07.7–2 INTERCALATION OF NATURAL FLAKE GRAPHITES.

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Intercalation compounds of pure single crystal graphites are well known and characterised (Ubbelohde, Chemistry and Industry, (1983) p. 636). Naturally occurring flake graphites also form intercalates but without the regularity or stoichiometry of the more ordered materials.

The intercalation abilities of a range of flake graphites with bromine and bisulphate have been studied. The effects of factors such as the distribution of mineral matter, flake thickness and fissuring have been examined and the stabilities of the intercalate assessed.

Semi-automatic image analysis has been used to measure the flake thickness, the degree of fissuring and the changes which take place on intercalation. Bromine intercalation and desorption has been measured and related to the crystal perfection of the flake graphite. Scanning electron microscopy, with elemental analysis, has been used to measure the distribution of mineral matter in the flakes, the distribution of crystallites and to assess the exfoliation of the graphite.

Results show that the flake thickness is a prime factor influencing the extent of intercalation in these materials, the optimum thickness for bromine intercalation being 25 μm. Bromine uptake gives an indication of the perfection of stacking within the flakes of the graphite. Mineral matter within the flakes influences the thickness and cleavage properties.

07.8–1 INFRARED ABSORPTION OF LOCAL MODE CAUSED BY IMPURITY HYDROGEN IN SINGLE CRYSTAL SILICON

By Gu Benyuan, Xu Zhenyi and Ge Peiwen, Institute of Physics, Academia Sinica, Beijing, China.

A series of characteristic infrared absorption lines within the range 1835 to 2220 cm^{-1} have been observed in both single crystal silicon implanted with proton (Stein, J. Electronic Mat. (1975) 4, 159) and grown in a hydrogen atmosphere (Cui et al., to be published in Scientia Sinica). For single crystal silicon grown in a hydrogen atmosphere we suggest that there are four configurations of H in crystal. A Si atom could be substituted by 1,2,3 or 4 H atoms which departed from the lattice site toward the nearest neighbour Si atoms and bonded with them. We designated them a,b,c and d, respectively. The vibration frequencies of local modes in these cases were calculated based on molecule model. The molecules consist of H atoms located at Si vacancy and the nearest neighbour Si. The relation between Si-Si force constants and valence force constants was presented and the Si-Si force constants were determined by the known Si valence force constants (Solbrig, Jr.,J. Phys. Chem. Solids (1971) 32, 1761). In calculations we considered the nearest neighbour Si-H force constants and the influence of local lattice distortion and take all of them as variable paramaters. Using group theory we can calculate infrared active modes. The comparison between calculated and experimental results is given in following table. Experimental data in the table are strong lines observed in all single crystal silicon grown in a hydrogen atmosphere. There are twenty-two H local modes which are infrared active. Considering natural degeneracy there are only fourteen different frequencies. But ten of them can be classed in four groups, two of them are quasi-accidental twofold degeneracy, and the other two threefold. Consequently, there are only eight different frequencies. It is just equal to the numbers of strong lines observed. In the case of single crystal silicon implanted with proton, some kind of defects could arise from implanting. Therefore, their line numbers in infrared spectra are more than that grown in a hydrogen atmosphere.

Frequency(cm^{-1}) Exper.	Cal.	Configuration	Point group	Representation
548	547	a	C_{3v}	E
634	658	b	C_{2v}	A_1
	658			B_1
	658			B_2
791	753	c	C_{3v}	A_1
	753			E
	754			E
812	838	d	T_d	T_2
1949	1947	a	C_{3v}	A_1
1994	2038	b	C_{2v}	A_1
	2038			B_1
2124	2125	c	C_{3v}	A_1
	2125			E
2210	2208	d	T_d	T_2

07.8–2 INTERFERENCE ANALYSES OF MULTILAYERED THIN FILMS OF III-V COMPOUNDS BY MULTIPLE X-RAY DIFFRACTION.

By C. Schiller, L.E.P. Limeil-Brévannes (France), W.J. Bartels, J. Hornstra and D.J.W. Lobeek, Philips Research Laboratories, Eindhoven (The Netherlands)

Crystal growers of thin films of III-V compound semiconductors need complete information about lattice parameters and thicknesses of epitaxial layers, uniformity of composition as a function of depth and about the shape of the concentration depth profile. This is essential for different applications, lasers or MIS structures and high-electron-mobility transistors.

X-ray double diffraction (Bartels and Nijman, J. Cryst. Growth, 44 (1978) 518) and X-ray multiple diffraction (Bartels, J. Vac. Sci. Technol. B, 1 (1983) 338) provide quick and non-destructive analyses of thin films in the range of 0.1 to 10 μm.
Anomalies in diffraction intensities are frequently observed when the stacking sequence increase in complexity, interference fringes commonly detected in monolayer thin films become more confusing with, in some cases, contrasts increasing up to 50 % as regards to a monolayer diffracted intensity.

