

**PS10.02.11 STRUCTURE OF NANOCRYSTALLINE SPINEL FERRITE PRODUCED BY HIGH-ENERGY BALL-MILLING METHOD.** V. Sepelak<sup>1</sup>, A. Yu. Rogachev<sup>2</sup>, U. Steinike<sup>3</sup>, D.-Chr. Uecker<sup>3</sup>, S. Wissmann<sup>4</sup> and K. D. Becker<sup>4</sup>, <sup>1</sup>Institute of Geotechnics of the Slovak Academy of Sciences, 04353 Kosice, Slovak Republic, <sup>2</sup>Institute of Solid State Chemistry of the Russian Academy of Sciences, K 630091 Novosibirsk, Russia, <sup>3</sup>Institute of Applied Chemistry e.V., 12484 Berlin, Germany, <sup>4</sup>Institute of Physical and Theoretical Chemistry, Technical University of Braunschweig, 38106 Braunschweig, Germany

First mechanochemical synthesis (synthesis induced by a high-energy ball-milling process) of zinc ferrite from zinc oxide and iron oxide powders at room temperature is presented. Structure of the formed zinc ferrite with a large amount of strain and defects introduced during the mechanical treatment is characterized by means of X-ray powder diffraction, scanning electron microscopy, and Mössbauer spectroscopy. The ball-milling of the ZnO/Fe<sub>2</sub>O<sub>3</sub> mixture in a planetary mill results in the formation of zinc ferrite with non-equilibrium distribution of cations between tetrahedral and octahedral positions [1,2]. Subsequent thermal treatment of the disordered zinc ferrite leads to its crystallization at temperatures, which are by about 300 K lower than those at which the crystalline zinc ferrite is formed by a conventional thermal method commonly involving solid-state reactions.

[1] Sepelak, V., Steinike, U., Tkacova, K., Uecker, D.-Chr., Richter-Mendau, J., Jancke, K., Buchal, A., Rykov, A. I., Rogachev, A. Yu.: *Z.Kristallogr.Suppl.* 8 (1994) 673.

[2] Sepelak, V., Tkacova, K., Steinike, U., Boldyrev, V.V.: *European Powder Diffraction Conference EPDIC-4*, Chester 1995, *Mat.Sci.Forum* 1996, in press.

**PS10.02.12 MODULATIONS OF INTERMETALLIC B8-TYPE PHASES.** Sven Lidin, Margareta Elding & Lars Stenberg. Inorganic chemistry, Arrhenius Laboratory, Stockholm University, 106 91 Stockholm

Intermetallic B8 type phases exhibit a full spectrum of modes of ordering from short period, commensurate, superstructures to long period or incommensurate modulations and short range order as indicated by highly structured diffuse scattering. We will present results on modulated structures from the systems Cu-Sn, Co-Sn, Cu-In, Ni-Bi and Mn-Sn derived by electron diffraction and high resolution electron microscopy, single crystal X-ray diffraction and X-ray powder diffraction.

### Materials III

#### Strain and Stress Measurements

**MS10.03.01 PROFILING STRAIN IN THIN BURIED LAYERS BY CONVERGENT BEAM ELECTRON DIFFRACTION TECHNIQUES.** D. Cherns Physics Department, University of Bristol, Bristol, BS8 1TL UK

Convergent beam electron diffraction (CBED) provides a powerful method of recording crystal rocking curves. In contrast to X-ray rocking curves, electron rocking curves are not restricted to low order reflections and have spatial resolutions down to about 1 nm. The method has now been used to profile strain and composition in a range of semiconductor multilayer structures in both plan-view and cross-sectional geometries.

