significantly with the applied voltage, a large fraction of this reverse current could be due to surface leakage.

The layers grown with substrate at earth potential were heavily twinned and had a high stacking fault density. This is attributed to the bombardment of the surface by high energy, positive ions. The substrate ion current due to these ions was as high as 100 μ A.

This technique of sputtering is particularly suitable for compound semiconductors. Some initial work on GaAs by this technique, to be reported later, has produced stoichiometric layers. The main reason for starting with silicon was to show that the technique is capable of achieving the extremely high purity required for semiconductor applications.

References

- 1. A.H. CLARK and R.G. ALIBOZEK, J. Appl. Phys. 39 (1968) 2156.
- 2. K. E. HAQ, J. Electrochem. Soc. 112 (1965) 500.
- 3. S. P. WOLSKY, T. R. PIWKOWSKI, and G. WALLIS, J. Vac. Sci. Tech. 2 (1965) 97.
- 4. E. KRIKORIAN and R. J. SNEED, J. Appl. Phys. 37 (1966) 3665.
- 5. C. K. LAYTON and K. B. CROSS, *Thin Solid Films* 1 (1967) 165.
- 6. S. LUBY, ibid 4 (1969) 81.

- 7. B. MOLNAR, J. J. FLOOD, and M. H. FRANCOMBE, J. Appl. Phys. 35 (1964) 3554.
- 8. R. S. NELSON and K. J. HILL, Nucl. Instr. & Methods 38 (1965) 15.
- 9. R. V. STUART and G. K. WEHNER, J. Appl. Phys. 33 (1962) 2345.

Short Notices

Numerical Data and Functional **Relationships in Science and Technology New Series**

Group III: Crystal and Solid State **Physics:** Volume 4: Magnetic and other

Properties of Oxides and Related Compounds, Part a. Landolt-Börnstein

Editors: K. H. Hellwege and A. M. Hellwege

Pp xv + 367, 519 figures (Springer Verlag, 1970) D.M. 218; \$60,00

This part contains four articles in English and partly in German on: (1) "Fe oxides and Fe-Me-O compound systems (other than ferrites garnets and perovskites)" by R. A. Lefever; (2) "Compounds with lanthanide and actinide elements of some special structure types" by F. Holtzberg, T. R. McGuire and S. Methfessel; (3) "Perovskites" by J. B. Goodenough and J. M. Longo: (4) "Yttrium and rare earth garnets" by D. L. Huber. Part b, due to appear later, will deal with other garnets, spinels and hexagonal ferrites. This book follows the familiar pattern of the Landolt-Börnstein volumes in that it provides very comprehensive numerical data in the form of tables, graphs and diagrams, prefaced by a brief introduction, interlaced with terse summaries of theories and concluded by long lists of references. The subject matter includes information on phase diagrams, details of crystalline and magnetic structure and it extends over a wide range of mechanical spectroscopic, electrical and magnetic data. Many of the results quoted have been established during the last few years and substantial gaps remain in the underlying theory.

The authors are well-known authorities in their fields and this is reflected in the high standard of presentation and selection of their material. The book meets a definite need and will inevitably become a standard source of reference for anyone who, in the next few years, will need information quickly on the physical constants Received 1 July B. A. UNVALA and accepted 8 July 1970 **K. PEARMAIN** Department of Metallurgy Imperial College, London SW7

which determine the properties of these transition metal compounds. The cost of producing a work of this kind is inevitably considerable, though the price of this series is high by any standard. This will inevitably limit its availability to wellendowed specialist centres. This is a pity and it raises the question of whether it continues to be an economic proposition to provide encyclopaedic information on Materials Science in the format of an expensively bound, bi-lingual dictionary. RP

Clean Surfaces: Their Preparation and Characterisation for Interfacial Studies

Edited by George Goldfinger

Pp 385 (Marcel Dekker, New York 1970) 178s

Based on a Symposium held at North Carolina State University at Raleigh in 1968, this volume contains amplified versions of the papers presented there. The symposium aimed at an interdisciplinary collection of information on the nature and preparation of clean surfaces. The papers reflect this objective and include surface studies of a wide variety of materials including polymer crystals, latexes, alkali halides, mercury, aqueous liquids, glass, minerals, and semiconductor surfaces. A similarly wide variety of techniques employed for these surface studies include low energy electron diffraction, scanning electron microscopy, ellipsometry, transmission electron microscopy, electrochemical techniques and methods for adsorption studies. The book includes a number of review and theoretical papers and, since most of the chapters have good introductions, pointing out the relevance of the work to applications etc., it can be strongly recommended to readers not conversant with the field, as well as specialists, who will find the papers generally of a high standard of presentation and technical content. The only regret is that no discussion of papers is included, but it is clear that such interchange of ideas across disciplines could bring significant advances in this and other fields of materials science. A.F.W.W.