The $Ga_{1-x}Al_xAs$ system was studied using MOCVD grown layers with values of x from 0 to 0.8, leading to a lattice parameter change from 0 to 2.1 10^{-3} for $\Delta a/a$ perpendicular to the growth plane.

The kinematical diffraction model has been applied to layer structures with total thicknesses in the range of 0.1 to 5 μm : computing of diffracted intensities by Fourier summation of a function containing stacking sequence, number of layers, lattice parameter changes and concentration profiles has been realized.

An optimization of observed X-ray intensities is necessary to obtain a good correlation between theoretical work and experiments : this is possible for layers showing 10 to 20 fringes with different position and intensity.

The sensitivity is about 100 Å for the thickness variation at a total thickness of 8000 Å and about $2\ 10^{-5}$ for $\Delta a/a$, which corresponds to a variation of composition $\Delta x = 0.007$ in $Ga_{1-x}Al_x As$ epitaxial layers.

A unique solution will be found by a proper choice of thicknesses and compositions of different layers. This allows a rapid checking of epitaxial layers and gives information for different kinds of epitaxial growth as regards to homogeneity and concentration profiles.

07.8–3 X-RAY INVESTIGATION OF STRAIN FIELDS IN Sb ION-IMPLANTED Si. By A.P. Pogany, T. Preuss and H.K. Wagenfeld, Department of Applied Physics, Royal Melbourne Institute of Technology, Melbourne, Victoria, Australia.

A thick (100) Si crystal surface was implanted with $5.10^{15}/cm^2$ Sb^+ ions at 100 keV, and sequentially annealed at increasing temperatures. The initial amorphous layer regrows epitaxially with substitutional incorporation of the Sb, causing a strain field normal to the surface. X-ray rocking curves were measured with a double-crystal diffractometer, and matched to theoretical curves obtained from model strain fields using the Takagi-Taupin equations. The inferred strain is found to extend much further into the crystal than the Sb distribution itself; this is thought to result from movement of Si self-interstitials. After high temperature annealing the strain field decreases and ultimately disappears. This correlates with precipitation of Sb out of the solid solution. The general picture is supported by electron microscopy and ion backscattering measurements.

07.8–4 THERMAL EXPANSION AND CARRIER CONCENTRATION IN GaAs DOPED CRYSTALS. By J.Bak-Misiuk and M.Banasikowski, Institute of Physics of the Polish Academy of Sciences, Warszawa, Poland.

Lattice constant a of p and n types GaAs single crystals have been determined by the Bond method (accuracy $\frac{\Delta a}{a} = 10^{-5}$) at the temperature range of 300-700 K. Two kinds of courses of a = f(T) are observed - straight lines and lines with kinks. The obtained results are analysed on the basis of the equation proposed by Pietsch and Unger (phys.stat.sol. a, 1983, 80, 165).
The thermal expansion coefficient α is dependent on the dopant concentrations. For example α equals $5{,}37 \times 10^{-6}$ and $6{,}18 \times 10^{-6}\ K^{-1}$ for the n type GaAs with Te concentrations 3×10^{16} and $5 \times 10^{17}\ cm^{-3}$, respectively. The kinks of a = f(T) courses are connected with the carrier excitation from the deeper levels. The carrier concentration and dopant level can be estimated from thermal expansion coefficient and temperature of kink.
The results suggest that the absolute values of the deformation potentials of top of valence band Dp and bottom of conduction band Dn in GaAs are almost the same.
Precise lattice constant measurement at the higher temperature makes it possible to obtain the similar information also for another semiconductors.
The courses a = f(T) with kinks are also observed for InP doped with Sn (U.Pietsch, J.Bak-Misiuk, V.Gottschalch, phys.stat.sol. in print) and for CdSe single crystals, too.

07.8–5 DISLOCATION REDUCTION IN HEAVILY DOPED GALLIUM ARSENIDE SINGLE CRYSTALS. By G.Attolini, R.Fornari, C.Paorici and L.Zanotti, MASPEC-CNR, Parma, Italy.

As is known, important GaAs applications (e.g., microwave and optoelectronics devices) are still limited due to undesired structural defects, mainly dislocations. Dislocations occur because of thermoelastic stress, inherent to the growth process, in crystals having very low yield stress.
However, many authors reported (see, e.g., R. Fornari et al., J.Crystal Growth, 63 (1983) 415) that consistent yield stress increase (= hardening) can be achieved via heavy doping with particular dopants (S, Se, Si, In, etc.).
Object of this communication is here to refer to new results on dislocation reduction in lec grown silicon-doped and sulphur-doped GaAs.
In the case of GaAs:Si, the dislocation density (EPD) is correlated not only with the electrical activity, but also with the chemical activity, as defined by AAS analysis.
The results seem to confirm a solution-hardening type mechanism as the main cause of dislocation reduction.
In the case of GaAs:S, a "cracking" tendency is evidenced in heavily-doped dislocation-free crystals.