Electron rocking curves have been most usefully recorded using the large angle CBED (LACBED) method which shows spatial variations directly. LACBED also provides a means of filtering out most of the phonon- and some of the plasmon-generated inelastic background, giving useful rocking curve detail up to a tilt of more than 10° from the Bragg orientation. Studies of Si/SiGe multilayers

in a plan-view geometry have shown that layer strains may be determined to ~20% accuracy (1). Corresponding studies on cross-sectional samples (2) suggest a similar sensitivity; in this case strain relaxation must be taken into account. Work on single buried layers in InP/InGaAs in plan-view has shown that, using low order reflections, strains may be analysed for layers down to 1 monolayer in thickness (3). Recently we have extended these studies to high order reflections which are more sensitive to layer strains. Results obtained for thin Ge layers in Si indicate that strain can thereby be profiled across Ge islands where 3-D growth has occurred.

(1) D. Cherns, R. Touaitia, A. R. Preston, C. J. Rossouw and D. Houghton, *Phil. Mag.* **A64** (1991) 597.

(2) X-F. Duan, D. Cherns and J. W. Steeds, *Phil. Mag.* **A70** (1994) 1091.

(3) N. Grigorieff, D. Cherns, M. J. Yates, M. Hockly, S. D. Perrin and M. R. Aylett, *Phil. Mag.* **A68** (1993) 121.

**MS10.03.02 X-RAY STRAIN MEASUREMENTS ON SEMICONDUCTORS.** Paul F Fewster, Philips Research Laboratories, Cross Oak Lane, Redhill, U.K.

A combination of reciprocal space mapping and absolute lattice parameter determination can yield highly accurate and unambiguous strain measurements. Strain measurements are used extensively in x-ray diffraction for determining composition phase extent, layer relaxation and the presence of defects after ion implantation. The method most commonly used is the "rocking curve" method that assumes that the substrate has a known lattice parameter. Simulation of the "rocking curve" is necessary in most circumstances to separate strain from diffraction effects[1].

Layer tilts can complicate this measurement considerably and a general tilt will lead to errors unless this is taken into consideration. However reciprocal space mapping in three-dimensions can resolve the complication of tilts[2]. The uncertainties in the lattice parameter of the substrate are resolved by combining this reciprocal space mapping with a very high precision absolute lattice parameter method[3]. Examples will be given to show how the strain in simple, complex and inhomogeneous semiconductors can be determined.

[1] Fewster & Curling (1987) *J Appl. Phys.* **62** 4154

[2] Fewster & Andrew (1995) *J Phys. D* **28** A154

[3] Fewster & Andrew (1995) *J Appl. Cryst.* **28** 451

**MS10.03.03 STRAIN DETERMINATION IN SEMICONDUCTORS BY CONVERGENT BEAM ELECTRON DIFFRACTION.** A. Armigliato, R. Balboni, A. Benedetti, S. Frabboni\* and J. Vanhellemont+, CNR-Istituto LAMEL, via P. Gobetti 101, I-40129 Bologna, Italy, \*Dipartimento di Fisica, Università di Modena, via Campi 213/A, I-41100 Modena, Italy, +IMEC, Kapeldreef 75, B-3001 Leuven, Belgium

The determination of strain in nanoscale structures of semiconductors requires the availability of techniques with a high spatial resolution. Convergent beam electron diffraction, which is performed in a transmission electron microscope (TEM/CBED), is a point-to-point technique, with a resolution of the order of ten nm, which allows the strain tensor to be deduced from the shift of HOLZ lines in the central spot of the pattern. In its large angle version (LACBED) this technique has the advantage of combining both real space and reciprocal space information, with a spatial resolution limited by the probe size; in this case strain information can be obtained from a single pattern in different points of the imaged area through the shift and splitting of the ZOLZ Bragg contours. However, when applied to cross-sectioned specimens, the strain measured by CBED or LACBED is affected by the relaxation, which occurs in a direction parallel to the thinning direction. Methods of quantitative determination of relaxation in both uniform and graded Si<sub>1-x</sub>Ge<sub>x</sub>/Si heterostructures will be discussed; the bulk tetragonal distortion values thus obtained from (L)ACBED are in good agreement with the