

CHARACTERIZATION

PURITY, COMPOSITION AND STRUCTURE

1.1 Chemical Methods

The work was done to develop accurate methods for the determination of traces of impurities in aluminium, copper, and germanium. A new water repellent chemical was developed for cellulose based materials. High purity acids, HCL and HNO₃ prepared by sub-boiling distillation, were analysed for ultra trace determination of metallic impurities. A number of milk samples were analysed for the evaluation of toxic pollutants like As, Cd, Pb and Cr for the preparation of milk standards.

A national '1991 Methane Campaign' for measurement of methane efflux rates from different major rice growing regions of the country was initiated. This was sponsored by the Deptt. of Environment & Forests and fourteen laboratories, institutes and universities participated. The absolute calibration compatibility at international level was established by exchanging samples with CSIRO, Australia and NIAES, Japan.

1.2 Indian Reference Materials

The preparation of Indian Reference Materials was continued. The data obtained from other laboratories was compiled and analysed. Nearly 80 bottles of IRM's were provided to different user organizations. Total organic carbon and inorganic carbon for a number of samples was determined. A two stage sub-boil distillation apparatus was designed and fabricated. A sequential optical emission spectrometer was installed and evaluated.

1.3 Surface Area & Porosity

Indigenous viscose rayon yarn was pre-treated and about twenty samples of activated carbon fibres were prepared. BET-surface area, yield of char and linear

shrinkage were determined. Samples of catalysts were also characterized.

1.4 FTIR Spectroscopy

The FTIR spectrophotometer and emission spectrometer were maintained and used for the characterization of materials. A number of samples from different projects and thin film coated silicon received from CEERI, Pilani, were characterized by IR absorption method using FTIR facilities. Samples of electronic materials received from, IIT, Bombay were characterized by FTIR transmission measurement.

1.5 EPR Spectroscopy

Microprocessor based EPR data acquisition system was set up with X-band EPR spectrometer. Research investigations were continued on different materials. A number of samples from NPL and outside organizations were characterized at ambient and low temperatures.

1.6 X-ray Fluorescence and Diffraction

Identification of elements was carried out on samples received from government deptts. and industry. Semiquantitative analysis of trace elements using Compton scatter method was attempted. In this method the matrix correction is simple and quick and requires only one standard sample. Trace elements determined by this method, in BDH silicon powder and other samples, agreed in general with the values determined by analytical method.

X-ray diffraction studies were carried out for samples of materials including superconductors, carbon fibres, poly-silicon films and lime-soil mixes etc. It was found that (2212) single phase could be transformed to nearly (2223) single phase by reacting at 860°C with CaO with addition of Pb for stabilization. However, a sample prepared under identical conditions but using the (2223) nominal composition showed only a very small amount of (2223) phase. In collaboration with CEERI, Pilani, crystallite size and X-ray texture studies were carried out on different LPCVD polysilicon films deposited at temperatures

of 570° and 620°C and annealed under nitrogen or oxygen atmosphere.

1.7 Electron Microscopy

Different sets of couples of gold/tin were prepared by changing the sequence of deposition of the metal films. Films with offset configuration were prepared by depositing Au and Sn sequentially onto copper grids. The structure and formation of various phases of the as-deposited as well as annealed samples were investigated by transmission electron microscope and diffraction techniques. For the formation of intermetallic alloy in this configuration the films had to be annealed. The study also revealed that the grain size in Au/Sn couples was smaller compared to that in Sn/Au couples.

Pure and lead doped superconducting materials were prepared by using solid state reaction technique. Effect of sintering time, cooling mode and composition on the superconducting properties of these materials were studied. The morphology of the phases formed were investigated by using scanning electron microscope. The microstructure of these materials was investigated by using transmission electron microscopy and electron diffraction techniques. T_c was found to increase with sintering time upto certain period, after which it showed a decreasing trend. The increase has been explained due to the increase in the volume fraction of high T_c (2223) phase in the material. The samples rich in Cu and Ca showed relatively higher values of T_c . TEM studies also showed the presence of isolated dislocations in some local regions.

A number of catalysts were investigated for particle size, shape and size distribution. In collaboration with IIT, New Delhi, the microstructural analysis of a black copper solar selective coating prepared by chemical conversion process was studied by using transmission electron microscopy technique.

CRYSTAL GROWTH AND CHARACTERIZATION

A high resolution X-ray diffraction system, developed at NPL, enabled first time direct observation of

anomalous transmission of X-rays Borrmann effect in thin natural diamond crystals. A new technique for isolating the forward diffracted beam from the residual direct beam was developed. This method in combination with the sharply defined highly collimated and monochromated exploring beam enabled direct observation of a well defined peak in the diffraction curve of the forward diffracted beam even when μt was only 0.3 and not ≥ 10 as expected theoretically.

High resolution X-ray diffraction, topography and curvature measurements were utilised for quantitative determination of stress induced by thin deposits of oxides, nitrides and silicides, in nearly perfect single crystals of silicon and also monitoring the degradation. The oxide film thicknesses were in the range of 250-1100Å and the nitride thicknesses in the range 700-1200Å. Large changes in radii of curvatures of the substrates were observed due to deposits. These investigations confirmed our earlier results on metallic deposits (ohmic contacts) on GaAs crystals that for measurement of stress it was necessary to take into account the initial bending of the blank wafers. The molybdenum films produced compressive stress of 2.17×10^9 dyne cm^{-2} . The oxide and nitride films produced stress of 5.6×10^9 dyne cm^{-2} . Interesting variations in contrast in topographs due to presence of stress in the case of molybdenum films were observed.

Lattice mismatch between gallium indium arsenide epitaxial films grown on indium phosphide substrates were determined by using high resolution X-ray diffractometry. Well defined peaks due to the substrate and the films could be observed in the diffraction curve of the specimen. The half widths of the diffraction curves (10-30 arc sec) indicated the good quality of the film and the substrate. It was observed that the films having compositions corresponding to near lattice match had a higher degree of perfection than those with finite mismatch. Also, films with the composition corresponding to compression mismatch were found to be of higher degree of crystalline perfection in comparison to those in tension. Composition of the films were obtained by using Vegard's law.

Single crystals of lithium niobate were grown successfully. Stoichiometry variations were studied and growth condition optimised. The diameter of the crystals was about 20 mm and length about 40mm. These crystals were characterized by using a five-crystal X-ray diffractometer in a three-crystal and a

one-crystal configuration. Typical diffraction curves recorded with (11.0) diffracting planes were very sharp with half widths in the range 10-30 arc secs. Traverse topographs were recorded in Laue geometry. Single crystals of K_2ZnCl_4 and K_2XCl_4 ($X=Cu, Fe$) were grown and their dielectric properties investigated.